

Structure of Diethyl *cis,cis*-3,8-Cyclodecadiene-*trans*-1,6-dicarboxylate

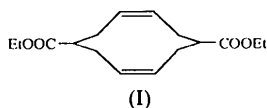
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Abstract. C₁₆H₂₄O₄, *M_r* = 280.36, triclinic, *P* $\bar{1}$, *a* = 6.5675 (8), *b* = 11.9949 (14), *c* = 5.1052 (6) Å, α = 93.418 (4), β = 100.420 (4), γ = 74.612 (6)°, *V* = 381.3 (2) Å³, *Z* = 1, *D_m* = 1.21 (2), *D_x* = 1.22 g cm⁻³, λ (Mo *K* α) = 0.71069 Å (Nb filter, no monochromator), μ = 0.81 cm⁻¹, *F*(000) = 152, *T* = 294 (2) K, *R* = 0.051 and *wR* = 0.060 for 977 unique observed reflections. The center of the molecule lies on a crystallographic inversion site so the molecule is required to be centrosymmetric. The diene groups have the *cis* configuration and the ester groups are oriented *trans* to each other. The ring has a chair conformation in which the double bonds mark upper and lower positions.

Experimental. M. Choubal prepared the title compound (I) from tetraethyl *cis,cis*-3,8-cyclodecadiene-1,1,6,6-tetracarboxylate according to Krapcho (1982), obtained colorless crystals (m.p. 342–343 K) from DMSO, and measured density by suspension in aqueous solutions of Zn(NO₃)₂. Cell parameters were



obtained from $\pm 2\theta$ measurements on 32 reflections in the range $20 < 2\theta < 30^\circ$. The prismatic data crystal had dimensions of $0.35 \times 0.25 \times 0.35$ mm. Two sets of intensity measurements were made using a modified Picker FACS-I diffractometer in the θ - 2θ scan mode over the range $-50 < 2\theta < 50^\circ$, maximum $\sin\theta/\lambda = 0.595 \text{ \AA}^{-1}$. A total of 5448 reflections were encountered between the *h*, *k*, *l* limits of ± 7 , ± 14 and $0 \rightarrow 6$, respectively. Five standard reflections (161, $14\bar{2}$, $31\bar{2}$, $1\bar{3}2$ and $33\bar{1}$) measured after every 150 showed less than 2% intensity change so corrections for crystal decay were not applied. Empirical absorption corrections were applied based on intensity variations of three reflections measured at regular intervals in φ with $\chi = 90^\circ$. Transmission factors ranged from 1.00 to 0.92. Intensity data were merged for symmetry equivalents to yield 1340 unique reflections (*R_{int}* = 0.049) and converted to

Table 1. Coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
O1	1105 (3)	-3382 (1)	-2906 (3)	80 (2)
O2	-1614 (2)	-3353 (1)	-835 (3)	64 (2)
C1	450 (3)	-2039 (2)	742 (4)	47 (2)
C2	-1621 (3)	-1083 (2)	905 (4)	50 (2)
C3	-2510 (3)	-448 (2)	-1656 (5)	48 (2)
C4	-2760 (3)	661 (2)	-2073 (5)	49 (2)
C5	-2246 (3)	1558 (2)	-113 (4)	48 (2)
C6	60 (3)	-2988 (2)	-1220 (4)	50 (2)
C7	-2208 (4)	-4243 (2)	-2602 (5)	67 (3)
C8	-3673 (5)	-3736 (3)	-5063 (6)	72 (3)

Table 2. Bond lengths (Å) and angles (°)

O1—C6	1.195 (2)	C1—C6	1.514 (3)
O2—C6	1.335 (2)	C2—C3	1.496 (3)
O2—C7	1.444 (3)	C3—C4	1.322 (3)
C1—C2	1.541 (3)	C4—C5	1.488 (3)
C1—C5'	1.532 (3)	C7—C8	1.489 (4)
C6—O2—C7	117.8 (2)	C3—C4—C5	128.0 (2)
C2—C1—C5'	112.3 (2)	C4—C5—C1'	112.7 (2)
C2—C1—C6	112.1 (2)	O1—C6—O2	123.1 (2)
C5'—C1—C6	110.7 (2)	O1—C6—C1	125.4 (2)
C1—C2—C3	112.4 (2)	O2—C6—C1	111.5 (2)
C2—C3—C4	127.4 (2)	O2—C7—C8	111.2 (2)

structure factors. 977 reflections were considered observed by the criterion $I > 3\sigma(I)$.

Statistical tests on intensity data indicated good probability for a centrosymmetric structure so space group *P* $\bar{1}$ was selected and later verified. The structure was solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and incorporated a model molecule generated by the program *PCMODEL* (Gilbert & Gajewski, 1987). The structure was refined for anisotropic non-H atoms by least-squares minimization of $\sum w(\Delta F)^2$ using *SHELX*76 (Sheldrick, 1976). Atomic scattering factors and real and imaginary components for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All H atoms were located in subsequent difference electron density maps. Refinement continued with H atoms in idealized positions (C—H = 0.95 Å) and used fixed isotropic temperature factors of 0.06 Å² for methyl H and 0.05 Å² for remaining H atoms. After the final cycle of refinement on 91 variables, *R* was unchanged

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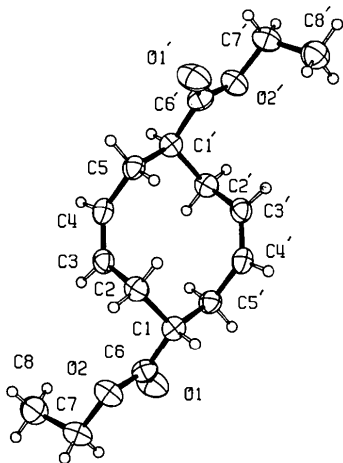


Fig. 1. An ORTEP diagram of the molecule using 40% probability ellipsoids for C and O atoms and showing the labeling scheme. Atoms with primed labels are at the symmetry related positions $-x$, $-y$, $-z$ of corresponding unprimed atoms. H atoms are shown as open spheres of arbitrary radius.

from 0.051, wR was unchanged from 0.060, the weighting factor was $1/[\sigma^2(F_o) + 0.001962(F_o)^2]$, the goodness of fit (S) was 1.17, $(\Delta/\sigma)_{\max} < |0.01|$, and $\Delta\rho_{\max}$ and $\Delta\rho_{\min}$ were 0.13 and $-0.19 \text{ e } \text{\AA}^{-3}$ respectively. Corrections for effects of extinction were deemed unnecessary. Final parameters are given in Table 1,* and Table 2 contains bond distances and angles. An ORTEP (Johnson, 1976) diagram of the

* Lists of U_{ij} values, H-atom coordinates and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54906 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0550]

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Structure of *r*-5-Cyano-5-methyl-*t*-4-methyl-*t*-7-oxo-7-phenyl-8-oxa-7-phosphabicyclo[4.3.0]non-2-ene Monohydrate

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Abstract. 6,7-Dimethyl-1-oxo-1-phenyl-1,3,6,7-tetrahydro-2,1-benzoxaphosphole-7-carbonitrile monohy-

molecule is shown in Fig. 1. In Table 2 and Fig. 1, atoms with primed labels are at the symmetry related positions $-x$, $-y$, $-z$ of corresponding unprimed atoms.

Related literature. Isolation and identification of the tetraester precursor to the title compound, and preliminary structural information on a stereospecific transannular cycloaddition product that it forms has been reported by Gipson, Guin, Simonsen, Skinner & Shive (1966). A boat conformation for crystals of *cis,cis*-3,8-cyclodecadiene-1,6-dione was deduced from space-group considerations and reported by Carrell, Roberts, Donohue & Vollmer (1968). Conformational features of the *cis,cis*-1,6-cyclodecadiene system have been reported by Allinger, Tribble & Sprague (1972).

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drate, C₁₆H₁₈NO₂P.H₂O, $M_r = 305.3$, monoclinic, Ia , $a = 12.058$ (4), $b = 7.791$ (1), $c = 17.006$ (3) Å, $\beta = 91.74$ (3)°, $V = 1596.9$ Å³, $Z = 4$, $D_m = 1.26$, $D_x = 1.269 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 1.75 \text{ cm}^{-1}$,

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