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[2.2](2,5)Furanoparacyclophane-12,13-dicarbaldehyde

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Key indicators

Single-crystal X-ray study
T = 178 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.133
Data-to-parameter ratio = 16.6

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

In the title compound, $C_{16}H_{14}O_3$, the furan ring makes an angle of 22.0 (1)° with the plane of the four non-bridgehead C atoms of the six-membered ring. The molecules are linked by a short C—H···O interaction to form helical chains parallel to the b axis.

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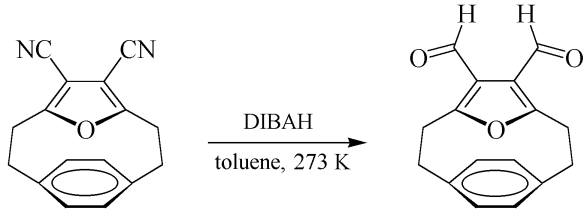
Comment

In our studies of the addition of highly reactive triple-bond dienophiles to strained aromatic compounds, we prepared the dinitrile (1) by the addition of cyanoacetylene to [2.2](2,5)-furanoparacyclophane (Hopf & Witulski, 1995; Witulski, 1992). To utilize this adduct for further transformations, we have reduced the nitrile to the bis-aldehyde, (2), using diisobutyl aluminium hydride (DIBAH) (Witulski, 1992).



(1) (2)

The structure of (2) is shown in Fig. 1. The six-membered ring shows the flattened boat form typical of paracyclophanes, with atom C3 lying 0.160 (3) Å and C6 0.170 (3) Å out of the plane of the other four atoms. The angle between the plane of these four atoms and the pane of the furan ring (r.m.s. deviation 0.02 Å) is 22.0 (1)°.

The packing is determined by a short C1—H1b···O3 interaction (Table 1), which links adjacent molecules related by the 2_1 screw axis, to form helical chains with an overall direction parallel to the b axis (Fig. 2). There are two such chains per unit cell.

Experimental

The title compound was prepared according to Witulski (1992) and was recrystallized from chloroform/pentane.

Crystal data

$C_{16}H_{14}O_3$
M_r = 254.27
Monoclinic, $P2_1/c$
a = 13.813 (3) Å
b = 8.909 (3) Å
c = 10.658 (3) Å
β = 107.44 (2)°
V = 1251.3 (6) Å^3
Z = 4
D_x = 1.350 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
θ = 10–11°
μ = 0.09 mm^{-1}
T = 178 (2) K
Prism, colourless
0.7 × 0.4 × 0.4 mm

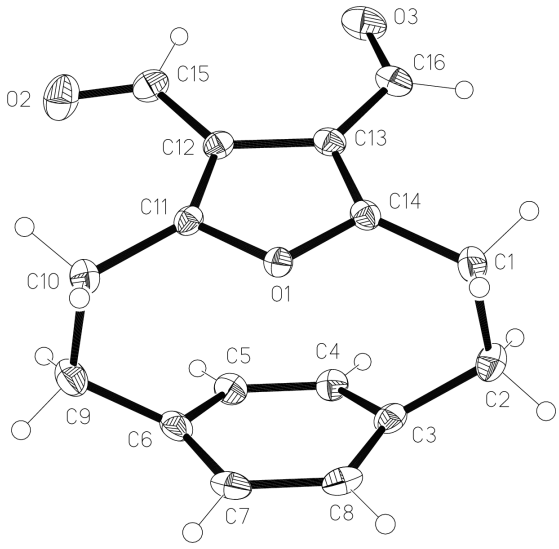


Figure 1
The molecule of compound (2) in the crystal. Ellipsoids are drawn at the 30% probability level and H-atom radii are arbitrary.

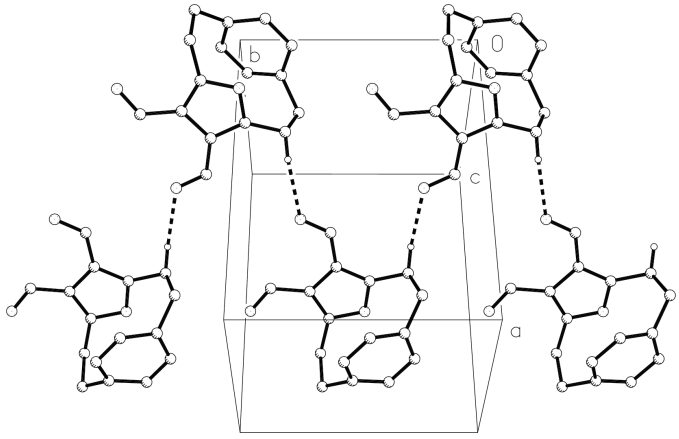


Figure 2
Packing diagram of compound (2). Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted. The view direction is perpendicular to $(10\bar{1})$.

Data collection

Nicolet *R*3 diffractometer	$\theta_{max} = 27.6^\circ$
ω scans	$h = -17 \rightarrow 17$
Absorption correction: none	$k = -1 \rightarrow 11$
3018 measured reflections	$l = 0 \rightarrow 13$
2857 independent reflections	3 standard reflections
1807 reflections with $I > 2\sigma(I)$	every 147 reflections
$R_{int} = 0.020$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$+ 0.8067P]$
$wR(F^2) = 0.133$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
2857 reflections	$\Delta\rho_{max} = 0.20$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}
H-atom parameters constrained	

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···O3^{i}	0.99	2.45	3.393 (3)	160

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

H atoms were included using a riding model, with fixed C—H bond lengths (sp^2 C—H = 0.95 Å and CH_2 = 0.98 Å); U_{iso}(H) values were fixed at 1.2 times the U_{eq} values of the parent atom.

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.

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