$C_{22}H_{25}F_2N_3O_2S$

C15A—N14A—C19A	109.1 (2)		C15B—N14B—C19B	108.9 (2)
N14A—C15A—C16A	111.1 (2)		N14B—C15B—C16B	111.2 (2)
C15A—C16A—C17A	111.9 (2)		C15B—C16B—C17B	112.2 (2)
C16A—C17A—N20A	112.4 (2)		C16B—C17B—N20B	112.2 (2)
C16A—C17A—C18A	110.5 (2)		C16B—C17B—C18B	110.3 (2)
C18A—C17A—N20A	112.9 (2)		C18B—C17B—N20B	113.2 (2)
C17A—C18A—C19A	110.7 (2)		C17B—C18B—C19B	110.6 (2)
N14A—C19A—C18A	109.3 (2)		N14B—C19B—C18B	110.1 (2)
C17A—N20A—C22A	119.9 (2)		C17B—N20B—C22B	118.7 (2)
C17A—N20A—C21A	119.0 (2)		C17B—N20B—C21B	120.8 (2)
C21A—N20A—C22A	119.9 (2)		C21B—N20B—C22B	118.8 (2)
N20A—C22A—S26A	118.9 (2)		N20B—C22B—S26B	118.8 (2)
N20A—C22A—N23A	125.0 (2)		N20B—C22B—N23B	124.5 (2)
N23A—C22A—S26A	116.1 (2)		N23B—C22B—S26B	116.7 (2)
C22A—N23A—C24A	110.2 (2)		C22B—N23B—C24B	109.4 (2)
N23A—C24A—C27A	125.3 (2)		N23B—C24B—C27B	124.6 (2)
N23A—C24A—C25A	115.5 (2)		N23B—C24B—C25B	115.4 (2)
C25A—C24A—C27A	119.2 (2)		C25B—C24B—C27B	119.9 (2)
C24A—C25A—C30A	121.0 (2)		C24B—C25B—C30B	120.6 (2)
C24A—C25A—S26A	110.1 (2)		C24B—C25B—S26B	110.1 (2)
S26A—C25A—C30A	128.9 (2)		S26B—C25B—C30B	129.3 (2)
C22A—S26A—C25A	88.0 (1)		C22B—S26B—C25B	88.4 (1)
C24A—C27A—C28A	119.7 (2)		C24B—C27B—C28B	119.2 (2)
C27A—C28A—C29A	121.2 (2)		C27B—C28B—C29B	121.0 (2)
C28A—C29A—C30A	121.2 (3)		C28B—C29B—C30B	121.6 (3)
C25A—C30A—C29A	117.7 (2)		C25B—C30B—C29B	117.7 (2)

C6A1—O9A—C10A—C11A	167.8 (2)
C6A2—O9A—C10A—C11A	168.1 (4)
O9A—C10A—C11A—O12A	−63.7 (2)
O9A—C10A—C11A—C13A	61.3 (3)
C10A—C11A—C13A—N14A	−172.1 (2)
O12A—C11A—C13A—N14A	−47.3 (2)
C6B—O9B—C10B—C11B	−169.0 (2)
O9B—C10B—C11B—O12B	77.3 (2)
O9B—C10B—C11B—C13B	−162.1 (2)
C10B—C11B—C13B—N14B	−168.5 (2)
O12B—C11B—C13B—N14B	−48.1 (2)

Molecule A shows rotational disorder of the difluorophenyl moiety. The atoms were split and refined using the SAME, DELU and SIMU restraint facilities of SHELXL93 (Sheldrick, 1993). The sum of the occupancy factors was constrained to 1. The occupancy factor for atoms C1A1–F8A1 refined to 0.732 (2). H atoms were calculated in geometrical positions and allowed to ride on their parent atom.

Data collection: XSCANS (Siemens, 1993). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: DIRDIF (Beurskens et al., 1992). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEX2.1 (McArdle, 1994). Software used to prepare material for publication: PARST (Nardelli, 1983).
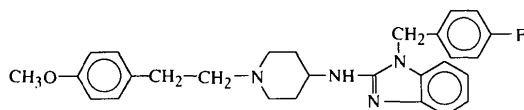
Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1175). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-[(4-Fluorophenyl)methyl]-N-{1-[2-(4-methoxyphenyl)ethyl]-4-piperidyl}-1H-benzimidazol-2-amine (Astemizole)†

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Abstract

Astemizole, $C_{28}H_{31}FN_4O$, is a non-sedating H_1 antihistamine. The crystal structure contains two molecules in the asymmetric unit related by a pseudo centre of symmetry. The conformations of the methoxyphenylethyl side chains are different. N—H···N intermolecular hydrogen bonds link the molecules into infinite chains in the c direction of the Cc space group.

Comment

Astemizole, (I), was developed from a series of structurally novel antihistamines. The oral antiallergic activity of astemizole in laboratory animals was found to be expressed at low doses of the order of 0.1 mg kg^{-1} and to be of long duration. Tight binding of astemizole (and a major metabolite, desmethylastemizole) to H_1 receptors and little penetration into the brain sus-

† Internal code of the Janssen Research Foundation: R43512.

tain the high clinical effectiveness of astemizole, in the virtual absence of sedation (Richards, Brogden, Heel, Speight & Avery, 1984).

(I)

Fig. 1 illustrates the two molecules in the asymmetric unit. They are related by a pseudo inversion centre near 0.575, 1/2, 0.275. The greatest deviations are at the atomic positions of the methoxyphenylethyl moiety, mainly at C26 and C34. The difference in conformation can be deduced from the torsion angles C21—N22—C25—C26 [−66.7 (6), 160.0 (6)°], C25—C26—C27—C28 [64.3 (7), −177.8 (6)°] and C29—C30—O33—C34 [177.7 (7), −4 (1)°]. The eclipsed position of C25B versus C32B forces the angles C25—C26—C27 and C26—C27—C32 to open from 111.8 (5) and 121.4 (5)°, respectively, in molecule A to 117.3 (5) and 125.1 (6)°, respectively, in molecule B. No explanation has been found for the abnormal short distance of 1.458 (9) Å for C25B—C26B.

In the piperidyl rings of both molecules, the N—C bond distances are significantly different [C21A—N22A 1.475 (8), N22A—C23A 1.436 (7), C21B—N22B 1.424 (9), N22B—C23B 1.464 (8) Å]. The shorter bonds are antiperiplanar with respect to the C25—C26 bonds. This suggests that there is some delocalization of the N-atom lone pair into the antiperiplanar N—C bonds. Other bond lengths and angles are within the expected ranges.
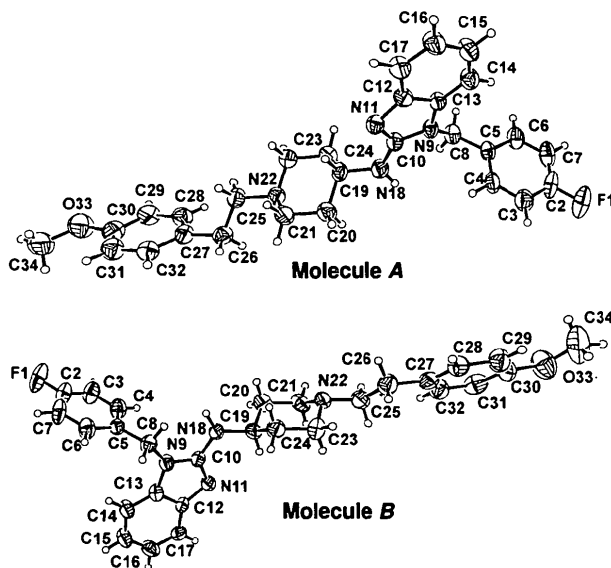

Molecule A

Molecule B

Fig. 1. Perspective views of the molecules of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

Both molecules in the asymmetric unit are linked to c-glide equivalent molecules by N—H· · ·N hydrogen bonds, forming endless chains in the c direction [N18A· · ·N11A^i 3.069 (7), H18A· · ·N11A^i 2.23 Å, N18A—H18A· · ·N11A^i 166°, N18B· · ·N11B^{ii} 3.076 (7), H18B· · ·N11B^{ii} 2.22 Å, N18B—H18B· · ·N11B^{ii} 172°; symmetry codes: (i) $x, -y - 2, z + \frac{1}{2}$, (ii) $x, -y, z - \frac{1}{2}$].

Experimental

Crystal data

$C_{28}H_{31}FN_4O$	Cu $K\alpha$ radiation
$M_r = 458.57$	$\lambda = 1.54184$ Å
Monoclinic	Cell parameters from 24
Cc	reflections
$a = 49.34 (1)$ Å	$\theta = 23–29°$
$b = 10.675 (2)$ Å	$\mu = 0.660$ mm^{-1}
$c = 9.372 (2)$ Å	$T = 293$ K
$\beta = 98.22 (2)°$	Prism
$V = 4885 (2)$ Å^3	$0.40 \times 0.10 \times 0.10$ mm
$Z = 8$	Colourless
$D_x = 1.247$ Mg m^{-3}	

Data collection

Syntex $P2_1$ four-circle	$\theta_{max} = 55.08°$
diffractometer	$h = 0 \rightarrow 52$
$\omega/2\theta$ scans	$k = 0 \rightarrow 11$
Absorption correction:	$l = -9 \rightarrow 9$
none	3 standard reflections
6618 measured reflections	monitored every 50
3098 independent reflections	reflections
2421 observed reflections	intensity decay: 5.0%
$[F^2 > 3\sigma(F^2)]$	

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.22$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.0396$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
$wR(F^2) = 0.1065$	Extinction correction:
$S = 1.067$	SHELXL93 (Sheldrick,
3098 reflections	1993)
616 parameters	Extinction coefficient:
H-atom parameters not	0.00040 (7)
refined	Atomic scattering factors
$w = 1/[\sigma^2(F_o^2) + (0.0835P)^2$	from International Tables
$+ 0.9343P]$	for X-ray Crystallography
where $P = (F_o^2 + 2F_c^2)/3$	(1974, Vol. IV, Tables
$(\Delta/\sigma)_{max} < 0.001$	2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
F1A	0.83585 (9)	−1.0221 (6)	0.9442 (6)	0.131 (3)
C2A	0.8120 (1)	−1.060 (1)	0.8660 (8)	0.086 (3)
C3A	0.7994 (1)	−0.9826 (8)	0.7612 (9)	0.082 (3)
C4A	0.7750 (1)	−1.0203 (6)	0.6861 (7)	0.067 (3)
C5A	0.7629 (1)	−1.1329 (6)	0.7138 (6)	0.055 (2)
C6A	0.7765 (1)	−1.2085 (7)	0.8195 (7)	0.071 (3)
C7A	0.8012 (2)	−1.1720 (9)	0.8954 (9)	0.092 (3)
C8A	0.7355 (1)	−1.1680 (6)	0.6371 (6)	0.056 (2)
N9A	0.73210 (8)	−1.1512 (4)	0.4806 (5)	0.048 (2)

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C10A	0.7128 (1)	−1.0822 (5)	0.3958 (6)	0.050 (2)
N11A	0.71125 (9)	−1.1093 (4)	0.2570 (5)	0.049 (2)
C12A	0.7306 (1)	−1.2038 (5)	0.2516 (6)	0.051 (2)
C13A	0.7439 (1)	−1.2302 (5)	0.3902 (6)	0.047 (2)
C14A	0.7643 (1)	−1.3185 (6)	0.4166 (7)	0.064 (2)
C15A	0.7711 (1)	−1.3828 (6)	0.2994 (8)	0.072 (3)
C16A	0.7582 (1)	−1.3595 (6)	0.1616 (8)	0.069 (3)
C17A	0.7378 (1)	−1.2698 (6)	0.1348 (7)	0.064 (3)
N18A	0.69760 (9)	−0.9920 (5)	0.4486 (5)	0.062 (2)
C19A	0.6714 (1)	−0.9531 (5)	0.3719 (6)	0.051 (2)
C20A	0.6677 (1)	−0.8135 (6)	0.3713 (8)	0.067 (2)
C21A	0.6395 (1)	−0.7760 (6)	0.2940 (7)	0.065 (2)
N22A	0.61719 (9)	−0.8385 (4)	0.3559 (5)	0.054 (2)
C23A	0.6208 (1)	−0.9719 (6)	0.3536 (7)	0.063 (2)
C24A	0.6478 (1)	−1.0137 (6)	0.4324 (7)	0.063 (2)
C25A	0.5902 (1)	−0.8060 (6)	0.2812 (7)	0.064 (2)
C26A	0.5820 (1)	−0.6693 (5)	0.2970 (6)	0.063 (2)
C27A	0.5520 (1)	−0.6483 (6)	0.2426 (6)	0.058 (2)
C28A	0.5314 (2)	−0.7049 (6)	0.3088 (8)	0.072 (3)
C29A	0.5041 (1)	−0.6887 (6)	0.2577 (8)	0.075 (3)
C30A	0.4966 (1)	−0.6128 (7)	0.1370 (9)	0.072 (3)
C31A	0.5166 (1)	−0.5573 (7)	0.0733 (9)	0.076 (3)
C32A	0.5437 (1)	−0.5735 (6)	0.1255 (7)	0.065 (2)
O33A	0.46861 (9)	−0.6082 (5)	0.0928 (7)	0.106 (3)
C34A	0.4597 (2)	−0.5289 (8)	−0.026 (1)	0.125 (4)
F1B	0.31487 (9)	0.0177 (6)	−0.4064 (6)	0.133 (3)
C2B	0.3392 (1)	0.0573 (6)	−0.3291 (6)	0.085 (4)
C3B	0.3518 (1)	−0.0187 (8)	−0.2256 (9)	0.089 (3)
C4B	0.3764 (1)	0.0196 (6)	−0.1495 (8)	0.072 (3)
C5B	0.3879 (1)	0.1329 (6)	−0.1783 (6)	0.050 (2)
C6B	0.3739 (1)	0.2087 (7)	−0.2813 (8)	0.074 (3)
C7B	0.3490 (2)	0.1718 (9)	−0.3583 (8)	0.087 (3)
C8B	0.4159 (1)	0.1713 (6)	−0.1036 (6)	0.057 (2)
N9B	0.41952 (9)	0.1559 (4)	0.0513 (5)	0.052 (2)
C10B	0.4387 (1)	0.0868 (5)	0.1385 (6)	0.047 (2)
N11B	0.44007 (9)	0.1114 (4)	0.2774 (5)	0.051 (2)
C12B	0.4207 (1)	0.2070 (5)	0.2831 (6)	0.047 (2)
C13B	0.4078 (1)	0.2353 (5)	0.1445 (7)	0.052 (2)
C14B	0.3877 (1)	0.3251 (6)	0.1175 (7)	0.065 (3)
C15B	0.3804 (1)	0.3883 (6)	0.2355 (9)	0.070 (3)
C16B	0.3930 (2)	0.3596 (6)	0.3735 (9)	0.072 (3)
C17B	0.4132 (1)	0.2697 (6)	0.3996 (7)	0.059 (2)
N18B	0.4537 (1)	−0.0015 (5)	0.0826 (6)	0.063 (2)
C19B	0.4790 (1)	−0.0500 (5)	0.1622 (7)	0.059 (2)
C20B	0.4798 (1)	−0.1902 (6)	0.1675 (8)	0.068 (2)
C21B	0.5062 (1)	−0.2365 (6)	0.2526 (8)	0.072 (2)
N22B	0.5294 (1)	−0.1946 (5)	0.1917 (5)	0.060 (2)
C23B	0.5302 (1)	−0.0576 (6)	0.1860 (8)	0.073 (3)
C24B	0.5038 (1)	−0.0069 (6)	0.0999 (8)	0.066 (2)
C25B	0.5545 (1)	−0.2439 (7)	0.2756 (7)	0.071 (3)
C26B	0.5780 (1)	−0.2439 (7)	0.1972 (7)	0.076 (2)
C27B	0.6043 (1)	−0.3016 (6)	0.2715 (6)	0.060 (2)
C28B	0.6265 (1)	−0.2985 (6)	0.2026 (7)	0.064 (2)
C29B	0.6514 (1)	−0.3507 (7)	0.2599 (8)	0.078 (3)
C30B	0.6536 (1)	−0.4081 (7)	0.3915 (8)	0.073 (3)
C31B	0.6316 (2)	−0.4157 (6)	0.4656 (9)	0.073 (3)
C32B	0.6073 (1)	−0.3606 (6)	0.4032 (7)	0.072 (3)
O33B	0.6765 (1)	−0.4710 (6)	0.4589 (6)	0.114 (3)
C34B	0.6986 (2)	−0.4859 (9)	0.384 (1)	0.145 (5)

Table 2. *Selected geometric parameters* (Å, °)

F1A—C2A	1.356 (8)	F1B—C2B	1.378 (8)
C2A—C3A	1.36 (1)	C2B—C3B	1.35 (1)
C2A—C7A	1.35 (1)	C2B—C7B	1.36 (1)
C3A—C4A	1.363 (9)	C3B—C4B	1.379 (9)
C4A—C5A	1.384 (9)	C4B—C5B	1.379 (9)
C5A—C6A	1.377 (9)	C5B—C6B	1.367 (9)
C5A—C8A	1.485 (8)	C5B—C8B	1.513 (8)
C6A—C7A	1.38 (1)	C6B—C7B	1.39 (1)
C8A—N9A	1.464 (7)	C8B—N9B	1.446 (7)
N9A—C10A	1.365 (7)	N9B—C10B	1.373 (7)
N9A—C13A	1.382 (7)	N9B—C13B	1.401 (8)
C10A—N11A	1.324 (7)	C10B—N11B	1.320 (7)
C10A—N18A	1.358 (8)	C10B—N18B	1.349 (8)
N11A—C12A	1.393 (7)	N11B—C12B	1.402 (7)
C12A—C13A	1.399 (8)	C12B—C13B	1.397 (8)
C12A—C17A	1.391 (9)	C12B—C17B	1.377 (9)
C13A—C14A	1.374 (8)	C13B—C14B	1.377 (8)
C14A—C15A	1.38 (1)	C14B—C15B	1.39 (1)
C15A—C16A	1.38 (1)	C15B—C16B	1.39 (1)
C16A—C17A	1.385 (9)	C16B—C17B	1.382 (9)
N18A—C19A	1.447 (8)	N18B—C19B	1.456 (7)
C19A—C20A	1.501 (8)	C19B—C20B	1.497 (8)
C19A—C24A	1.510 (9)	C19B—C24B	1.501 (9)
C20A—C21A	1.527 (8)	C20B—C21B	1.512 (9)
C21A—N22A	1.475 (8)	C21B—N22B	1.424 (9)
N22A—C23A	1.436 (7)	N22B—C23B	1.464 (8)
N22A—C25A	1.454 (7)	N22B—C25B	1.466 (8)
C23A—C24A	1.497 (8)	C23B—C24B	1.529 (9)
C25A—C26A	1.528 (8)	C25B—C26B	1.458 (9)
C26A—C27A	1.511 (7)	C26B—C27B	1.514 (8)
C27A—C28A	1.40 (1)	C27B—C28B	1.35 (1)
C27A—C32A	1.372 (8)	C27B—C32B	1.374 (9)
C28A—C29A	1.375 (9)	C28B—C29B	1.385 (9)
C29A—C30A	1.40 (1)	C29B—C30B	1.37 (1)
C30A—C31A	1.36 (1)	C30B—C31B	1.37 (1)
C30A—O33A	1.383 (8)	C30B—O33B	1.388 (9)
C31A—C32A	1.369 (9)	C31B—C32B	1.387 (9)
O33A—C34A	1.42 (1)	O33B—C34B	1.39 (1)
F1A—C2A—C7A	119.3 (7)	F1B—C2B—C7B	118.6 (7)
F1A—C2A—C3A	118.8 (7)	F1B—C2B—C3B	118.2 (7)
C3A—C2A—C7A	121.9 (7)	C3B—C2B—C7B	123.2 (7)
C2A—C3A—C4A	118.1 (7)	C2B—C3B—C4B	118.4 (8)
C3A—C4A—C5A	122.2 (6)	C3B—C4B—C5B	120.9 (7)
C4A—C5A—C8A	121.2 (6)	C4B—C5B—C8B	121.3 (5)
C4A—C5A—C6A	117.7 (6)	C4B—C5B—C6B	118.6 (6)
C6A—C5A—C8A	121.1 (6)	C6B—C5B—C8B	120.0 (5)
C5A—C6A—C7A	120.7 (7)	C5B—C6B—C7B	121.1 (7)
C2A—C7A—C6A	119.4 (8)	C2B—C7B—C6B	117.6 (8)
C5A—C8A—N9A	114.8 (5)	C5B—C8B—N9B	114.2 (5)
C8A—N9A—C13A	122.8 (4)	C8B—N9B—C13B	123.6 (5)
C8A—N9A—C10A	128.3 (5)	C8B—N9B—C10B	129.4 (5)
C10A—N9A—C13A	107.0 (4)	C10B—N9B—C13B	105.4 (4)
N9A—C10A—N18A	123.1 (5)	N9B—C10B—N18B	120.9 (5)
N9A—C10A—N11A	113.1 (5)	N9B—C10B—N11B	114.4 (5)
N11A—C10A—N18A	123.7 (5)	N11B—C10B—N18B	124.6 (5)
C10A—N11A—C12A	104.4 (4)	C10B—N11B—C12B	104.1 (4)
N11A—C12A—C17A	130.5 (5)	N11B—C12B—C17B	130.3 (5)
N11A—C12A—C13A	110.3 (5)	N11B—C12B—C13B	110.2 (5)
C13A—C12A—C17A	119.3 (5)	C13B—C12B—C17B	119.5 (5)
N9A—C13A—C12A	105.2 (5)	N9B—C13B—C12B	105.9 (5)
C12A—C13A—C14A	122.7 (5)	C12B—C13B—C14B	122.9 (6)
N9A—C13A—C14A	132.1 (5)	N9B—C13B—C14B	131.2 (6)
C13A—C14A—C15A	117.1 (6)	C13B—C14B—C15B	117.1 (6)
C14A—C15A—C16A	121.6 (6)	C14B—C15B—C16B	120.3 (6)
C15A—C16A—C17A	121.4 (6)	C15B—C16B—C17B	122.4 (7)
C12A—C17A—C16A	118.0 (6)	C12B—C17B—C16B	117.9 (6)
C10A—N18A—C19A	121.6 (5)	C10B—N18B—C19B	121.8 (5)
N18A—C19A—C24A	111.8 (5)	N18B—C19B—C24B	112.3 (5)
N18A—C19A—C20A	112.7 (5)	N18B—C19B—C20B	112.9 (5)
C20A—C19A—C24A	109.1 (5)	C20B—C19B—C24B	107.5 (5)
C19A—C20A—C21A	111.3 (5)	C19B—C20B—C21B	111.1 (5)
C20A—C21A—N22A	112.1 (5)	C20B—C21B—N22B	111.7 (6)
C21A—N22A—C25A	112.7 (5)	C21B—N22B—C25B	108.8 (5)
C21A—N22A—C23A	110.0 (5)	C21B—N22B—C23B	110.7 (5)
C23A—N22A—C25A	109.7 (5)	C23B—N22B—C25B	110.8 (5)
N22A—C23A—C24A	113.1 (5)	N22B—C23B—C24B	110.5 (5)
C19A—C24A—C23A	111.5 (5)	C19B—C24B—C23B	111.4 (5)
N22A—C25A—C26A	114.8 (5)	N22B—C25B—C26B	113.6 (5)
C25A—C26A—C27A	111.8 (5)	C25B—C26B—C27B	117.3 (5)
C26A—C27A—C32A	121.4 (5)	C26B—C27B—C32B	125.1 (6)
C26A—C27A—C28A	121.6 (5)	C26B—C27B—C28B	118.1 (6)
C28A—C27A—C32A	117.0 (6)	C28B—C27B—C32B	116.7 (6)
C27A—C28A—C29A	121.9 (6)	C27B—C28B—C29B	122.7 (6)
C28A—C29A—C30A	119.2 (6)	C28B—C29B—C30B	118.8 (7)
C29A—C30A—O33A	113.6 (6)	C29B—C30B—O33B	125.5 (7)
C29A—C30A—C31A	118.9 (7)	C29B—C30B—C31B	121.2 (7)
C31A—C30A—O33A	127.5 (7)	C31B—C30B—O33B	113.2 (6)
C30A—C31A—C32A	121.5 (7)	C30B—C31B—C32B	117.4 (7)
C27A—C32A—C31A	121.5 (6)	C27B—C32B—C31B	123.3 (6)
C30A—O33A—C34A	116.1 (6)	C30B—O33B—C34B	118.3 (7)

C4A—C5A—C8A—N9A	47.9 (8)
C5A—C8A—N9A—C10A	−123.8 (6)
C5A—C8A—N9A—C13A	73.9 (7)

C8A—N9A—C13A—C14A	−14.8 (9)
C8A—N9A—C10A—N18A	17.3 (9)
N9A—C10A—N18A—C19A	−156.9 (5)
C10A—N18A—C19A—C20A	−136.9 (6)
C10A—N18A—C19A—C24A	99.7 (6)
C21A—N22A—C25A—C26A	−66.7 (6)
C23A—N22A—C25A—C26A	170.5 (5)
N22A—C25A—C26A—C27A	−168.6 (5)
C25A—C26A—C27A—C28A	64.3 (7)
C29A—C30A—O33A—C34A	177.7 (7)
C4B—C5B—C8B—N9B	−48.9 (8)
C5B—C8B—N9B—C10B	122.3 (6)
C5B—C8B—N9B—C13B	−74.3 (7)
C8B—N9B—C13B—C14B	13 (1)
C8B—N9B—C10B—N18B	−15.4 (9)
N9B—C10B—N18B—C19B	161.9 (5)
C10B—N18B—C19B—C20B	129.0 (6)
C10B—N18B—C19B—C24B	−109.2 (6)
C21B—N22B—C25B—C26B	160.0 (6)
C23B—N22B—C25B—C26B	−77.4 (7)
N22B—C25B—C26B—C27B	−175.8 (5)
C25B—C26B—C27B—C28B	−177.8 (6)
C29B—C30B—O33B—C34B	−4 (1)

The data were collected with a variable scan speed between 1.96 and 29.30° min^{-1}. The scan width was 1° below $K\alpha_1$ and 1° above $K\alpha_2$ with a ratio of total background time to scan time of 1. The intensity data were corrected for the 5% decay. Systematic absences indicated $C2/c$ or Cc as space group. Although intensity statistics indicated the centrosymmetric space group, structure solution and refinement with full-matrix least squares on F^2 for all reflections resulted in an R value not lower than 0.15. Structure solution and refinement in space group Cc converged to $R = 0.0396$. H atoms were calculated at geometrical positions and were allowed to ride on their parent atom.

Data collection: $P2_1$ *Diffractometer Program* (Syntex, 1975). Cell refinement: $P2_1$ *Diffractometer Program*. Data reduction: *REDU*4 (Stoe & Cie, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEX*2.1 (McArdle, 1994). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Methyl 2α,3β,23-Triacetoxyurs-12,18-dien-28-oate

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Abstract

The crystal and molecular structure of the novel triterpenoid methyl 2α,3β,23-triacetoxyurs-12,18-dien-28-oate, $C_{37}H_{54}O_8$, has been determined. There are two molecules in the asymmetric unit, each having a long bond due to steric effects and considerable out-of-plane bending at the C12═C13—C18═C19 chromophores.

Comment

A number of compounds have been isolated from *Rubus pinfaensis* Levl. et Vant, a herb used in Chinese medicine to promote wound healing (Liu, 1994). We have reported previously the structures of two triterpenoids from this source (Cox, Durham, Liu & Richards, 1993, 1994) and now report on a further novel triterpenoid. Overall, the molecule adopts a slightly bow-shaped conformation (Fig. 2) with the β-face concave; the stereochemistry has been established as 2α-OAc, 3β-OAc, 4β-Me, 4α-COAc, 8β-Me, 10β-Me, 14α-Me, 17β-COOMe, 20α-Me. Ring conformations are: *A* chair, *B* chair, *C* distorted C9 sofa, *D* chair, *E* C21 sofa. The C12═C13—C18═C19 torsion angles for the two molecules in the asymmetric unit are −54.2 (6) and −55.1 (6)°, departing considerably from an ideal strain-free *cis* torsion angle of 0°. After 1000 block-diagonal Newton–Raphson iterations (Hypercube Inc, 1994) starting with the atom coordinates of molecule 1, this torsion
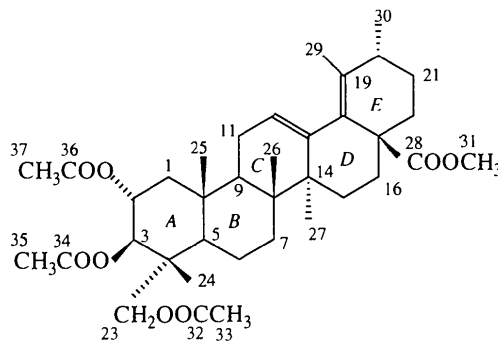

Fig. 1. A schematic view of the molecule showing the numbering scheme used.