

3842 are independent ($R_{\text{merge}} = 0.018$). Standard reflection (052) checked every 50 reflections: no significant deviation. Structure solved by direct methods using *SHELXS86* (Sheldrick, 1985). H atoms from difference Fourier synthesis. Anisotropic least-squares refinement (*SHELX76*; Sheldrick, 1976) using *F*; H atoms isotropic with common refined temperature factor ($B = 5.2 \text{ \AA}^2$). $w = 1/[\sigma^2(F) + 0.0298F^2]$, $R = 0.047$, $wR = 0.055$, $S = 0.42$ for 3842 observed reflections. Final maximum shift/e.s.d. = 0.08 (non-H atoms). Maximum and minimum heights in final difference Fourier synthesis 0.27 and -0.38 e \AA^{-3} . Atomic scattering factors from *International Tables for X-ray Crystallography*, (1974, Vol. IV). The atomic parameters are given in Table 1.* Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

Related literature. Numerous friedelanes and friedelane-like structures have been studied by X-ray

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53341 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (\pm)-(3*aR**,5*S**,6*R**,7*aS**)-2,2-Dimethyl-2,3,3*a*,4,5,6,7,7*a*-octahydroindene-5,6-diol Bis(*p*-bromobenzoate)

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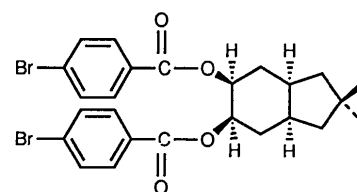
Abstract. (3*aR**,5*S**,6*R**,7*aS**)-2,2-Dimethylperhydroindene-5,6-diyl bis(*p*-bromobenzoate), $C_{25}H_{26}Br_2O_4$, $M_r = 550.30$, monoclinic, $P2_1/c$, $a = 10.643$ (2), $b = 21.927$ (6), $c = 11.423$ (3) \AA , $\beta = 113.87$ (3) $^\circ$, $V = 2438$ (2) \AA^3 , $Z = 4$, $D_x = 1.50 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$, $\mu = 33.2 \text{ cm}^{-1}$, $F(000) = 1112$, $T = 298 \text{ K}$, $R = 0.065$ for 1614 reflections with intensities $I \geq 2.0\sigma(I)$. The carbinol and the ring-juncture hydrogen substituents are all found to be *cis* with respect to the six-membered ring.

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diffraction. A complete list of references is given by Mo *et al.* (1989). Friedelin, obtained from *H. madagascariensis*, was further investigated by spectrometric methods (IR; MS; 500 MHz ^1H NMR; ^{13}C NMR; Prakash, Roy, Garg & Bhakuni, 1987). The compound, crystallized from CHCl_3 , had a melting point (541 K) in agreement with the one reported by Corey & Ursprung (1956).

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(I)

Table 1. Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Br(1)	482 (1)	805 (1)	3918 (1)	83 (1)
Br(2)	5996 (1)	4341 (1)	5312 (1)	104 (1)
C(1)	2340 (8)	685 (4)	4894 (8)	51 (5)
C(2)	3325 (8)	1082 (4)	4840 (7)	57 (5)
C(3)	4714 (7)	975 (4)	5588 (7)	56 (4)
C(4)	5120 (8)	470 (4)	6376 (7)	43 (4)
C(5)	4120 (9)	80 (4)	6404 (8)	58 (5)
C(6)	2744 (9)	185 (4)	5654 (9)	57 (5)
C(7)	6595 (8)	348 (3)	7175 (7)	50 (4)
O(1)	6999 (5)	-92 (3)	7847 (5)	83 (3)
O(2)	7418 (4)	791 (2)	7076 (5)	58 (3)
C(8)	8877 (7)	709 (4)	7812 (6)	51 (4)
C(9)	9346 (7)	1025 (4)	9072 (6)	54 (4)
C(10)	10894 (7)	961 (4)	9862 (7)	54 (4)
C(11)	11449 (8)	1388 (4)	11041 (7)	70 (5)
C(12)	12143 (9)	1935 (4)	10694 (8)	60 (5)
C(13)	13662 (12)	1935 (7)	11543 (11)	117 (7)
C(14)	11461 (14)	2531 (4)	10817 (11)	115 (8)
C(15)	11894 (8)	1826 (3)	9286 (7)	56 (4)
C(16)	11740 (8)	1137 (4)	9097 (7)	58 (5)
C(17)	11145 (6)	919 (4)	7698 (6)	56 (4)
C(18)	9598 (7)	967 (3)	7008 (7)	50 (4)
O(4)	9165 (5)	1602 (2)	6731 (4)	48 (3)
O(3)	7795 (6)	1391 (2)	4671 (5)	72 (3)
C(19)	8192 (7)	1732 (4)	5559 (7)	47 (4)
C(20)	7675 (8)	2366 (4)	5556 (9)	44 (5)
C(21)	6768 (9)	2606 (4)	4366 (9)	59 (5)
C(22)	6263 (9)	3188 (5)	4314 (8)	70 (5)
C(23)	6648 (9)	3522 (4)	5393 (9)	57 (5)
C(24)	7505 (8)	3303 (4)	6568 (8)	57 (5)
C(25)	8020 (8)	2724 (4)	6639 (8)	56 (5)

Table 2. Bond lengths (\AA) and angles ($^\circ$)

Br(1)—C(1)	1.855 (8)	Br(2)—C(23)	1.914 (8)
C(1)—C(2)	1.383 (13)	C(1)—C(6)	1.355 (12)
C(2)—C(3)	1.396 (10)	C(3)—C(4)	1.382 (10)
C(4)—C(5)	1.377 (13)	C(4)—C(7)	1.487 (10)
C(5)—C(6)	1.384 (11)	C(7)—O(1)	1.198 (9)
C(7)—O(2)	1.343 (10)	O(2)—C(8)	1.447 (7)
C(8)—C(9)	1.491 (10)	C(8)—C(18)	1.524 (12)
C(9)—C(10)	1.531 (9)	C(10)—C(11)	1.547 (10)
C(10)—C(16)	1.537 (14)	C(11)—C(12)	1.542 (13)
C(12)—C(13)	1.511 (13)	C(12)—C(14)	1.528 (15)
C(12)—C(15)	1.539 (12)	C(15)—C(16)	1.527 (11)
C(16)—C(17)	1.538 (10)	C(17)—C(18)	1.514 (9)
C(18)—O(4)	1.461 (8)	O(4)—C(19)	1.351 (8)
O(3)—C(19)	1.192 (9)	C(19)—C(20)	1.495 (12)
C(20)—C(21)	1.412 (12)	C(20)—C(25)	1.383 (13)
C(21)—C(22)	1.377 (14)	C(22)—C(23)	1.347 (13)
C(23)—C(24)	1.370 (11)	C(24)—C(25)	1.371 (11)
Br(1)—C(1)—C(2)	121.8 (6)	Br(1)—C(1)—C(6)	119.0 (7)
C(2)—C(1)—C(6)	119.2 (7)	C(1)—C(2)—C(3)	120.1 (7)
C(2)—C(3)—C(4)	120.5 (8)	C(3)—C(4)—C(5)	118.2 (7)
C(3)—C(4)—C(7)	121.2 (7)	C(5)—C(4)—C(7)	120.5 (7)
C(4)—C(5)—C(6)	121.0 (8)	C(1)—C(6)—C(5)	120.9 (9)
C(4)—C(7)—O(1)	123.6 (7)	C(4)—C(7)—O(2)	112.3 (6)
O(1)—C(7)—O(2)	124.0 (6)	C(7)—O(2)—C(8)	116.3 (5)
O(2)—C(8)—C(9)	111.3 (6)	O(2)—C(8)—C(18)	106.7 (5)
C(9)—C(8)—C(18)	110.8 (6)	C(8)—C(9)—C(10)	112.7 (7)
C(9)—C(10)—C(11)	113.5 (7)	C(9)—C(10)—C(16)	112.3 (6)
C(11)—C(10)—C(16)	103.7 (6)	C(10)—C(11)—C(12)	107.5 (7)
C(11)—C(12)—C(13)	109.5 (8)	C(11)—C(12)—C(14)	110.3 (9)
C(13)—C(12)—C(14)	111.5 (8)	C(11)—C(12)—C(15)	104.5 (6)
C(13)—C(12)—C(15)	111.0 (9)	C(14)—C(12)—C(15)	109.9 (7)
C(12)—C(15)—C(16)	105.3 (6)	C(10)—C(16)—C(15)	102.8 (7)
C(10)—C(16)—C(17)	115.1 (6)	C(15)—C(16)—C(17)	115.4 (6)
C(16)—C(17)—C(18)	115.1 (7)	C(8)—C(18)—C(17)	111.6 (6)
C(8)—C(18)—O(4)	107.1 (6)	C(17)—C(18)—O(4)	111.1 (6)
C(18)—O(4)—C(19)	117.7 (5)	O(4)—C(19)—O(3)	125.2 (7)
O(4)—C(19)—C(20)	109.4 (6)	O(3)—C(19)—C(20)	125.4 (6)
C(19)—C(20)—C(21)	117.1 (8)	C(19)—C(20)—C(25)	124.2 (7)
C(21)—C(20)—C(25)	118.7 (8)	C(20)—C(21)—C(22)	119.3 (9)
C(21)—C(22)—C(23)	119.8 (8)	Br(2)—C(23)—C(22)	119.8 (6)
Br(2)—C(23)—C(24)	117.5 (7)	C(22)—C(23)—C(24)	122.7 (8)
C(23)—C(24)—C(25)	118.3 (8)	C(20)—C(25)—C(24)	121.1 (7)

from hot hexane solutions by cooling to room temperature and keeping the mixture at 278 K in a refrigerator overnight. A crystal measuring approximately $0.52 \times 0.43 \times 0.06$ mm was used for data collection using Mo $K\alpha$ radiation on a Nicolet R3m/ μ diffractometer equipped with a graphite monochromator. Cell dimensions were obtained from a least-squares refinement of the setting angles of 15 reflections with 2θ values between 18 and 23° . Intensity data were collected by the $\theta/2\theta$ scan technique between 5 and 42° in 2θ at variable scan speeds between 4 and $29.3^\circ \text{ min}^{-1}$ depending on intensity; range in hkl : $h, -10 \rightarrow 10$; $k, 0 \rightarrow 22$; $l, 0 \rightarrow 11$. Stationary backgrounds were measured for half of scan time on each side of a peak; two standards measured after every 48 reflections showed an average variation of 3.6%. 2886 reflections were measured which yielded 1614 reflections with intensities $I \geq 2\sigma(I)$; $R_{int} = 0.014$. The intensities were corrected for background, Lorentz and polarization effects, and absorption (empirical ψ -scan method, maximum and minimum transmission factors 0.47 and 0.35, respectively).

The structure was solved by Patterson and difference Fourier techniques and refined by blocked-cascade least squares (Sparks, 1961). The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/(\sigma_F^2 + 0.0001 F^2)$. The non-H atoms

were refined with anisotropic thermal parameters. The H atoms were placed in theoretical positions and were refined in the riding mode with fixed isotropic thermal parameters 1.2 times larger than those of the attached C atoms. The final $R = 0.065$, $wR = 0.040$, goodness-of-fit parameter $S = 1.50$, maximum $\Delta\rho = 0.08$, maximum and minimum difference electron density in the final ΔF map $\pm 0.42 \text{ e \AA}^{-3}$. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71–102). Computations were performed on a Data General Microclipse Desktop computer using *SHELXTL* (Sheldrick, 1985). Fractional coordinates for non-H atoms are given in Table 1, bond distances and angles not involving hydrogens in Table 2 and selected torsion angles in Table 3.* The stereo-

* Lists of anisotropic thermal parameters, H-atom coordinates and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53287 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

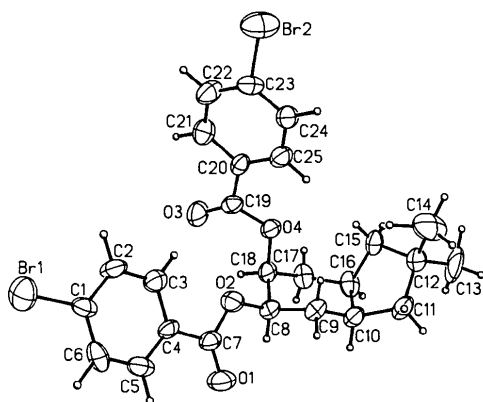


Fig. 1. View of the molecule (40% thermal ellipsoids) showing the atom-numbering scheme, the *cis*-fused hydrindane ring system and the *cis* orientation with respect to the six-membered ring of the four hydrogen substituents on C(8), C(10), C(16) and C(18), respectively.

chemistry of the molecule showing the *cis*-fused hydrindane ring skeleton and the orientations of the four hydrogens shown in (I) is depicted in Fig. 1.

Related literature. The stereochemistry observed here is similar to that reported for the hydrindane skele-

Table 3. Selected torsion angles ($^{\circ}$)

C(3)—C(4)—C(7)—O(2)	3 (1)	C(25)—C(20)—C(19)—O(4)	-8 (1)
C(7)—O(2)—C(8)—C(18)	144.2 (7)	C(19)—O(4)—C(18)—C(8)	-101.5 (7)
O(2)—C(8)—C(9)—C(10)	-178.5 (6)	O(4)—C(18)—C(17)—C(16)	72.0 (8)
C(8)—C(9)—C(10)—C(11)	168.4 (7)	C(18)—C(17)—C(16)—C(15)	-79.8 (9)
C(8)—C(9)—C(10)—C(16)	51.1 (9)	C(18)—C(17)—C(16)—C(10)	39.8 (9)
C(9)—C(10)—C(11)—C(12)	-101.2 (8)	C(17)—C(16)—C(15)—C(12)	166.0 (7)
C(10)—C(11)—C(12)—C(14)	121.3 (7)	C(16)—C(15)—C(12)—C(13)	91.4 (9)

ton of the sesquiterpenoids illudol and marasmic acid (Cradwick & Sim, 1971).

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The Structure of Urea–Oxalic Acid (2/1) Determined by Neutron Diffraction at 100 K*

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Abstract. $2\text{CH}_4\text{N}_2\text{O}\cdot\text{C}_2\text{H}_2\text{O}_4$, $M_r = 210.15$, monoclinic, $P2_1/c$, $a = 5.048$ (1), $b = 12.374$ (3), $c = 6.876$ (2) Å, $\beta = 96.40$ (2) $^{\circ}$, $V = 426.8$ (1) Å 3 , $Z = 2$, $D_x = 1.63$ Mg m $^{-3}$, $\lambda = 1.296$ Å, $\mu = 0.231$ mm $^{-1}$, $T = 100.0$ (1) K, $R = 0.045$, $wR = 0.035$ for 895 unique observed reflections with $I > 2\sigma(I)$. The crystal structure contains urea and oxalic acid molecules in a hydrogen-bonded network, complex-

ing in a 2:1 ratio. The determination is in agreement with previous X-ray work [Harkema, Bats, Weyenburg & Feil (1972). *Acta Cryst.* **B28**, 1646–1648] and provides additional information on the H atoms.

Experimental. A solution containing urea and oxalic acid in the correct stoichiometric ratio was slowly evaporated at room temperature, resulting in crystal-line plates of a few millimeters in thickness and an area of several millimeters in the other dimensions. A flat triangular crystal with edges of length *ca* 2.5, 4.2

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