

Table 2. Selected bond lengths (Å), angles (°) and torsion angles (°)

S(1)—C(2)	1.779 (2)	C(9)—C(11)	1.530 (4)
S(1)—C(12)	1.791 (2)	C(14)—O(15)	1.376 (2)
C(7)—C(8)	1.514 (3)	C(17)—O(18)	1.366 (2)
C(8)—C(9)	1.518 (3)	O(15)—C(16)	1.418 (3)
C(9)—N(10)	1.508 (3)	O(18)—C(19)	1.438 (3)
C(2)—S(1)—C(12)	104.4 (1)	N(10)—C(9)—C(11)	107.8 (2)
S(1)—C(2)—C(7)	118.6 (2)	C(13)—C(14)—C(17)	120.1 (2)
S(1)—C(2)—C(3)	122.1 (2)	C(14)—C(17)—C(20)	119.5 (2)
S(1)—C(12)—C(13)	119.0 (2)	C(13)—C(14)—O(15)	125.6 (2)
S(1)—C(12)—C(21)	120.8 (2)	C(20)—C(17)—O(18)	125.7 (2)
C(3)—C(2)—C(7)	119.3 (2)	O(15)—C(14)—C(17)	114.3 (2)
C(13)—C(12)—C(21)	120.1 (2)	O(18)—C(17)—C(14)	114.8 (2)
C(7)—C(8)—C(9)	116.2 (2)	C(14)—O(15)—C(16)	119.1 (2)
C(8)—C(9)—N(10)	110.3 (2)	C(17)—O(18)—C(19)	117.9 (2)
C(8)—C(9)—C(11)	111.3 (2)		
C(12)—S(1)—C(2)—C(7)	-177.5 (2)	C(13)—C(14)—O(15)—C(16)	-4.8 (3)
C(12)—S(1)—C(2)—C(3)	2.0 (2)	C(20)—C(17)—O(18)—C(19)	1.1 (3)
C(2)—S(1)—C(12)—C(13)	-99.7 (2)	C(14)—O(15)—C(16)—H(161)	-160 (2)
C(2)—S(1)—C(12)—C(21)	81.6 (2)	C(14)—O(15)—C(16)—H(162)	69 (2)
S(1)—C(2)—C(3)—C(4)	-179.7 (2)	C(14)—O(15)—C(16)—H(163)	-52 (2)
S(1)—C(2)—C(7)—C(8)	1.4 (3)	C(17)—O(18)—C(19)—H(191)	166 (2)
S(1)—C(12)—C(13)—C(14)	-179.9 (2)	C(17)—O(18)—C(19)—H(192)	66 (2)
C(2)—C(7)—C(8)—C(9)	-77.5 (3)	C(17)—O(18)—C(19)—H(193)	-59 (2)
C(7)—C(8)—C(9)—N(10)	-55.7 (3)	C(8)—C(9)—N(10)—H(103)	179 (3)
C(7)—C(8)—C(9)—C(11)	-175.4 (2)	C(8)—C(9)—C(11)—H(112)	-177 (2)

The S1 atom as well as the non-H atoms of both methoxy groups lie nearly in this plane with greatest deviations for C(16): -0.187 (3) and C(19): 0.070 (3) Å. Angles O(15)—C(14)—C(17) and O(18)—C(17)—C(14) (see Table 2) are lower than their ideal value of 120° because of repulsion between the methoxy groups and the phenyl ring,

and attraction between the O(15) and O(18) atoms and the H(101<sup>i</sup>) atom. H atoms of the methyl groups minimize overlap with the phenyl ring [see Table 2; H(131)···H(162) 2.47 (3), H(131)···H(163) 2.36 (4), H(201)···H(192) 2.48 (4), H(201)···H(193) 2.33 (4) Å].

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## Structure of 3-Phenyl-3-piperidino-2,4-pentanedione Monooxime

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**Abstract.** C<sub>16</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>, *M<sub>r</sub>* = 274.4, monoclinic, *P*2<sub>1</sub>/*a*, *a* = 14.253 (9), *b* = 9.994 (6), *c* = 10.468 (4) Å, β = 93.70 (4)°, *V* = 1488.0 Å<sup>3</sup>, *Z* = 4, *D<sub>m</sub>* = 1.21, *D<sub>x</sub>* = 1.22 Mg m<sup>-3</sup>, λ(Mo Kα) = 0.71069 Å, μ = 0.076 mm<sup>-1</sup>, *F*(000) = 592, *T* = 293 K, *R* = 0.06 for 2341 observed reflections. The conformation of the piperidine ring is a chair. The dihedral angle between the least-squares planes through the benzene and the six-membered heterocyclic rings is 56.7 (1)°. Crystal cohesion is mainly governed by van der Waals contacts, although there is a weak intermolecular H bond.

**Introduction.** The title compound, supplied by Professor Gómez-Sánchez and co-workers, is a product of the reaction of 2,4-dinitropentanedione with piperidine (Cabrera-Escribano, Derri Alcantara & Gómez-Sánchez, 1989). Because of the unusual character of the compound it was decided to carry out an X-ray diffraction study to confirm the structure and to obtain details about its stereochemistry and conformation.

**Experimental.** Crystals were colourless prisms (0.11 × 0.28 × 0.25 mm). *D<sub>m</sub>* by flotation. Enraf–Nonius

Table 1. Fractional atomic and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^4$ ) for the non-hydrogen atoms
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j \cos(\mathbf{a}_i, \mathbf{a}_j).$$

	x	y	z	$U_{eq}$
O	0.4418 (2)	0.4398 (2)	0.2303 (2)	454
O(4)	0.6767 (2)	0.3742 (2)	-0.0870 (2)	457
N(3)	0.6477 (2)	0.3235 (2)	0.3493 (2)	324
N(4)	0.6260 (2)	0.3940 (3)	0.0233 (2)	365
C(1)	0.5712 (3)	0.5848 (3)	0.2174 (3)	486
C(2)	0.5257 (2)	0.4489 (3)	0.2204 (3)	355
C(3)	0.5893 (2)	0.3242	0.2277 (3)	296
C(4)	0.6504 (2)	0.3125 (3)	0.1136 (3)	306
C(5)	0.7211 (2)	0.2032 (3)	0.1083 (3)	403
C(31)	0.5296 (2)	0.1934 (3)	0.2178 (3)	291
C(32)	0.4627 (2)	0.1789 (3)	0.1161 (3)	358
C(33)	0.4128 (2)	0.0603 (3)	0.0981 (3)	432
C(34)	0.4299 (2)	-0.0466 (3)	0.1796 (3)	451
C(35)	0.4971 (2)	-0.0341 (3)	0.2803 (3)	459
C(36)	0.5476 (2)	0.0846 (3)	0.2984 (3)	395
C(3'2)	0.5953 (2)	0.3424 (3)	0.4641 (3)	391
C(3'3)	0.6541 (3)	0.2931 (4)	0.5815 (3)	452
C(3'4)	0.7490 (3)	0.3625 (4)	0.5939 (3)	532
C(3'5)	0.7982 (2)	0.3517 (4)	0.4698 (3)	515
C(3'6)	0.7343 (2)	0.4025 (3)	0.3578 (3)	421

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

O(4)—N(4)	1.415 (4)	C(3'4)—C(3'3)	1.518 (5)
O—C(2)	1.209 (4)	C(3'4)—C(3'5)	1.519 (5)
N(3)—C(3)	1.474 (4)	C(2)—C(1)	1.506 (4)
N(3)—C(3'2)	1.466 (4)	C(3'2)—C(3'3)	1.523 (5)
N(3)—C(3'6)	1.463 (4)	C(35)—C(34)	1.382 (5)
N(4)—C(4)	1.279 (4)	C(35)—C(36)	1.394 (4)
C(31)—C(3)	1.559 (3)	C(5)—C(4)	1.489 (5)
C(31)—C(32)	1.390 (4)	C(32)—C(33)	1.388 (5)
C(31)—C(36)	1.389 (4)	C(33)—C(34)	1.379 (5)
C(3)—C(2)	1.539 (3)	C(3'6)—C(3'5)	1.524 (5)
C(3)—C(4)	1.528 (4)		
C(3'2)—N(3)—C(3'6)	110.6 (2)	C(3)—C(2)—C(1)	118.5 (3)
C(3)—N(3)—C(3'6)	118.4 (2)	O—C(2)—C(1)	119.9 (3)
C(3)—N(3)—C(3'2)	114.7 (3)	N(3)—C(3'2)—C(3'3)	109.6 (3)
O(4)—N(4)—C(4)	112.6 (3)	C(34)—C(35)—C(36)	120.4 (3)
C(32)—C(31)—C(36)	118.2 (3)	C(3)—C(4)—C(5)	120.4 (2)
C(3)—C(31)—C(36)	122.4 (3)	N(4)—C(4)—C(5)	126.3 (3)
C(3)—C(31)—C(32)	118.8 (2)	N(4)—C(4)—C(3)	112.7 (2)
N(3)—C(3)—C(31)	109.1 (2)	C(31)—C(32)—C(33)	120.8 (3)
C(31)—C(3)—C(4)	102.6 (2)	C(32)—C(33)—C(34)	120.5 (3)
C(31)—C(3)—C(2)	111.0 (2)	C(35)—C(34)—C(33)	119.1 (3)
N(3)—C(3)—C(4)	110.8 (2)	C(3'4)—C(3'3)—C(3'2)	111.2 (3)
N(3)—C(3)—C(2)	110.1 (2)	N(3)—C(3'6)—C(3'5)	109.1 (3)
C(2)—C(3)—C(4)	112.7 (2)	C(31)—C(36)—C(35)	120.6 (3)
C(3'3)—C(3'4)—C(3'5)	110.5 (3)	C(3'4)—C(3'5)—C(3'6)	110.3 (3)
O—C(2)—C(3)	121.0 (3)		

CAD-4 diffractometer with graphite monochromator and Mo  $K\alpha$  radiation. Unit-cell parameters were obtained from 25 reflections with  $2 < \theta < 14^\circ$ . 4492 independent reflections ( $0 \leq h \leq 19$ ,  $0 \leq k \leq 14$ ,  $-14 \leq l \leq 14$ ) measured to  $2\theta = 60^\circ$  in the  $\omega$ - $2\theta$  mode. Two standard reflections (204 and  $\bar{2}04$ ) monitored every 100 reflections showed only statistical fluctuations. 2341 reflections considered observed [ $I \geq 2\sigma(I_o)$ ] and used for structure determination. Lp correction applied, absorption and extinction ignored; direct methods, MULTAN80 (Main, Fiske,

Hull, Lessinger, Germain, Declercq & Woolfson, 1980), full-matrix least-squares refinement based on  $F$ , with anisotropic thermal parameters for all non-H atoms [ $w = 1/\sigma^2(F)$ ]; difference Fourier synthesis revealed H atoms which were included in fixed positions with  $U_{iso}$  values (corresponding to those of carrier atoms). Further least-squares refinement produced convergence with  $R = 0.063$ ,  $wR = 0.062$  and  $S = 2.12$ . Final difference Fourier synthesis showed  $\Delta\rho = +0.3 \text{ e \AA}^{-3}$ .  $(\Delta/\sigma)_{\max} = 0.002$ . The  $y$  coordinate of C(3) was held fixed. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974). The XRAY system of computer programs (Stewart, Kundell & Baldwin, 1970) was used.

**Discussion.** The positional and isotropic thermal parameters for non-H atoms are given in Table 1.\* Table 2 provides the bond distances and angles. Fig. 1 is a view of the molecule along the  $a$  axis, showing the atomic numbering, together with thermal ellipsoids. With respect to the oxime group, the values for the C(4)—N(4) and N(4)—O(4) bond lengths of 1.280 (4) and 1.415 (4)  $\text{\AA}$  compare well with the values ranging from 1.22 (3) to 1.33 (1) and from 1.357 (18) to 1.439 (39)  $\text{\AA}$  for the same bond in other oximes (Ansell, Moore & Nielsen, 1971). The lengths

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52892 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

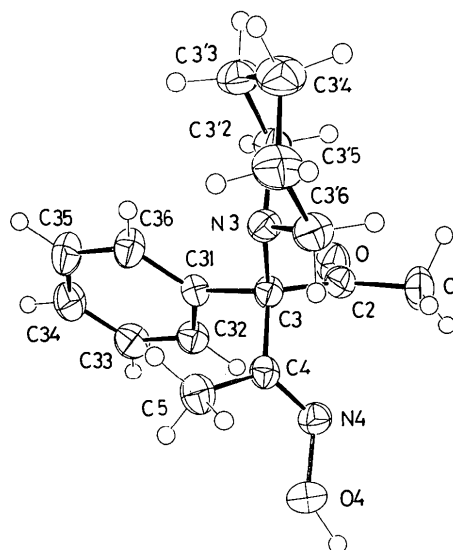


Fig. 1. ORTEP (Johnson, 1976) view of the title compound along the  $a$  axis showing the atomic numbering. Thermal ellipsoids enclose 50% probability and H atoms are represented by spheres of arbitrary size.

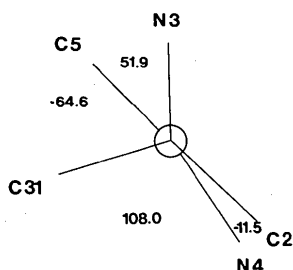


Fig. 2. Newman projection along C(3)—C(4). Torsion angles ( $^{\circ}$ ); mean e.s.d.  $0.3^{\circ}$ .

of C(4)—C(5) at 1.490 (4) and C(2)—C(1) at 1.507 (5) Å are shorter than C(4)—C(3) at 1.528 (4) and C(3)—C(2) at 1.540 (4) Å because of the double bonds (*International Tables for X-ray Crystallography*, 1974). Lengths and angles in the phenyl ring agree well with the standard values and the average C—H bond length is 1.010 (3) Å. This group can be considered planar as the maximum deviation from the next plane is 0.010 (3) Å. The average C—C bond length is 1.388 (4) Å. The substituent C(3) is  $-0.113$  (3) Å from the least-squares phenyl plane. Likewise, in the piperidine ring, lengths and angles agree well with those reported by Herbstein, Schwotzer, Addae-Mensah, Torto & Woode (1981) and Jaskolski (1987). The C—H bond lengths range from 1.005 (3) to 1.013 (4) Å with a mean value of 1.010 (4) Å. The molecular conformation of the piperidine ring in the crystal has been examined in terms of asymmetry and puckering parameters. Cremer & Pople's (1975) puckering parameters for the atomic sequence C(3'2)—C(3'3)—C(3'4)—C(3'5)—C(3'6)—N(3) are  $\theta = 175$  (1) $^{\circ}$ ,  $\varphi = 100$  (3) $^{\circ}$  and  $Q = 0.59$  (1) Å, and the Nardelli (1983) asym-

metry parameters are  $\Delta C_3[C(3'4)] = 0.013$  (2) and  $\Delta C_2[C(3'4)—C(3'3)] = 0.007$  (1) showing that the piperidine ring adopts the chair conformation with the substituent C(3) axial. The Newman projection (Fig. 2) shows clearly the distribution of the atoms attached to the C(3) chiral centre. Crystal packing is mainly governed by van der Waals contacts, although there is a weak intermolecular H bond: O(4)—H $\cdots$ O( $-x + 1, -y + 1, -z$ ) = 2.869 (3) Å [O(4)—H $\cdots$ O = 123.6 (2) $^{\circ}$ ].

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### (+)-Jaboromagellone, a New Withanolide from *Jaborosa magellanica*

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(Received 1 September 1989; accepted 5 December 1989)

**Abstract.** C<sub>28</sub>H<sub>36</sub>O<sub>6</sub>,  $M_r = 470.61$ , monoclinic,  $P2_1$ ,  $a = 11.516$  (2),  $b = 7.479$  (3),  $c = 14.558$  (3) Å,  $\beta = 103.16$  (2) $^{\circ}$ ,  $V = 1220.9$  Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.27$ ,  $D_x = 1.28$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu =$

$0.083$  mm<sup>-1</sup>,  $F(000) = 504$ ,  $T = 293$  (1) K,  $R = 0.045$  for 2093 observed reflections with  $I \geq 3\sigma(I)$ . The crystal structure consists of discrete molecules of (+)-jaboromagellone separated by normal van der Waals distances. The mean bond lengths are C(sp<sup>3</sup>)—C(sp<sup>3</sup>) 1.529 (6), C(sp<sup>3</sup>)—C(sp<sup>2</sup>) 1.513 (5), C(sp<sup>2</sup>)—C(sp<sup>2</sup>) 1.468 (6), C=C 1.342 (7), C(sp<sup>3</sup>)—O

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