Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

# [2.2](2,7)Oxepinoparacyclophane-4,5-dicarbonitrile

**Peter G. Jones,[a]* Peter Bubenitschek,[b] Henning Hopf[b] and Bernhard Witulski[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: p.jones@tu-bs.de

Received 14 January 2003
Accepted 15 January 2003
Online 24 January 2003

**Key indicators**

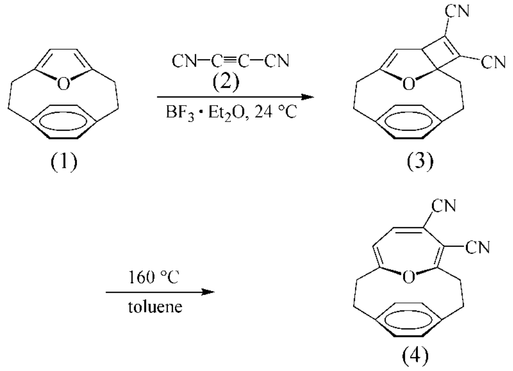
Single-crystal X-ray study
$T$ = 178 K
Mean $\sigma$(C–C) = 0.006 Å
$R$ factor = 0.068
$wR$ factor = 0.219
Data-to-parameter ratio = 12.7

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

In the title compound, $C_{18}H_{14}N_2O$, the O atom of the oxepine ring lies above the six-membered ring [3.008 (4) Å from the plane of the non-bridgehead atoms], but the rest of the oxepine ring is bent away from that plane and thus away from the molecule. Three C—H···N contacts connect the molecules to form layers parallel to the *ac* plane.

## Comment

When dicyanoacetylene (2) is added to [2.2](2,5)furanocyclophane, (1), in the presence of $BF_3$–etherate at room temperature, the [2+2]-cycloadduct (3) is formed in low yield (7%) (Witulski, 1992). To confirm its structure, we heated this addition product in toluene solution at 433 K and observed the expected ring opening to the oxepinophane (4), accompanied by a colour change to deep red (yield 68%; Witulski, 1992). We report here the structure of (4).



The molecule of (4) is shown in Fig. 1. Except for the carbonitrile substituents, the molecule shows approximate mirror symmetry. The six-membered ring shows the flattened boat form typical of paracyclophanes, whereby atom C11 lies

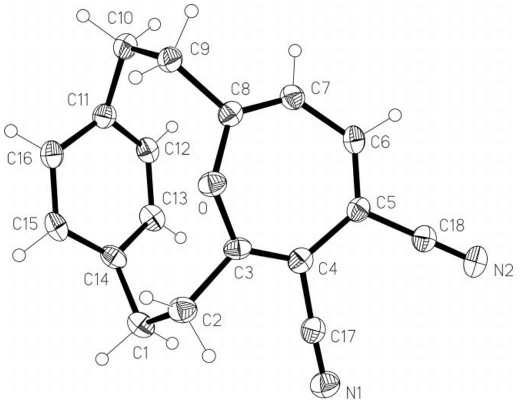


**Figure 1**
The molecule of compound (4) in the crystal. Ellipsoids are drawn at the 30% probability level and H-atom radii are arbitrary.

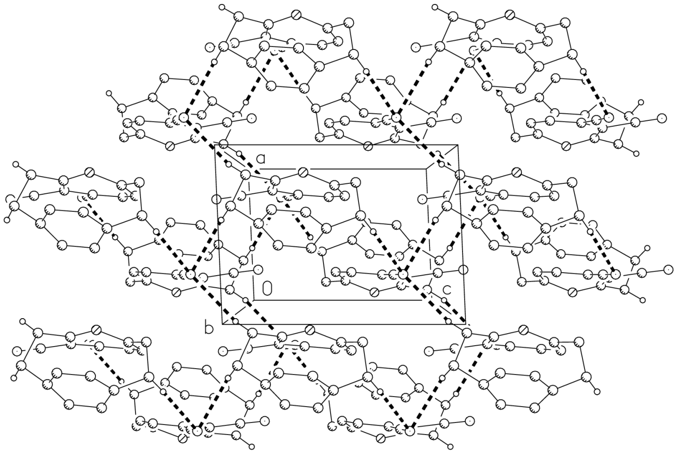


**Figure 2**
Packing diagram of compound (4). Hydrogen bonds are shown as thick dashed lines. H atoms not involved in hydrogen bonds have been omitted.

0.172 (6) Å and C14 0.152 (6) Å out of the plane of the other four atoms. The oxepine ring is non-planar, with absolute torsion angles of *ca* 31° about the bonds involving the O atom (and of *ca* 17° about C4—C5 and C6—C7). The net effect is that the O atom lies above the six-membered ring [3.008 (4) Å from the C12/C13/C15/C16 plane], but the rest of the oxepine ring is bent away from the six-membered ring and thus away from the molecule as a whole.

Three non-bonded contacts of the type C—H···N could be interpreted as weak hydrogen bonds (Table 2). The overall effect of these is to produce thick layers of molecules parallel to the *ac* plane (Fig. 2) in the regions $y \simeq 0, \frac{1}{2}, 1,$ *etc*.

## Experimental

Compound (4) was prepared as described above, isolated by thick-layer chromatography (Witulski, 1992), and recrystallized from chloroform/pentane.

*Crystal data*

$C_{18}H_{14}N_2O$
$M_r = 274.31$
Monoclinic, $P2_1/n$
$a = 6.494$ (3) Å
$b = 23.899$ (12) Å
$c = 8.783$ (4) Å
$\beta = 92.48$ (4)°
$V = 1361.8$ (11) Å$^3$
$Z = 4$
$D_x = 1.338$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 49 reflections
$\theta = 10$–11.5°
$\mu = 0.08$ mm$^{-1}$
$T = 178$ (2) K
Column, red
0.65 × 0.20 × 0.20 mm

*Data collection*

Nicolet $R3$ diffractometer
$\omega$ scans
2567 measured reflections
2409 independent reflections
1211 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\rm max} = 25.0°$
$h = -7 \rightarrow 7$
$k = -28 \rightarrow 0$
$l = 0 \rightarrow 10$
3 standard reflections every 147 reflections
intensity decay: none

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.219$
$S = 0.93$
2409 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.13P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.27$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O—C3 | 1.364 (5) | C1—C2 | 1.547 (6) |
| O—C8 | 1.415 (5) | C9—C10 | 1.571 (6) |
| | | | |
| C3—O—C8 | 127.4 (3) | C8—C9—C10 | 114.7 (3) |
| C14—C1—C2 | 106.0 (3) | C11—C10—C9 | 105.5 (3) |
| C3—C2—C1 | 114.8 (3) | | |
| | | | |
| C14—C1—C2—C3 | −51.7 (5) | C5—C6—C7—C8 | 17.1 (7) |
| C8—O—C3—C4 | 30.9 (6) | C6—C7—C8—O | −0.6 (7) |
| O—C3—C4—C5 | 0.4 (7) | C3—O—C8—C7 | −30.8 (6) |
| C3—C4—C5—C6 | −17.1 (7) | C8—C9—C10—C11 | 53.0 (5) |
| C4—C5—C6—C7 | 0.1 (7) | | |

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C1—H1*A*···N2$^{\rm i}$ | 0.99 | 2.66 | 3.541 (6) | 148 |
| C2—H2*B*···N2$^{\rm ii}$ | 0.99 | 2.70 | 3.582 (6) | 149 |
| C10—H10*B*···N2$^{\rm iii}$ | 0.99 | 2.66 | 3.649 (6) | 177 |

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

H atoms were included using a riding model with fixed C—H bond lengths ($sp^2$ C—H = 0.95 Å and methylene C—H = 0.99 Å); $U_{\rm iso}$(H) values were fixed at $1.2U_{\rm eq}$ 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.

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

## References

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.
Witulski, B. (1992). PhD thesis, Technical University of Braunschweig, Germany.

*Acta Cryst.* (2003). E**59**, o218–o219 Peter G. Jones *et al.* • $C_{18}H_{14}N_2O$ **o219**