Table 4. *Bond distances (Å), angles (°), and selected torsion angles (°) for (2)*

O(1)—C(7)	1·410 (1)	C(3)—C(4)	1·513 (2)
O(1)—C(10)	1·420 (1)	C(4)—C(5)	1·564 (1)
O(2)—C(7)	1·411 (1)	C(4)—C(7)	1·540 (1)
O(2)—C(11)	1·416 (1)	C(5)—C(6)	1·575 (1)
C(1)—C(2)	1·510 (1)	C(5)—C(9)	1·526 (1)
C(1)—C(6)	1·566 (1)	C(6)—C(8)	1·530 (2)
C(1)—C(7)	1·546 (1)	C(8)—C(9)	1·544 (1)
C(2)—C(3)	1·329 (1)		

C(7)—O(1)—C(10)	114·51 (7)	C(6)—C(5)—C(9)	118·16 (8)
C(7)—O(2)—C(11)	114·07 (7)	C(1)—C(6)—C(5)	102·81 (6)
C(2)—C(1)—C(6)	107·16 (9)	C(1)—C(6)—C(8)	112·43 (8)
C(2)—C(1)—C(7)	99·50 (7)	C(5)—C(6)—C(8)	117·59 (8)
C(6)—C(1)—C(7)	100·17 (7)	O(1)—C(7)—O(2)	109·94 (9)
C(1)—C(2)—C(3)	107·49 (9)	O(1)—C(7)—C(1)	108·94 (6)
C(2)—C(3)—C(4)	108·19 (8)	O(1)—C(7)—C(4)	116·30 (8)
C(3)—C(4)—C(5)	106·94 (9)	O(2)—C(7)—C(1)	117·44 (8)
C(3)—C(4)—C(7)	99·42 (7)	O(2)—C(7)—C(4)	109·81 (6)
C(5)—C(4)—C(7)	100·54 (6)	C(1)—C(7)—C(4)	93·88 (8)
C(4)—C(5)—C(6)	102·30 (7)	C(6)—C(8)—C(9)	114·39 (9)
C(4)—C(5)—C(9)	111·53 (7)	C(5)—C(9)—C(8)	115·83 (8)

C(10)—O(1)—C(7)—O(2)	−60·08 (11)	C(11)—O(2)—C(7)—O(1)	−68·69 (11)
C(7)—C(1)—C(2)—C(3)	34·32 (11)	C(7)—C(1)—C(6)—C(5)	−36·36 (9)
C(1)—C(2)—C(3)—C(4)	−0·81 (12)	C(7)—C(4)—C(5)—C(9)	164·32 (7)
C(4)—C(5)—C(6)—C(1)	−0·30 (9)	C(9)—C(5)—C(6)—C(8)	0·89 (13)
C(6)—C(5)—C(9)—C(8')	−77·93 (10)	C(6)—C(8)—C(9')—C(5')	−117·83 (9)
C(5)—C(6)—C(8)—C(9')	73·37 (11)		

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Redetermination of the Structure of Friedelin

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Abstract. D:A-Friedooleanan-3-one, $C_{30}H_{50}O$, $M_r = 426·73$, orthorhombic, $P2_12_12_1$, $a = 6·362$ (2), $b = 13·923$ (2), $c = 28·419$ (5) Å, $V = 2517$ (1) Å^3, $Z = 4$, $D_x = 1·13$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·5417$ Å, $\mu = 4·92$ cm^{-1}, $F(000) = 952$, $T = 291$ K, $R = 0·047$ for 3842 observed reflections. Friedelin was isolated for the first time from *Harungana madagascariensis* Lam. ex Poir (Clusiaceae). Recently, the crystal structure has been described [Mo, Winther & Scrimgeour (1989). *Acta Cryst.* B45, 261–270], but with relatively low precision ($R = 0·133$ for 2201 reflections). Our data are of much better quality: mean e.s.d.'s of bond distances and angles are 0·003 Å and 0·2° here, com-
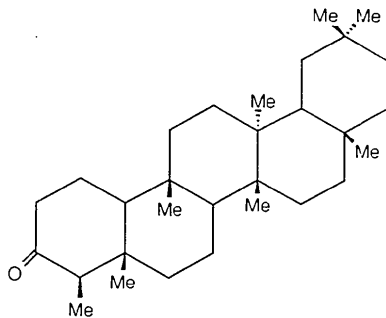
* Research Director of the Belgian 'National Fund for Scientific Research'.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å²)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i{}^*a_j{}^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
C1	2206 (4)	2781 (1)	3997 (1)	3·27 (3)
C2	649 (4)	2969 (2)	4394 (1)	3·83 (4)
C3	485 (3)	2105 (2)	4709 (1)	3·23 (3)
C4	-107 (4)	1183 (2)	4453 (1)	3·17 (3)
C5	1601 (3)	960 (1)	4074 (1)	2·90 (3)
C6	928 (4)	76 (2)	3788 (1)	3·32 (3)
C7	2179 (4)	-78 (1)	3337 (1)	3·38 (3)
C8	2041 (3)	808 (1)	3017 (1)	2·70 (3)
C9	3001 (3)	1704 (1)	3270 (1)	2·63 (3)
C10	1691 (3)	1847 (1)	3732 (1)	2·53 (3)
C11	2662 (4)	2580 (1)	2949 (1)	3·15 (3)
C12	3411 (4)	2425 (1)	2443 (1)	3·08 (3)
C13	2333 (3)	1570 (1)	2197 (1)	2·60 (3)
C14	2714 (3)	638 (1)	2496 (1)	2·79 (3)
C15	1392 (4)	-194 (2)	2283 (1)	3·68 (4)
C16	977 (5)	-125 (2)	1746 (1)	4·32 (4)
C17	2517 (4)	471 (2)	1442 (1)	3·55 (3)
C18	3261 (3)	1430 (1)	1693 (1)	2·88 (3)
C19	2890 (4)	2322 (2)	1382 (1)	3·60 (4)
C20	3648 (4)	2248 (2)	870 (1)	3·95 (4)
C21	2751 (7)	1319 (2)	650 (1)	5·27 (5)
C22	1387 (5)	741 (2)	980 (1)	4·66 (5)
C23	-548 (6)	370 (2)	4800 (1)	4·72 (5)
C24	3694 (4)	752 (2)	4326 (1)	3·84 (4)
C25	5384 (4)	1648 (2)	3376 (1)	3·71 (4)
C26	-52 (4)	1819 (2)	2168 (1)	3·39 (3)
C27	5050 (4)	323 (2)	2484 (1)	3·56 (3)
C28	4380 (5)	-176 (2)	1306 (1)	4·70 (5)
C29	2839 (7)	3126 (2)	610 (1)	5·83 (6)
C30	6067 (6)	2274 (3)	858 (1)	6·31 (7)
O	876 (3)	2133 (1)	5126 (1)	4·15 (3)

Table 2. *Bond distances* (Å) *and angles* (°)

C2—C1	1·522 (3)	C10—C1	1·538 (3)
C3—C2	1·503 (3)	C4—C3	1·523 (3)
O—C3	1·213 (2)	C5—C4	1·561 (3)
C23—C4	1·526 (3)	C6—C5	1·536 (3)
C10—C5	1·573 (3)	C24—C5	1·539 (3)
C7—C6	1·525 (3)	C8—C7	1·534 (3)
C9—C8	1·564 (3)	C14—C8	1·560 (3)
C10—C9	1·569 (3)	C11—C9	1·539 (3)
C25—C9	1·547 (3)	C12—C11	1·529 (3)
C13—C12	1·541 (3)	C14—C13	1·570 (2)
C18—C13	1·560 (3)	C26—C13	1·559 (3)
C15—C14	1·554 (3)	C27—C14	1·550 (3)
C16—C15	1·551 (3)	C17—C16	1·548 (4)
C18—C17	1·587 (3)	C22—C17	1·544 (4)
C28—C17	1·538 (4)	C19—C18	1·543 (3)
C20—C19	1·536 (3)	C21—C20	1·546 (4)
C29—C20	1·519 (4)	C30—C20	1·539 (5)
C22—C21	1·511 (4)		

C10—C1—C2	111·6 (2)	C3—C2—C1	110·4 (2)
C4—C3—C2	114·0 (2)	O—C3—C2	122·9 (2)
O—C3—C4	122·9 (2)	C5—C4—C3	108·9 (2)
C23—C4—C3	111·2 (2)	C23—C4—C5	115·2 (2)
C6—C5—C4	109·3 (2)	C10—C5—C4	107·2 (2)
C10—C5—C6	108·2 (1)	C24—C5—C4	108·6 (2)
C24—C5—C6	109·7 (2)	C24—C5—C10	113·8 (2)
C7—C6—C5	114·3 (2)	C8—C7—C6	110·8 (2)
C9—C8—C7	110·3 (2)	C14—C8—C7	115·2 (1)
C14—C8—C9	116·8 (1)	C10—C9—C8	106·2 (1)
C11—C9—C8	107·7 (1)	C11—C9—C10	108·8 (1)
C25—C9—C8	115·6 (2)	C25—C9—C10	111·3 (2)
C25—C9—C11	107·0 (2)	C5—C10—C1	111·7 (1)
C9—C10—C1	113·9 (2)	C9—C10—C5	115·9 (1)
C12—C11—C9	113·7 (2)	C13—C12—C11	113·4 (2)
C14—C13—C12	108·9 (1)	C18—C13—C12	110·2 (2)
C18—C13—C14	109·5 (1)	C26—C13—C12	106·6 (2)
C26—C13—C14	111·2 (2)	C26—C13—C18	110·4 (2)
C13—C14—C8	110·3 (1)	C15—C14—C8	109·5 (2)
C15—C14—C13	108·8 (2)	C27—C14—C8	109·1 (2)
C27—C14—C13	111·7 (2)	C27—C14—C15	107·4 (2)
C16—C15—C14	115·4 (2)	C17—C16—C15	118·3 (2)
C18—C17—C16	112·8 (2)	C22—C17—C16	108·1 (2)
C22—C17—C18	108·5 (2)	C28—C17—C16	108·3 (2)
C28—C17—C18	112·1 (2)	C28—C17—C22	106·8 (2)
C17—C18—C13	113·9 (2)	C19—C18—C13	111·5 (2)
C19—C18—C17	111·9 (2)	C20—C19—C18	116·2 (2)
C21—C20—C19	108·9 (2)	C29—C20—C19	107·5 (2)
C29—C20—C21	110·5 (2)	C30—C20—C19	109·5 (2)
C30—C20—C21	112·3 (3)	C30—C20—C29	108·0 (3)
C22—C21—C20	113·9 (2)	C21—C22—C17	113·0 (3)

pared with 0·009 Å and 0·5° in the earlier study. A least-squares molecular fit was also computed between our results (I), the previous X-ray results (II) and calculated 'force-field' coordinates (III). The final root-mean-square deviations, excluding H atoms, are: (I)–(II): r.m.s. = 0·023 Å, (I)–(III): r.m.s. = 0·097 Å, (II)–(III): r.m.s. = 0·096 Å. Differences between the observed and the calculated coordinates are not due to the precision of the crystal structure determinations.

Experimental. Crystals, suitable for the X-ray crystallographic analysis, were obtained by very slow evaporation (over a period of two weeks) of a chloroform solution.

From the determination of the unit-cell parameters, use of the Cambridge Structural Database (Allen, Bellard, Brice, Cartwright, Doubleday,
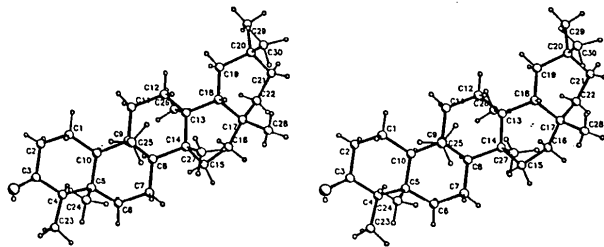
Fig. 1. Stereoscopic view of the molecule and atom numbering.

Higgs, Hummelink, Hummelink-Peters, Kennard, Motherwell, Rodgers & Watson, 1979) identified the molecule as friedelin (Mo, Winther & Scrimgeour, 1982). Parallelepiped crystal with dimensions 0·2 × 0·2 × 0·4 mm. Lattice parameters refined using 16 reflections in the range $7 \le 2\theta \le 50°$. Huber four-circle diffractometer, ω-2θ scan, graphite-monochromated Cu $K\alpha$ radiation. 5175 $hk\pm l$ reflections $(0 \le h \le 7, \quad 0 \le k \le 16, \quad -34 \le l \le 34)$ with $\sin\theta/\lambda \le 0.60$ Å^{-1}, 4271 with $I \ge 2.5\sigma(I)$, of which

3842 are independent ($R_{merge} = 0.018$). Standard reflection (052) checked every 50 reflections: no significant deviation. Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1985). H atoms from difference Fourier synthesis. Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976) using *F*; H atoms isotropic with common refined temperature factor ($B = 5.2$ Å^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 Å^{-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

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* 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.

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 ^{1}H 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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Structure of (±)-(3aR*,5S*,6R*,7aS*)-2,2-Dimethyl-2,3,3a,4,5,6,7,7a-octahydroindene-5,6-diol Bis(p-bromobenzoate)

BY P. SINGH,* SAMUEL G. LEVINE* AND NINA E. HEARD

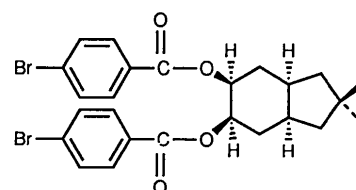*Department of Chemistry, North Carolina State University, Raleigh, NC 27695-8204, USA*

Abstract. (3aR*,5S*,6R*,7aS*)-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) Å, $\beta = 113.87$ (3)°, $V = 2438$ (2) Å^3, $Z = 4$, $D_x = 1.50$ g cm^{-3}, λ(Mo Kα) = 0.71069 Å, $\mu = 33.2$ cm^{-1}, $F(000) = 1112$, $T = 298$ 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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Experimental. The title compound (I) was synthesized and purified as a single isomer as reported elsewhere (Heard, 1988). Colorless crystals suitable for X-ray analysis were grown as rhombic plates

(I)