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Key indicators

Single-crystal X-ray study
 $T = 173\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.038
 wR factor = 0.088
 Data-to-parameter ratio = 13.2

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

trans-[8]Paracyclophan-4-ene-3,6-dione

The title compound, $\text{C}_{14}\text{H}_{14}\text{O}_2$, displays approximate twofold symmetry. The six-membered rings shows distortions typical of strained cyclophanes, but these effects do not extend to the bridges. The packing is determined by three hydrogen bonds of the type $\text{C}-\text{H}\cdots\text{O}$.

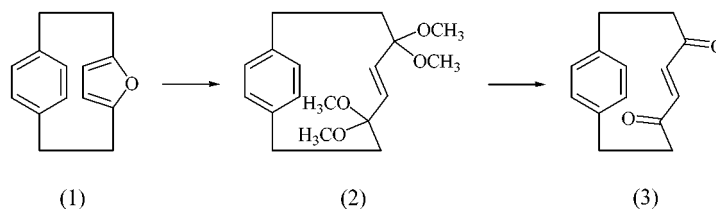
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Comment

The title enedione, (3), is useful as a starting material in cyclophane chemistry. Having investigated its chemical behaviour and spectroscopic data (Noble *et al.*, 1984*a,b*), we now describe its crystal structure.



The molecule of (3) (Fig. 1) possesses approximate twofold symmetry, as can be seen from the torsion angles in Table 1. Of the usual features of strained cyclophanes (see *e.g.* Jones *et al.*, 2002), it shows a flattened boat shape for the six-membered ring (atoms C9 and C12 both lie 0.106 (3) Å out of the plane of C10,C11,C13,C14), with narrow ring angles at C9 and C12. However, bond lengths and angles in the bridges C1–C2 and C7–C8 are normal for sp^3 C atoms. Contacts involving the bridgehead atoms are $\text{C3}\cdots\text{C12}$ 2.800 (3) and $\text{C6}\cdots\text{C9}$ 2.834 (3) Å.

The double bond $\text{C4}=\text{C5}$ is significantly twisted, with a torsion angle $\text{C3}-\text{C4}-\text{C5}-\text{C6}$ of 167.20 (15)°.

The crystal packing involves three $\text{H}\cdots\text{O}$ contacts that could be considered as hydrogen bonds (Table 2). These connect the molecules in a three-dimensional network, a section of which is shown in Fig. 2. The hydrogen bond $\text{C1}-\text{H1}\cdots\text{O1}$ forms rings of graph set $R^2_2(10)$ in the regions $z \sim 0, 1, \dots$ and $\text{C8}-\text{H8A}\cdots\text{O2}$ rings of the same set in the regions $z \sim 1/2, 3/2, \dots$; the two hydrogen bonds for which O2 is the acceptor combine to form larger rings $R^4_6(18)$. There are no short contacts of the form $\text{C}-\text{H}\cdots\text{Cg}$, where Cg is the ring centroid of C10,C11,C13,C14 (Jones *et al.*, 2002); the shortest such contact is $\text{H2A}\cdots\text{Cg}$ 3.11 Å (C–H normalized to 1.08 Å).

Experimental

[2](2,5)Furano[2]paracyclophane (1) was treated with bromine in methanol in the presence of potassium acetate to produce the bis-

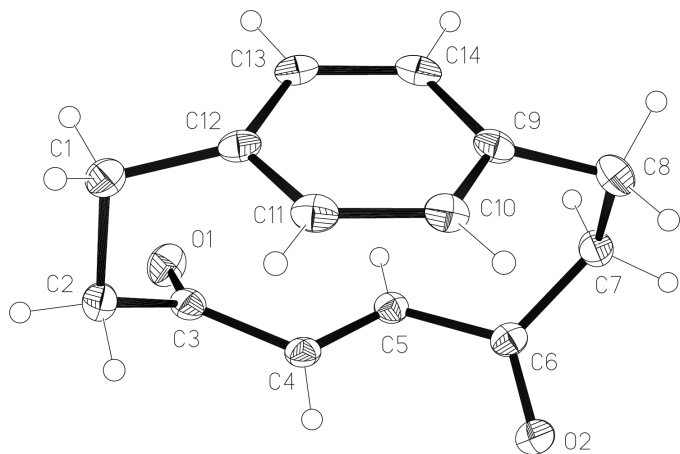


Figure 1
The molecule of compound (3) in the crystal. Ellipsoids represent 30% probability levels. H atom radii are arbitrary.

ketal (2), which on hydrolysis with dilute sulfuric acid provided the title compound (3) (Cope & Pawson, 1968; *cf.* Cram *et al.*, 1966). Single crystals were obtained from ethanol.

Crystal data

$C_{14}H_{14}O_2$	$D_x = 1.301 \text{ Mg m}^{-3}$
$M_r = 214.25$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 50 reflections
$a = 11.164 (3) \text{ \AA}$	$\theta = 10\text{--}11.5^\circ$
$b = 7.662 (3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 13.063 (4) \text{ \AA}$	$T = 173 (2) \text{ K}$
$\beta = 101.84 (3)^\circ$	Prism, colourless
$V = 1093.6 (6) \text{ \AA}^3$	$0.52 \times 0.42 \times 0.40 \text{ mm}$
$Z = 4$	

Data collection

Nicolet R3 diffractometer	$\theta_{\text{max}} = 25.0^\circ$
ω scans	$h = -13 \rightarrow 2$
Absorption correction: none	$k = -9 \rightarrow 0$
2335 measured reflections	$l = -15 \rightarrow 15$
1913 independent reflections	3 standard reflections
1263 reflections with $I > 2\sigma(I)$	every 147 reflections
$R_{\text{int}} = 0.025$	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.038$	$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2]$
$wR(F^2) = 0.088$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.89$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1913 reflections	$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$
145 parameters	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$

Table 1
Selected geometric parameters (\AA , $^\circ$).

C1—C2	1.551 (2)	C7—C8	1.554 (3)
C4—C5	1.327 (2)		
C12—C1—C2	109.33 (14)	C10—C9—C14	117.95 (18)
C9—C8—C7	110.32 (15)	C13—C12—C11	117.95 (17)
C12—C1—C2—C3	−45.7 (2)	C5—C6—C7—C8	86.32 (19)
C1—C2—C3—C4	91.75 (18)	C6—C7—C8—C9	−42.9 (2)
C2—C3—C4—C5	−141.63 (17)	C7—C8—C9—C10	100.1 (2)
C3—C4—C5—C6	167.20 (15)	C2—C1—C12—C13	101.53 (19)
C4—C5—C6—C7	−142.65 (17)		

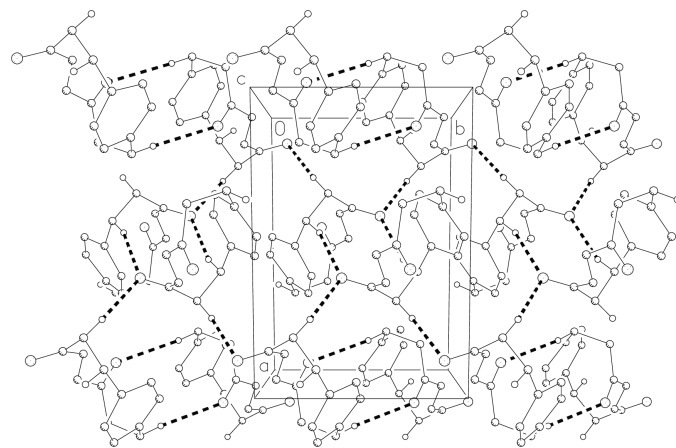


Figure 2
Packing of compound (3) in the crystal, viewed parallel to the c axis. Only those H atoms involved in H bonding (dashed bonds) are shown.

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
C1—H1A \cdots O1 ⁱ	0.99	2.67	3.633 (3)	163
C7—H7A \cdots O2 ⁱⁱ	0.99	2.66	3.613 (3)	161
C8—H8A \cdots O2 ⁱⁱⁱ	0.99	2.59	3.553 (3)	164

Symmetry codes: (i) $1 - x, -y, -z$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $1 - x, 1 - y, 1 - z$.

H atoms were included using a riding model with fixed C—H bond lengths (aromatic 0.95, methylene 0.99 \AA); $U(\text{H})$ values were fixed at $1.2 \times U(\text{eq})$ of the parent atom.

Data collection: *P3* (Nicolet, 1987); cell refinement: *P3*; data reduction: *XDISK* (Nicolet, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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