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### ***cis-cisoid-cis-transoid-cis-cisoid-cis-Perhydro-cis-6,13-pentacenediyl Diacetate***

BY P. VAN NUFFEL, G. H. PETIT, A. T. H. LENSTRA AND J. VAN LOOCK

*University of Antwerp (UIA), Department of Chemistry, Universiteitsplein 1, B-2610 Wilrijk, Belgium*

AND P. VANHEE

*Department of Organic Chemistry, State University Gent, Krijgslaan 271 (S-4bis), B-9000 Gent, Belgium*

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#### **Abstract**

$C_{26}H_{40}O_4$  is orthorhombic, *Pbca*, with  $Z = 8$ ,  $a = 9.972(4)$ ,  $b = 21.285(4)$ ,  $c = 21.825(9)$  Å,  $D_c = 1.195$  Mg m<sup>-3</sup>.  $R_w = 0.035$  for 2160 reflections. Ring B occurs in a boat-like conformation, while the other four six-membered rings are in a distorted chair conformation.

#### **Introduction**

The title compound, shown in Fig. 1, is important in a study of the ring inversion in perhydropolycenes (De Pessenier, Anteunis & Tavernier, 1978; Vanhee, De Pessenier, Anteunis & Tavernier, 1979). It was first synthesized by Vanhee (1981). A determination of its configuration from spectroscopic data only was difficult

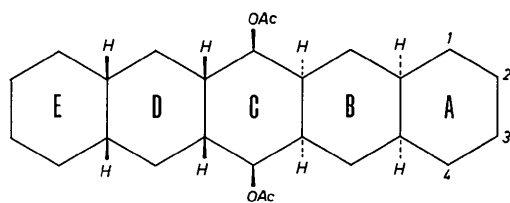


Fig. 1. Structural formula with nomenclature of the rings.

because of the complexity of the structure, the number of possible configurations (ten asymmetric C atoms) and the fact that the NMR investigation had shown the existence of two conformers, each present for about 50%. Therefore an X-ray investigation was undertaken to establish the configuration as well as the conformation in the solid state.

3207 independent reflections ( $0 < \theta < 23^\circ$ ) were measured at room temperature on an Enraf-Nonius CAD-4 automated diffractometer with Zr-filtered Mo  $K\alpha$  radiation. A pure  $\omega$  scan was used. Intensities were corrected for Lorentz and polarization factors. In view of the low absorption coefficient ( $\mu = 0.0847 \text{ mm}^{-1}$ ) and the size of the crystal ( $0.2 \times 0.2 \times 0.25 \text{ mm}$ ) no absorption correction was made.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). An *E* map using terms with  $E > 1.6$  revealed all non-hydrogen atoms. After refinement a difference map showed the positions of most of the H atoms; the five remaining H atoms were placed at expected positions.

The isotropic temperature factors of the H atoms attached to the ring system were fixed at  $B_{\text{iso}} = 4.0 \text{ \AA}^2$ , those of the acetyl groups at  $5.0 \text{ \AA}^2$ . The structure was refined by block-diagonal least squares to a final  $R_w = 0.035$  {defined by  $[\sum w(|F_o| - |F_c|)^2 / \sum w |F_o|^2]^{1/2}$ } for 2160 reflections with  $I > 2.0\sigma(I)$ .

A weighting scheme based on counting statistics was employed. The final difference map showed no maxima  $> 0.1 \text{ e \AA}^{-3}$ . The atomic parameters are presented in Table 1. Fig. 2 shows the conformation of the molecule.\*

### Discussion

The first result of this work is the assessment of the *cis-cisoid-cis-transoid-cis-cisoid-cis* positioning of the rings A–E and of the *cis* orientation of the acetyl groups, confirming the tentative conclusions about the configuration drawn from NMR data.

Bond distances and angles are presented in Tables 2 and 3. The large spread in C–C distances (1.510–

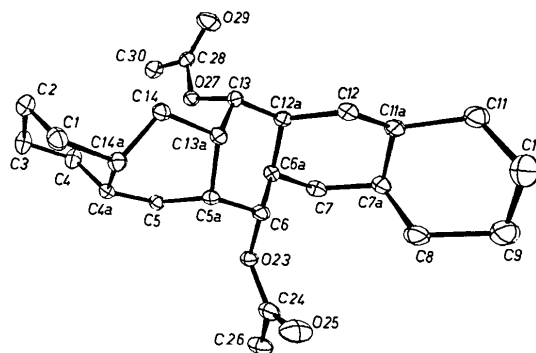


Fig. 2. The conformation of the title compound and the numbering of the atoms.

1.551 Å) and CCC angles ( $109.5$ – $116.9^\circ$ ) is not unusual. It reflects the non-equivalence of the various  $sp^3$ – $sp^3$  bonds. Similar phenomena were established in the perhydrophenanthrene skeleton (Romers, Altona, Jacobs & De Graaff, 1974). No significant differences are observed between the equatorially oriented acetyl group on C(6) and the axial one on C(13). The orientation of the acetyl groups differs considerably from the usual *gauche* position: torsion angles C(5a)–C(6)–O(23)–C(24) and C(12a)–C(13)–O(27)–C(28) are  $-93.2$  and  $-89.8^\circ$ , respectively.

The title compound has an unusual conformation. Rings A, C, D and E occur in a distorted chair form, but ring B takes on a boat-like conformation. At first sight this may seem surprising. A Dreiding model of the all-chair conformation, however, shows severe steric hindrance between ring A and the axial acetyl group on C(13). This steric compression cannot be released by converting ring C into a twist-boat form, but it can be diminished by converting ring B into a boat-like conformation. Furthermore, the energy difference between the non-substituted all-chair conformation and the form in which ring B has a boat-like conformation is not expected to be large. The arguments for this proposition stem from results of molecular mechanics calculations on *cis-cisoid-cis*-perhydroanthracene. These results may to some extent be extrapolated to perhydropentacene, since the latter can be considered as a combination of two perhydroanthracene units. The calculations (Allinger & Wuesthoff, 1971) show that in *cis-cisoid-cis*-perhydroanthracene the chair–chair–chair form is only  $8.8 \text{ kJ mol}^{-1}$  lower in energy than the chair–boat–chair form. The occurrence of a flexible form of ring B is a strong indication that the temperature dependence of the NMR spectra of the title compound is correctly attributed to a conformational equilibrium within ring B in the liquid state.

The ring torsion angles, Table 4, indicate that the rings are flattened with respect to an ideal six-membered ring. This flattening is undoubtedly introduced to decrease the unfavourable axial–axial 1,3 interactions, such as C(6)⋯C(8) in ring D and

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35935 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters in fractions of cell edges*

The e.s.d.'s given in parentheses refer to the last significant digit. Isotropic temperature factors ( $\text{\AA}^2$ ) of non-hydrogen atoms are calculated from anisotropic temperature parameters assuming equal volume of the 50% probability region. All anisotropic thermal parameters were physically acceptable.  $H(x_j)$  ( $j = 1,2,3$ ) is attached to  $C(x)$ ,  $H(xa)$  to atom  $C(xa)$ .  $B_{\text{iso}}$  was calculated according to Lipson & Cochran (1968):  $B_{\text{iso}} = 8\pi^2(U_{11}^2 + U_{22}^2 + U_{33}^2)^{1/3}$ .

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{iso}}$ ( $\text{\AA}^2$ )
C(1)	-0.2225 (3)	0.0014 (2)	0.3621 (1)	4.32 (14)
C(2)	-0.1927 (3)	0.0708 (2)	0.3531 (1)	4.68 (14)
C(3)	-0.1256 (3)	0.0809 (1)	0.2919 (1)	4.91 (13)
C(4)	0.0016 (3)	0.0426 (1)	0.2879 (1)	4.46 (13)
C(4a)	-0.0207 (3)	-0.0279 (1)	0.2976 (1)	3.43 (15)
C(5)	0.1143 (3)	-0.0640 (1)	0.2953 (1)	3.19 (14)
C(5a)	0.1260 (3)	-0.1126 (1)	0.3461 (1)	2.81 (14)
C(6)	0.2507 (3)	-0.1549 (1)	0.3450 (1)	3.06 (13)
C(6a)	0.3797 (3)	-0.1257 (1)	0.3692 (1)	2.96 (13)
C(7)	0.5019 (3)	-0.1698 (1)	0.3692 (1)	3.88 (15)
C(7a)	0.5063 (3)	-0.2209 (1)	0.4194 (1)	3.46 (14)
C(8)	0.4247 (3)	-0.2797 (1)	0.4056 (1)	4.37 (18)
C(9)	0.4340 (3)	-0.3282 (1)	0.4563 (1)	4.46 (15)
C(10)	0.3870 (3)	-0.2997 (1)	0.5162 (1)	4.34 (13)
C(11)	0.4708 (3)	-0.2426 (1)	0.5329 (1)	3.94 (12)
C(11a)	0.4743 (3)	-0.1927 (1)	0.4827 (1)	3.37 (16)
C(12)	0.3472 (3)	-0.1526 (1)	0.4814 (1)	3.19 (15)
C(12a)	0.3575 (3)	-0.0998 (1)	0.4342 (1)	3.05 (17)
C(13)	0.2382 (3)	-0.0553 (1)	0.4360 (1)	3.07 (12)
C(13a)	0.1101 (3)	-0.0831 (1)	0.4101 (1)	2.87 (13)
C(14)	-0.0073 (3)	-0.0359 (1)	0.4123 (1)	3.40 (14)
C(14a)	-0.0993 (3)	-0.0405 (1)	0.3572 (1)	3.37 (12)
O(23)	0.2824 (2)	-0.17499 (8)	0.28235 (7)	3.48 (11)
C(24)	0.2321 (3)	-0.2302 (1)	0.2629 (1)	3.98 (18)
O(25)	0.1511 (2)	-0.2604 (1)	0.2906 (1)	5.83 (19)
C(26)	0.2940 (3)	-0.2472 (2)	0.2026 (1)	5.09 (25)
O(27)	0.2747 (2)	0.00013 (9)	0.39923 (8)	3.53 (17)
C(28)	0.3352 (3)	0.0480 (1)	0.4278 (1)	3.88 (17)
O(29)	0.3496 (3)	0.0505 (1)	0.48218 (9)	6.00 (22)
C(30)	0.3773 (3)	0.0969 (2)	0.3836 (2)	5.40 (24)
H(11)	-0.265 (2)	-0.005 (1)	0.4015 (9)	4.00
H(12)	-0.299 (2)	-0.011 (1)	0.329 (1)	4.00
H(21)	-0.280 (2)	0.093 (1)	0.3540 (9)	4.00
H(22)	-0.131 (2)	0.087 (1)	0.3910 (9)	4.00
H(31)	-0.110 (2)	0.129 (1)	0.2845 (9)	4.00
H(32)	-0.178 (2)	0.071 (1)	0.259 (1)	4.00
H(41)	0.077 (2)	0.056 (1)	0.322 (1)	4.00
H(42)	0.056 (2)	0.047 (1)	0.253 (1)	4.00
H(4a)	-0.083 (2)	-0.042 (1)	0.263 (1)	4.00
H(51)	0.127 (2)	-0.0853 (9)	0.255 (1)	4.00
H(52)	0.176 (2)	-0.036 (1)	0.2982 (9)	4.00
H(5a)	0.059 (2)	-0.141 (1)	0.3418 (9)	4.00
H(61)	0.224 (2)	-0.191 (1)	0.3682 (9)	4.00
H(6a)	0.397 (2)	-0.089 (1)	0.341 (1)	4.00
H(71)	0.512 (2)	-0.191 (1)	0.330 (1)	4.00
H(72)	0.593 (2)	-0.144 (1)	0.3743 (9)	4.00
H(7a)	0.604 (2)	-0.237 (1)	0.422 (1)	4.00
H(81)	0.454 (2)	-0.296 (1)	0.3669 (9)	4.00
H(82)	0.344 (2)	-0.272 (1)	0.3994 (9)	4.00
H(91)	0.539 (2)	-0.341 (1)	0.4610 (9)	4.00
H(92)	0.376 (2)	-0.370 (1)	0.449 (1)	4.00
H(101)	0.309 (2)	-0.289 (1)	0.510 (1)	4.00
H(102)	0.385 (2)	-0.332 (1)	0.5509 (9)	4.00
H(111)	0.568 (2)	-0.263 (1)	0.5378 (9)	4.00
H(112)	0.428 (2)	-0.225 (1)	0.570 (1)	4.00
H(11a)	0.558 (2)	-0.163 (1)	0.4922 (9)	4.00
H(121)	0.330 (2)	-0.134 (1)	0.5225 (9)	4.00

Table 1 (cont.)

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{iso}}$ ( $\text{\AA}^2$ )
H(122)	0.266 (2)	-0.177 (1)	0.4747 (9)	4.00
H(12a)	0.428 (2)	-0.075 (1)	0.442 (1)	4.00
H(131)	0.212 (2)	-0.041 (1)	0.4792 (9)	4.00
H(13a)	0.095 (2)	-0.118 (1)	0.441 (1)	4.00
H(141)	0.027 (2)	0.008 (1)	0.417 (1)	4.00
H(142)	-0.055 (2)	-0.040 (1)	0.4506 (9)	4.00
H(14a)	-0.133 (2)	-0.084 (1)	0.3556 (9)	4.00
H(261)	0.240 (2)	-0.278 (1)	0.183 (1)	5.00
H(262)	0.377 (2)	-0.271 (1)	0.212 (1)	5.00
H(263)	0.318 (2)	-0.211 (1)	0.177 (1)	5.00
H(301)	0.385 (2)	0.138 (1)	0.401 (1)	5.00
H(302)	0.442 (2)	0.088 (1)	0.371 (1)	5.00
H(303)	0.309 (2)	0.107 (1)	0.352 (1)	5.00

Table 2. *Bond distances (\AA) with e.s.d.'s in parentheses*

C(1)–C(2)	1.520 (6)	C(8)–C(9)	1.515 (6)
C(2)–C(3)	1.510 (6)	C(9)–C(10)	1.515 (6)
C(3)–C(4)	1.511 (6)	C(10)–C(11)	1.520 (6)
C(4)–C(4a)	1.530 (6)	C(11)–C(11a)	1.525 (4)
C(4a)–C(5)	1.551 (6)	C(11a)–C(12)	1.528 (4)
C(5)–C(5a)	1.521 (6)	C(12)–C(12a)	1.528 (4)
C(5a)–C(6)	1.534 (4)	C(12a)–C(13)	1.520 (4)
C(6)–C(6a)	1.523 (4)	C(13)–C(13a)	1.517 (4)
C(6a)–C(7)	1.538 (6)	C(13a)–C(14)	1.544 (6)
C(7)–C(7a)	1.544 (6)	C(14)–C(14a)	1.517 (4)
C(7a)–C(8)	1.524 (6)	C(14a)–C(1)	1.522 (6)
C(4a)–C(14a)	1.542 (4)	C(6a)–C(12a)	1.539 (4)
C(5a)–C(13a)	1.540 (4)	C(7a)–C(11a)	1.541 (4)
C(6)–O(23)	1.467 (4)	C(13)–O(27)	1.473 (4)
O(23)–C(24)	1.347 (4)	O(27)–C(28)	1.338 (4)
C(24)–O(25)	1.196 (4)	C(28)–O(29)	1.196 (4)
C(24)–C(26)	1.499 (6)	C(28)–C(30)	1.481 (6)

Average C–H bond 0.98 (3)

Table 3. *Bond angles (°) with e.s.d.'s in parentheses*

C(2)–C(1)–C(14a)	113.7 (4)	C(7a)–C(8)–C(9)	112.5 (4)
C(1)–C(2)–C(3)	109.8 (4)	C(8)–C(9)–C(10)	109.8 (4)
C(2)–C(3)–C(4)	110.3 (4)	C(9)–C(10)–C(11)	111.0 (4)
C(3)–C(4)–C(4a)	113.6 (4)	C(10)–C(11)–C(11a)	113.4 (4)
C(4)–C(4a)–C(5)	110.8 (3)	C(7a)–C(11a)–C(11)	112.2 (3)
C(4)–C(4a)–C(14a)	111.1 (3)	C(7a)–C(11a)–C(12)	111.9 (3)
C(5)–C(4a)–C(14a)	112.5 (3)	C(11)–C(11a)–C(12)	112.6 (3)
C(4a)–C(5)–C(5a)	112.4 (3)	C(11a)–C(12)–C(12a)	111.6 (3)
C(5)–C(5a)–C(6)	116.7 (3)	C(6a)–C(12a)–C(12)	111.6 (3)
C(5)–C(5a)–C(13a)	112.1 (3)	C(6a)–C(12a)–C(13)	111.1 (3)
C(6)–C(5a)–C(13a)	109.7 (3)	C(12)–C(12a)–C(13)	112.8 (3)
C(5a)–C(6)–C(6a)	116.1 (3)	C(12a)–C(13)–C(13a)	114.0 (3)
C(6)–C(6a)–C(7)	114.9 (3)	C(5a)–C(13a)–C(13)	114.3 (3)
C(6)–C(6a)–C(12a)	110.2 (3)	C(5a)–C(13a)–C(14)	111.9 (3)
C(7)–C(6a)–C(12a)	109.5 (3)	C(13)–C(13a)–C(14)	111.9 (3)
C(6a)–C(7)–C(7a)	116.9 (3)	C(13a)–C(14)–C(14a)	113.0 (3)
C(7)–C(7a)–C(8)	115.1 (4)	C(1)–C(14a)–C(4a)	111.6 (3)
C(7)–C(7a)–C(11a)	110.9 (3)	C(1)–C(14a)–C(14)	113.2 (3)
C(8)–C(7a)–C(11a)	112.7 (4)	C(4a)–C(14a)–C(14)	110.5 (3)
C(5a)–C(6)–O(23)	111.1 (3)	C(12a)–C(13)–O(27)	107.0 (3)
C(6a)–C(6)–O(23)	105.1 (3)	C(13a)–C(13)–O(27)	108.5 (3)
C(6)–O(23)–C(24)	117.9 (3)	C(13)–O(27)–C(28)	117.8 (3)
O(23)–C(24)–O(25)	124.1 (4)	O(27)–C(28)–O(29)	123.5 (4)
O(23)–C(24)–C(26)	109.5 (4)	O(27)–C(28)–C(30)	111.0 (4)
O(25)–C(24)–C(26)	126.4 (4)	O(27)–C(28)–C(30)	125.5 (4)

Average H–C–H angle 106 (3)  
Average C–C–H angle 109 (2)

Table 4. Ring torsion angles (°) with e.s.d.'s in parentheses

Ring A		Ring C		Ring E	
C(14a)–C(1)–C(2)–C(3)	56.6 (7)	C(13a)–C(5a)–C(6)–C(6a)	–50.1 (7)	C(11a)–C(7a)–C(8)–C(9)	–51.9 (7)
C(1)–C(2)–C(3)–C(4)	–57.7 (7)	C(5a)–C(6)–C(6a)–C(12a)	54.2 (7)	C(7a)–C(8)–C(9)–C(10)	57.8 (7)
C(2)–C(3)–C(4)–C(4a)	57.4 (7)	C(6)–C(6a)–C(12a)–C(13)	–53.4 (7)	C(8)–C(9)–C(10)–C(11)	–58.7 (7)
C(3)–C(4)–C(4a)–C(14a)	–52.1 (7)	C(6a)–C(12a)–C(13)–C(13a)	53.3 (7)	C(9)–C(10)–C(11)–C(11a)	55.2 (7)
C(4)–C(4a)–C(14a)–C(1)	48.0 (7)	C(12a)–C(13)–C(13a)–C(5a)	–50.9 (7)	C(10)–C(11)–C(11a)–C(7a)	–48.5 (7)
C(4a)–C(14a)–C(1)–C(2)	–51.8 (7)	C(13)–C(13a)–C(5a)–C(6)	46.9 (7)	C(11)–C(11a)–C(7a)–C(8)	46.4 (7)
Ring B		Ring D			
C(14a)–C(4a)–C(5)–C(5a)	10.8 (7)	C(12a)–C(6a)–C(7)–C(7a)	49.4 (7)		
C(4a)–C(5)–C(5a)–C(13a)	–57.2 (7)	C(6a)–C(7)–C(7a)–C(11a)	–47.5 (7)		
C(5)–C(5a)–C(13a)–C(14)	44.0 (7)	C(7)–C(7a)–C(11a)–C(12)	49.5 (7)		
C(5a)–C(13a)–C(14)–C(14a)	13.7 (7)	C(7a)–C(11a)–C(12)–C(12a)	–57.1 (7)		
C(13a)–C(14)–C(14a)–C(4a)	–59.7 (7)	C(11a)–C(12)–C(12a)–C(6a)	59.6 (7)		
C(14)–C(14a)–C(4a)–C(5)	46.1 (7)	C(12)–C(12a)–C(6a)–C(7)	–53.8 (7)		

Table 5. Orientation of the substituents on the various rings, in terms of the polar angle  $\alpha$  (e.s.d.'s 0–7°)

Ring system	Substituent	On atom	Orientation	$\alpha$ (°)
Ring A	C(5)	C(4a)	Equatorial	21.4
	C(14)	C(14a)	Axial	76.4
Ring B	C(4)	C(4a)	Axial	60.6
	C(6)	C(5a)	Equatorial	–1.0
	C(13)	C(13a)	Inclinal	56.2
	C(1)	C(14a)	Equatorial	1.3
Ring C	C(5)	C(5a)	Axial	70.7
	O(23)	C(6)	Equatorial	22.3
	C(7)	C(6a)	Equatorial	–17.3
	C(12)	C(12a)	Axial	–80.3
	O(27)	C(13)	Axial	86.1
	C(14)	C(13a)	Equatorial	18.6
Ring D	C(6)	C(6a)	Axial	–76.7
	C(8)	C(7a)	Axial	–70.6
	C(11)	C(11a)	Equatorial	–15.6
	C(13)	C(12a)	Equatorial	–13.5
Ring E	C(7)	C(7a)	Equatorial	–17.5
	C(12)	C(11a)	Axial	–73.2

Table 6. Ring-puckering coordinates of the rings A–E

For a definition and the way they are calculated see Cremer & Pople (1975) and Cremer (1975).

Ring	$q_2$ (Å)	$q_3$ (Å)	$Q$ (Å)	$\theta$ (°)	$\phi$ (°)
A	0.056	–0.547	0.550	174.2	273.3
B	0.733	0.012	0.733	89.10	288.5
C	0.035	0.515	0.516	3.94	138.3
D	0.076	–0.539	0.544	172.0	81.4
E	0.078	0.538	0.544	8.3	133.8
Ideal chair	0	0.560	0.560	0 180	–
Ideal boat	0.7	0	0.7	90	$n\pi/3$

C(5)···O(27) in ring C. These and similar interactions can best be seen from Table 5, which gives a summary of the positions of the substituents on the individual rings in terms of the polar angle  $\alpha$ ;  $\alpha$  is the angle between a substituent and the mean plane of the ring

(Cremer & Pople, 1975). Substituents for which  $\alpha > 0$  are above, those for which  $\alpha < 0$  are below the plane of the ring. An orientation is considered equatorial when  $0 \leq |\alpha| \leq 30^\circ$ , inclinal when  $30 < |\alpha| < 60^\circ$  and axial when  $60 \leq |\alpha| \leq 90^\circ$ . Other meaningful observables of ring geometries are the ring-puckering coordinates listed in Table 6. In comparison with an unsubstituted cyclohexane ring, the values found for the title compound show that rings A–E are rather distorted.

Finally, the value  $\phi(\text{ring B}) = 288.5^\circ$  indicates that ring B is in a twist form nearest to a true boat with C(5a) and C(14a) as bowsprits.

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