Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

4,13-Diacetyl-[2.2]paracyclophane

Peter G. Jones,[a]* Henning Hopf[b] and Jörg Hillmer[b]

[a]Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany, and [b]Institut für Organische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany

Correspondence e-mail:
jones@xray36.anchem.nat.tu-bs.de

Received 7 February 2002
Accepted 12 February 2002
Online 22 February 2002

The molecule of the title compound, $C_{20}H_{20}O_2$, displays crystallographic twofold symmetry. The bridgehead bond lengths are 1.584 (3) and 1.590 (3) Å. There is slight distortion at one bridgehead C atom [C2—C3—C4 124.71 (13)° in standard cyclophane numbering]. The molecules are linked by a weak hydrogen bond of the form C—H···O to form a layer structure.

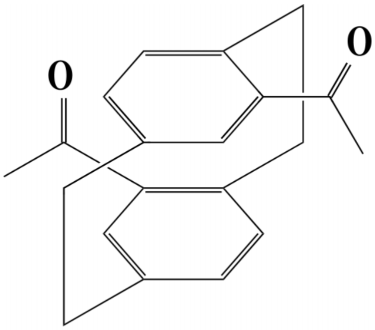
Key indicators

Single-crystal X-ray study
T = 178 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.134
Data-to-parameter ratio = 16.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Among the chiral [2.2]paracyclophanes relatively little is known about the 4,13-disubstituted derivatives, otherwise known as 'pseudo-*meta*' compounds. If the two substituents are identical, the compounds may, in principle, display ideal C_2 symmetry. Our interest in these compounds has led us to prepare the title compound, (I), by our established synthetic method (see *Experimental*); here we report its structure.



(I)

The molecule (Fig. 1) displays imposed twofold symmetry, with the twofold axis (at x = 0.5, z = 0.25) passing through the midpoints of C2—C2^i and C9—C9^i [symmetry code (i): 1−x, y, 1/2−z]; the atom numbering is standard for one half of a cyclophane molecule. The six-membered rings display the distortion towards a boat form that is typical of [2.2]paracyclophanes, whereby the bridgehead atoms C3 and C6 are displaced by 0.182 (2) and 0.174 (2) Å, respectively, from the plane of the remaining four atoms (mean deviation 0.004 Å). Also typical is the lengthening of the bridge bonds to 1.584 (3) and 1.590 (3) Å. The carbonyl group is rotated out of the corresponding ring plane, with a torsion angle C3—C4—C17—O of −32.2 (2)°. The substituent is associated with some distortion at C3, with a C4—C3—C2 angle of 124.71 (13)°.

The molecules are connected by a weak C18—H18A···O hydrogen bond *via* a twofold screw axis (at x = 0.75, z = 0.25) to form layers parallel to the ab plane (Fig. 2).

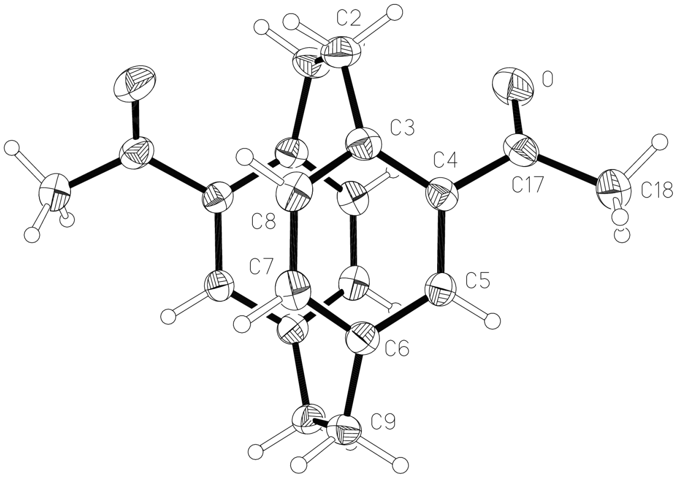


Figure 1
The molecule of the title compound in the crystal. Ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was prepared by the standard method (Hopf *et al.*, 1981) by cycloaddition of but-3-yn-2-one (ethynyl methyl ketone) to 1,2,4,5-hexatetraene (biallenyl) in toluene at 348 K. Apart from the title compound, which is formed in 8% yield, other isomers are produced. These were separated by preparative middle pressure chromatography on silica gel with dichloromethane (Hillmer, 1991). Crystals were grown by evaporation from 2-propanol.

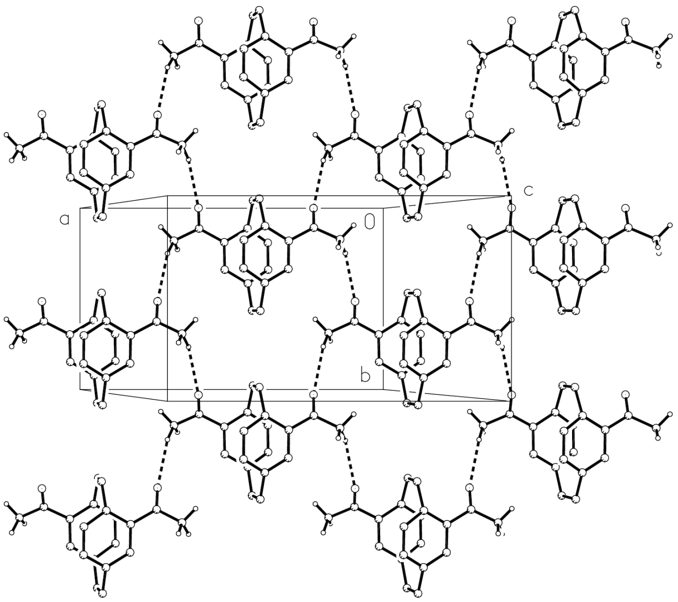


Figure 2
Packing diagram of the title compound, with the view direction perpendicular to the *ab* plane. The hydrogen bond is indicated by a dashed line; H atoms, other than those of the methyl group, have been excluded for clarity. There are two such layers, related by inversion symmetry, per *c* axis repeat. Radii are arbitrary.

Crystal data

$C_{20}H_{20}O_2$
$M_r = 292.36$
Monoclinic, $C2/c$
$a = 15.827$ (6) Å
$b = 9.442$ (2) Å
$c = 11.423$ (4) Å
$\beta = 119.34$ (2)°
$V = 1488.1$ (8) Å^3
$Z = 4$
$D_x = 1.305$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
$\theta = 10$–12°
$\mu = 0.08$ mm^{-1}
$T = 178$ (2) K
Prism, colourless
0.70 × 0.45 × 0.40 mm

Data collection

Nicolet $R3$ diffractometer
ω scans
Absorption correction: none
3126 measured reflections
1710 independent reflections
1411 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\rm max} = 27.5$°
$h = -20 \rightarrow 0$
$k = -12 \rightarrow 8$
$l = -12 \rightarrow 14$
3 standard reflections every 147 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.134$
$S = 1.03$
1710 reflections
101 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 1.4684P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—C2$^{\rm i}$	1.584 (3)	C9—C9$^{\rm i}$	1.590 (3)
C3—C2—C2$^{\rm i}$	112.31 (7)	C7—C6—C5	116.50 (13)
C8—C3—C4	116.08 (13)	C7—C6—C9	120.95 (12)
C8—C3—C2	118.33 (13)	C5—C6—C9	121.63 (12)
C4—C3—C2	124.71 (13)	C6—C9—C9$^{\rm i}$	112.88 (7)
C3—C4—C17—O	−32.2 (2)	C6—C9—C9$^{\rm i}$—C6$^{\rm i}$	−5.6 (2)
C3—C2—C2$^{\rm i}$—C3$^{\rm i}$	14.1 (3)		

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C18—H18A···O$^{\rm ii}$	0.98	2.48	3.288 (2)	139

Symmetry code: (ii) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

H atoms on sp^2 C atoms were included using a riding model, starting from idealized positions. Methyl H atoms were located as rather weak, but distinct, maxima in difference syntheses, idealized and refined as rigid groups allowed to rotate but not tip.

Data collection: *P*3 (Nicolet, 1987); cell refinement: *P*3; data reduction: *XDISK* (Nicolet, 1987); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

Financial support from the Fonds der Chemischen Industrie is gratefully acknowledged. We thank Mr A. Weinkauf for technical assistance.

References

Hillmer, J. (1991). PhD thesis, Technical University of Braunschweig, Germany.

Hopf, H., Böhm, I. & Kleinschroth, M. (1981). *Org. Synth.* **60**, 41–48.

Nicolet (1987). *P*3 and *XDISK*. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.