

5 α ,8 β ,14 α ,17 α -5,6,8,9,14,15,17,18-Octahydro-5,17:8,14-diepoxy-dibenzo[*e,e'*]benzo[1,2-*a:4,5-*a'**]dicyclooctene

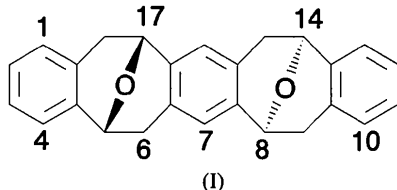
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Abstract. C₂₆H₂₂O₂, *M_r* = 366.46, monoclinic, *P*2₁/*n*, *a* = 6.521 (2), *b* = 9.223 (2), *c* = 15.235 (5) Å, β = 91.91 (3)°, *V* = 915.8 (8) Å³, *Z* = 2, *D_x* = 1.33 g cm⁻³, $\lambda(\text{Mo } K\alpha)$ = 0.71073 Å, μ = 0.8 cm⁻¹, *F*(000) = 388, room temperature, *R* = 0.044 for 1002 observed reflections. The relatively high density indicates the efficiency of the packing scheme, which is dominated by edge-to-face stacking of the aromatic moieties of the *Z*-shaped molecules.

Experimental. 5 α ,8 β ,14 β ,17 α -5,6,8,9,14,15,17,18-Octahydro-5,17:8,14-diepoxydibenzo[*e,e'*]benzo[1,2-*a:4,5-*a'**]dicyclooctene (I), an analog of Kagan's ether (Kagan, Chen & Agdeppa, 1977), crystallizes as colorless plates, dimensions 0.10 × 0.20 × 0.35 mm.



Density not measured. Unit-cell parameters and intensity data obtained on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo *K* α radiation in ω -2 θ mode ($0 < \theta < 23^\circ$). Cell dimensions refined by least-squares fitting of the θ values of 25 reflections with $10 < \theta < 15^\circ$. No significant drop in intensity of three standard reflections (23 $\bar{4}$, 226 2 $\bar{2}6$) checked every 7200 s. 1400 total reflections measured in *h, k* \pm *l* ranges, 0 to 0, -16 to 7 and 10 to 16, respectively; 1272 unique reflections, *R_{int}* = 0.01; 1002 reflections with $I \geq 2\sigma(I)$ used in subsequent calculations. Corrections applied for Lorentz and polarization effects but not for absorption. Structure solved by direct methods with *SHELXS86* (Sheldrick, 1985). H atoms located by ΔF synthesis and refined. Full-matrix least-squares refinement minimized $\sum w(|F_o| - |F_c|)^2$ with $w = 4F_o^2[\sigma^2(I) + (0.05F_o^2)^2]^{-1}$; anisotropic thermal parameters for C and O atoms and isotropic thermal parameters for H atoms. Final refinement with 171 parameters gave *R* = 0.044, *wR* = 0.065, *S* = 2.00 for observed data.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²) for non-H atoms with *e.s.d.*'s in parentheses

$$B_{\text{eq}} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
O(5)	0.2674 (2)	0.1752 (2)	0.8968 (1)	4.51 (4)
C(1)	0.4616 (4)	0.3609 (3)	0.6650 (2)	4.42 (6)
C(2)	0.6291 (4)	0.3038 (3)	0.6238 (2)	4.89 (6)
C(3)	0.7466 (4)	0.2000 (3)	0.6639 (2)	5.07 (6)
C(4)	0.6998 (4)	0.1510 (3)	0.7456 (2)	4.69 (6)
C(4a)	0.5309 (3)	0.2064 (2)	0.7887 (2)	3.54 (5)
C(5)	0.4798 (4)	0.1470 (3)	0.8780 (2)	4.38 (6)
C(6)	0.6159 (4)	0.2062 (3)	0.9525 (2)	4.15 (5)
C(6)	0.5570 (3)	0.3591 (2)	0.9759 (1)	3.29 (5)
C(16)	0.3142 (3)	0.5554 (3)	0.9709 (2)	3.67 (5)
C(16a)	0.3689 (3)	0.4165 (3)	0.9463 (1)	3.51 (5)
C(17)	0.2248 (3)	0.3271 (3)	0.8891 (2)	4.21 (5)
C(18)	0.2247 (4)	0.3701 (3)	0.7927 (2)	4.57 (6)
C(18a)	0.4110 (3)	0.3110 (2)	0.7490 (2)	3.51 (5)

Table 2. Bond distances (Å) and angles (°) with *e.s.d.*'s in parentheses

O(5)—C(5)	1.448 (3)	C(5)—C(6)	1.518 (4)
O(5)—C(17)	1.433 (3)	C(6)—C(6a)	1.508 (3)
C(1)—C(2)	1.382 (4)	C(6a)—C(16)	1.392 (3)
C(1)—C(18a)	1.410 (3)	C(6a)—C(16a)	1.397 (3)
C(2)—C(3)	1.359 (4)	C(16)—C(16a)	1.385 (3)
C(3)—C(4)	1.369 (4)	C(16a)—C(17)	1.506 (3)
C(4)—C(4a)	1.397 (3)	C(17)—C(18)	1.521 (4)
C(4a)—C(5)	1.514 (3)	C(18)—C(18a)	1.506 (3)
C(4a)—C(18a)	1.370 (3)		
C(5)—O(5)—C(17)	110.1 (2)	C(6)—C(6a)—C(16a)	120.4 (2)
C(2)—C(1)—C(18a)	120.1 (2)	C(16)—C(6a)—C(16a)	118.6 (2)
C(1)—C(2)—C(3)	120.4 (2)	C(6a)—C(16)—C(16a)	121.7 (2)
C(2)—C(3)—C(4)	120.2 (2)	C(6a)—C(16a)—C(16)	119.6 (2)
C(3)—C(4)—C(4a)	120.7 (2)	C(6a)—C(16a)—C(17)	120.2 (2)
C(4)—C(4a)—C(5)	119.3 (2)	C(16)—C(16a)—C(17)	120.1 (2)
C(4)—C(4a)—C(18a)	119.8 (2)	O(5)—C(17)—C(16a)	111.9 (2)
C(5)—C(4a)—C(18a)	120.8 (2)	O(5)—C(17)—C(18)	109.2 (2)
O(5)—C(5)—C(4a)	110.7 (2)	C(16a)—C(17)—C(18)	113.3 (2)
O(5)—C(5)—C(6)	109.0 (2)	C(17)—C(18)—C(18a)	111.0 (2)
C(4a)—C(5)—C(6)	113.6 (2)	C(1)—C(18a)—C(4a)	118.9 (2)
C(5)—C(6)—C(6a)	111.5 (2)	C(1)—C(18a)—C(18)	120.0 (2)
C(6)—C(6a)—C(16)	120.9 (2)	C(4a)—C(18a)—C(18)	121.1 (2)

Max. Δ/σ = 0.01 in final cycle, max. residual density 0.18 e Å⁻³. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV) and Enraf–Nonius (1982) *SDP* programs were used for all calculations.

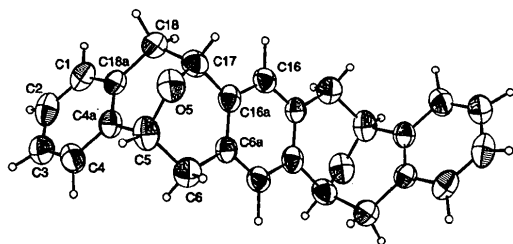


Fig. 1. Perspective view (ORTEP; Johnson, 1965) of the molecule giving the numbering scheme of the unique atoms. Thermal ellipsoids are drawn at the 50% probability level.

Table 1* gives the atomic coordinates and the molecular dimensions are given in Table 2. The atom-numbering scheme for the molecule, which lies on a center of symmetry, is given in Fig. 1. A stereoview of the packing scheme dominated by edge-face interactions of the aromatic rings is seen in Fig. 2.

Related literature. The title compound was prepared in the course of our work on molecular tweezers (Harmata & Barnes, 1990).

* Lists of H-atom parameters, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55416 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR0203]

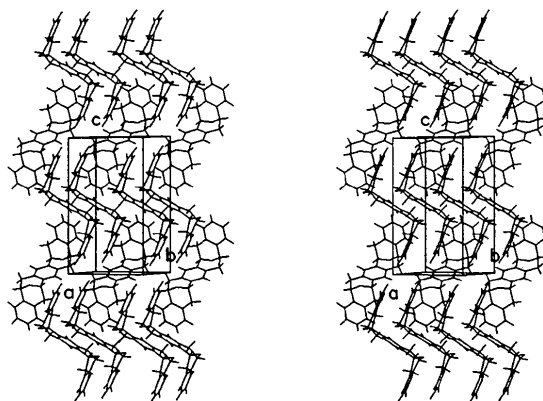


Fig. 2. Stereoview (PLUTO; Motherwell & Clegg, 1978) of the packing down the *a* axis.

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Structure of a Substituted 2-(Ethyl glycinat- α -ylidene)-1,4-benzodiazepine

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Abstract. α -(7-Chloro-1,3-dihydro-5-phenyl-2*H*-1,4-benzodiazepin-2-ylidene)-2,2,5,5-tetramethyl-1-aza-2,5-disilacyclopentane-1-acetic acid ethyl ester, C₂₅H₃₂ClN₃O₂Si₂, *M*_r = 498.17, triclinic, *P* $\bar{1}$, *a* = 10.938 (2), *b* = 13.626 (2), *c* = 10.700 (2) Å, α = 108.37 (1), β = 114.36 (1), γ = 73.79 (1)°, *V* = 1358.1 (4) Å³, *Z* = 2, *D*_x = 1.218 g cm⁻³, m.p. 409–

411 K, λ (Cu *K* α) = 1.54178 Å, μ = 23.16 cm⁻¹, *F*(000) = 528, *T* = 296 K, *R* = 0.056, *wR* = 0.076 for 2495 unique reflections having *I* > 3.00 σ (*I*). The compound has *E* geometry at the exocyclic double bond. A disordered C-atom position in the 2,5-disilazacyclopentane ring was identified. Internal hydrogen bonding of N(1)—H(1*N*) to the carbonyl