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## Structure of Pentacyclo[8.5.0.0<sup>2,7</sup>.0<sup>4,13</sup>.0<sup>5,1</sup>]pentadeca-8,14-diene-3,6-dione

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(Received 29 November 1991; accepted 14 February 1992)

**Abstract.** C<sub>15</sub>H<sub>14</sub>O<sub>2</sub>, *M<sub>r</sub>* = 226.27, monoclinic, *P*<sub>2</sub><sub>1</sub>/*n*, *a* = 7.7505 (9), *b* = 16.484 (2), *c* = 8.7383 (9) Å, β = 101.16 (1)°, *V* = 1095.2 (2) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.372 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.7107 Å, μ = 0.84 cm<sup>-1</sup>, *F*(000) = 480, *T* = 295 K, *R*(*F<sub>o</sub>*) = 0.041, *wR* = 0.049 for 1205 observed reflections with |*F<sub>o</sub>*| ≥ 5.0(*F<sub>o</sub>*). The five-membered ring has an envelope conformation, while the two six-membered rings are in twist form. In the unit cell, the molecules are stabilized purely by van der Waals forces.

**Introduction.** It is well known that synthesis of higher prismanic frameworks is a formidable task owing to the problems posed by considerably higher steric energy and the large deviation in the tetrahedral C—C—C angle from the normal range. A series of

probing experiments on the heptacyclic ketones in quest for [7]-prismane homo- and secologues have been carried out. In this process three novel polyhedranes were synthesized through thermal [2 + 2] cyclo reversion of heptacyclic triones; a decarboxylated rearrangement product was also obtained (Mehta, Harikrishna Reddy & Padma, 1991). The title compound is this rearrangement product, a crystallographic study of which was undertaken to establish its molecular structure.

**Experimental.** Thin transparent colourless needle-shaped crystals were crystallized from dichloromethane–hexane mixture. A single crystal of size 0.25 × 0.15 × 0.1 mm was used for collection of a three-dimensional intensity-data set on an Enraf–Nonius CAD-4 automated X-ray diffractometer with monochromated Mo *K*α radiation; ω/2θ scan mode with ω = (0.6 + 0.35tanθ)°; aperture width = (1.8 +

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† DCB contribution No. 786.

Table 1. Fractional atomic coordinates for the non-H atoms and their equivalent isotropic temperature factors ( $\text{\AA}^2$ )
$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	$B_{\text{eq}}$
O1	0.0393 (3)	0.7425 (1)	0.6660 (2)	5.09 (6)
O2	0.1716 (3)	0.5819 (1)	1.0584 (2)	5.68 (7)
C3	0.4974 (3)	0.5769 (2)	0.8173 (3)	3.44 (6)
C4	0.2107 (4)	0.5855 (1)	0.9306 (3)	3.31 (6)
C5	0.4022 (3)	0.5262 (2)	0.6784 (3)	3.44 (7)
C6	0.3751 (3)	0.6251 (1)	0.9038 (3)	3.15 (6)
C7	0.1111 (3)	0.6030 (2)	0.6412 (3)	3.33 (6)
C8	0.0930 (3)	0.5504 (2)	0.7865 (3)	3.39 (7)
C9	0.3265 (4)	0.6380 (2)	0.4676 (3)	4.69 (10)
C10	0.2598 (4)	0.5714 (2)	0.5593 (3)	3.67 (8)
C11	0.4188 (4)	0.7008 (2)	0.5320 (4)	5.15 (9)
C12	0.1466 (3)	0.6890 (2)	0.6962 (3)	3.39 (7)
C13	0.1619 (4)	0.4650 (2)	0.7777 (3)	4.15 (8)
C14	0.3249 (4)	0.7014 (1)	0.7996 (3)	3.69 (7)
C15	0.3125 (4)	0.4541 (1)	0.7328 (3)	3.91 (8)
C16	0.4777 (4)	0.7116 (2)	0.7051 (4)	4.85 (9)
C17	0.6099 (4)	0.6451 (2)	0.7724 (4)	5.20 (9)

1.05tan $\theta$ ) mm;  $2 \leq 2\theta \leq 50^\circ$ ;  $0 \leq h \leq 9$ ,  $0 \leq k \leq 19$ ,  $-10 \leq l \leq 10$ . 2260 intensities were measured, of which 1920 were unique and 1205 observed with  $|F_o| \geq 5.0\sigma(F_o)$ ;  $R_{\text{int}} = 0.044$ . Cell dimensions were refined from 25 reflections in the range  $8.12 \leq \theta \leq 15.88^\circ$ . Three standard reflections ( $34\bar{2}$ , 342, 364) monitored every 100 reflections showed no significant variation. The maximum time spent on any measurement was 60 s and the background count was half the scan time. The intensity data were corrected for direct-beam polarization and Lorentz effects, but not for absorption. The structure was solved by direct methods (Sheldrick, 1990) and H atoms were located in  $\Delta\rho$  maps. Full-matrix least-squares refinement on  $|F_o|$  (Sheldrick, 1976), with anisotropic displacement parameters for non-H atoms and isotropic for H atoms, converged to  $R = 0.041$ ,  $wR = 0.049$ , with individual weights based on counting statistics where  $w = 1/[\sigma^2|F_o| + 0.00318(F_o)^2]$ ;  $(\Delta/\sigma)_{\text{max}} = 0.003$ ,  $(\Delta/\sigma)_{\text{min}} = 0.001$ ;  $(\Delta\rho)_{\text{max}} = 0.13$ ,  $(\Delta\rho)_{\text{min}} = -0.19 \text{ e \AA}^{-3}$ ;  $S = 0.96$  for 154 parameters.\* Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149).

**Discussion.** Fractional atomic positional parameters and  $B_{\text{eq}}$  values are given in Table 1. Bond lengths and angles involving non-H atoms are given in Table 2. Atom labelling† and a stereoview of the molecule

\* Lists of structure factors, anisotropic displacement parameters, H-atom parameters, torsion angles, bond distances and angles involving H atoms, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55197 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0267]

† The numbering of the structure is independent of the IUPAC numbering.

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) for the non-H atoms

O1—C12	1.206 (3)	O2—C4	1.214 (4)
C3—C6	1.542 (4)	C3—C17	1.520 (5)
C3—C5	1.539 (4)	C4—C6	1.490 (4)
C4—C8	1.519 (3)	C5—C10	1.553 (4)
C5—C15	1.500 (4)	C7—C10	1.559 (4)
C6—C14	1.557 (3)	C7—C8	1.566 (4)
C7—C12	1.505 (5)	C8—C13	1.513 (5)
C9—C11	1.321 (4)	C11—C16	1.504 (5)
C9—C10	1.508 (5)	C12—C14	1.511 (4)
C13—C15	1.314 (5)	C14—C16	1.578 (5)
C16—C17	1.538 (4)		
C6—C3—C17	100.6 (2)	C5—C3—C17	114.2 (2)
C5—C3—C6	114.8 (2)	O2—C4—C8	121.8 (2)
O2—C4—C6	122.6 (2)	C6—C4—C8	115.5 (2)
C3—C5—C15	111.2 (2)	C3—C5—C10	116.0 (3)
C10—C5—C15	106.2 (2)	C3—C6—C4	118.5 (2)
C4—C6—C14	108.7 (2)	C3—C6—C14	103.7 (2)
C10—C7—C12	110.8 (2)	C8—C7—C12	107.6 (2)
C8—C7—C10	112.2 (2)	C4—C8—C7	109.1 (2)
C7—C8—C13	112.7 (2)	C4—C8—C13	103.6 (2)
C10—C9—C11	123.9 (3)	C7—C10—C9	111.1 (3)
C5—C10—C9	115.7 (2)	C5—C10—C7	110.2 (2)
C9—C11—C16	123.7 (3)	O1—C12—C7	122.9 (2)
C7—C12—C14	114.1 (2)	O1—C12—C14	123.0 (3)
C8—C13—C15	119.0 (3)	C6—C14—C12	109.7 (2)
C12—C14—C16	113.2 (2)	C6—C14—C16	105.3 (2)
C5—C15—C13	118.7 (2)	C11—C16—C14	113.7 (3)
C14—C16—C17	103.6 (2)	C11—C16—C17	110.5 (3)
C3—C17—C16	104.7 (3)		

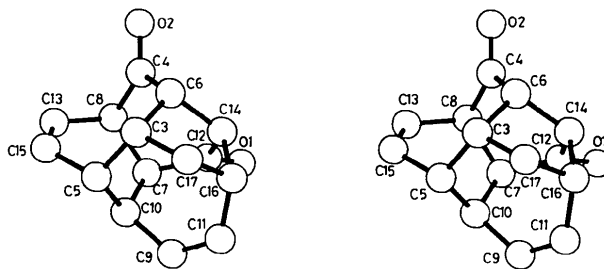


Fig. 1. Stereoview of the molecule with atomic numbering.

(Motherwell & Clegg, 1978) are shown in Fig. 1. The C7—C8 and C14—C16 bonds are significantly elongated as compared to the standard C—C bond length. This may be caused by the overcrowding of the atoms at these positions and the ring closure. The five-membered ring (C3, C6, C14, C16 and C17) has an envelope conformation with atom C17 deviating by 0.64  $\text{\AA}$  from the best plane, whereas the two six-membered rings (C4, C8, C7, C12, C14, C6 and C8, C7, C10, C5, C15, C13) are in twist forms. In the unit cell, the packing of the molecules is governed purely by van der Waals forces.

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