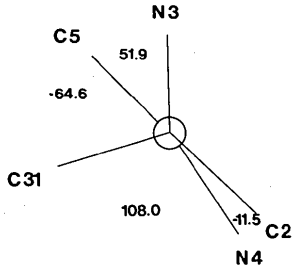

Fig. 2. Newman projection along C(3)—C(4). Torsion angles (°); mean e.s.d. 0·3°.

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 $\theta = 175\ (1)°$, $\varphi = 100\ (3)°$ and $Q = 0{\cdot}59\ (1)$ Å, and the Nardelli (1983) asymmetry parameters are $\Delta C_s[C(3'4)] = 0{\cdot}013\ (2)$ and $\Delta C_2[C(3'4—C(3'3)] = 0{\cdot}007\ (1)$ 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···O($-x+1$, $-y+1$, $-z$) = 2·869 (3) Å [O(4)—H···O = 123·6 (2)°].

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

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Abstract. $C_{28}H_{36}O_6$, $M_r = 470{\cdot}61$, monoclinic, $P2_1$, $a = 11{\cdot}516\ (2)$, $b = 7{\cdot}479\ (3)$, $c = 14{\cdot}558\ (3)$ Å, $\beta = 103{\cdot}16\ (2)°$, $V = 1220{\cdot}9$ Å^3, $Z = 2$, $D_m = 1{\cdot}27$, $D_x = 1{\cdot}28$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}083$ mm^{-1}, $F(000) = 504$, $T = 293\ (1)$ K, $R = 0{\cdot}045$ 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^3)—C(sp^3) 1·529 (6), C(sp^3)—C(sp^2) 1·513 (5), C(sp^2)—C(sp^2) 1·468 (6), C=C 1·342 (7), C(sp^3)—O

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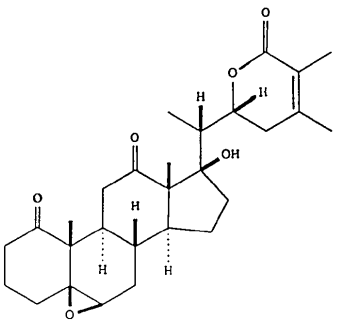
1·442 (5), C(sp^2)—O 1·348 (4) and C=O 1·201 (5) Å. Ring *A* is in a twist-boat conformation, ring *B* a half-chair, ring *C* a regular chair, and ring *D* is a C(14) envelope. The δ-lactone ring *E* adopts a half-chair conformation. Atoms O(2), C(18), C(19) and O(4) are β and the lactone moiety is α oriented.

Introduction. The withanolides are a group of C_{28} steroids found solely among the Solanaceae. Our current investigation of *Jaborosa magellanica* (Griseb.) Dusen (Solanaceae) has yielded several withanolides (Parvez, Fajardo & Shamma, 1987*a*,*b*; Fajardo, Freyer, Minard & Shamma, 1987). Interestingly enough, all proved to be new, and oxygenated at both C(1) and C(12). The C(17) side chain of the *J. magellanica* withanolides includes either a γ-lactone or a δ-lactone.

Presently, we describe the structure and relative configuration of (+)-jaboromagellone which incorporates the δ-lactone arrangement; the absolute configuration was not determined.



(+)-Jaboromagellone

Experimental. A colorless prismatic crystal of (+)-jaboromagellone of approximate size 0·35 × 0·35 × 0·45 mm was chosen for data collection. D_m measured by flotation. Accurate cell parameters and a crystal orientation matrix were determined on an Enraf–Nonius CAD-4 diffractometer by least-squares refinement of the setting angles of 25 reflections with $10 < \theta < 15°$. Intensity data were collected by the $\omega/2\theta$ scan method using monochromated radiation in the range $2 < \theta < 25°$ with $h \le 13$, $k \le 19$ and $-16 \le l \le 16$. The intensities of three reflections, chosen as standards, were monitored every two hours of exposure time and showed no significant variation. The intensities of 2322 unique reflections were measured, of which 2093 had $I > 3\sigma(I)$, where $\sigma^2(I) = S + 2B + [0{\cdot}04(S - B)]^2$, S = scan count and B = time-averaged background count. Data were corrected for Lorentz and polarization effects; an absorption correction was not considered necessary.

The structure was solved by direct methods using *MULTAN*82 (Main *et al.*, 1982). The structure was refined by full-matrix least-squares calculations employing *F*'s with anisotropic thermal parameters for the non-H atoms. A difference Fourier synthesis calculated at this stage of the refinement revealed all H atoms. These were included in the refinement with idealized geometry (C—H and O—H 0·95 Å) and an overall fixed isotropic temperature factor in the subsequent rounds of calculations. Atomic scattering factors for carbon and oxygen were taken from Cromer & Mann (1968), and those for hydrogen were from Stewart, Davidson & Simpson (1965). At the conclusion of the refinement, $(\Delta/\sigma)_{max} < 0{\cdot}1$, the difference electron density map was essentially featureless with $\Delta\rho \pm 0{\cdot}23$ e Å^{-3}. The final values of *R* and *wR* were 0·045 and 0·067, respectively, where $w = [\sigma^2(F_o) + (0{\cdot}020F_o)^2]^{-1}$ and goodness of fit, S = 1·34. The computer programs used in this study were from the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates Inc., 1985) and *ORTEP* (Johnson, 1976). Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s are given in Table 1.*

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	*y*	*z*	B_{eq}
O(1)	0·3272 (2)	0·6250†	0·7494 (2)	4·35 (6)
O(2)	0·6679 (2)	0·4599 (4)	0·6365 (2)	4·06 (6)
O(3)	0·5631 (2)	0·4230 (5)	1·0695 (2)	4·55 (7)
O(4)	0·8571 (2)	0·2417 (4)	1·1634 (2)	3·31 (5)
O(5)	0·8078 (2)	0·6402 (4)	1·3316 (2)	3·80 (6)
O(6)	0·7864 (3)	0·6975 (6)	1·4744 (2)	5·75 (8)
C(1)	0·4181 (3)	0·6708 (6)	0·7293 (2)	2·97 (7)
C(2)	0·4414 (4)	0·8599 (6)	0·7035 (3)	4·37 (9)
C(3)	0·4759 (4)	0·8723 (7)	0·6079 (3)	4·4 (1)
C(4)	0·5157 (3)	0·6942 (7)	0·5759 (2)	3·89 (9)
C(5)	0·5893 (3)	0·5959 (5)	0·6581 (2)	3·00 (7)
C(6)	0·7188 (3)	0·6213 (6)	0·6827 (2)	3·44 (8)
C(7)	0·7891 (3)	0·6011 (7)	0·7809 (2)	3·53 (8)
C(8)	0·7288 (3)	0·4836 (5)	0·8418 (2)	2·66 (7)
C(9)	0·6012 (3)	0·5483 (5)	0·8348 (2)	2·50 (7)
C(10)	0·5210 (3)	0·5371 (5)	0·7326 (2)	2·54 (7)
C(11)	0·5399 (3)	0·4569 (7)	0·9061 (2)	3·73 (8)
C(12)	0·6118 (3)	0·4275 (6)	1·0044 (2)	3·12 (7)
C(13)	0·7431 (3)	0·3780 (6)	1·0135 (2)	3·00 (7)
C(14)	0·7960 (3)	0·4946 (5)	0·9452 (2)	2·70 (7)
C(15)	0·9283 (3)	0·4491 (6)	0·9720 (2)	3·09 (7)
C(16)	0·9531 (3)	0·4451 (6)	1·0786 (2)	3·18 (7)
C(17)	0·8375 (3)	0·4049 (5)	1·1107 (2)	2·75 (7)
C(18)	0·7375 (3)	0·1797 (6)	0·9842 (3)	3·63 (8)
C(19)	0·4703 (4)	0·3504 (6)	0·7070 (3)	3·88 (9)
C(20)	0·8017 (3)	0·5521 (6)	1·1755 (2)	2·98 (7)
C(21)	0·7898 (3)	0·7413 (6)	1·1348 (3)	3·74 (8)
C(22)	0·8781 (3)	0·5462 (6)	1·2758 (3)	3·17 (8)
C(23)	1·0014 (4)	0·6239 (7)	1·2975 (3)	4·33 (9)
C(24)	1·0540 (4)	0·6226 (6)	1·4030 (3)	4·29 (9)
C(25)	0·9802 (4)	0·6238 (6)	1·4623 (3)	4·03 (9)
C(26)	0·8521 (4)	0·6537 (6)	1·4254 (3)	3·90 (9)
C(27)	1·0205 (5)	0·6150 (8)	1·5678 (3)	5·8 (1)
C(28)	1·1872 (4)	0·6206 (9)	1·4327 (4)	6·1 (1)

† The *y* coordinate of O(1) was not allowed to refine in order to fix the origin.

* Lists of anisotropic temperature factors, H-atom parameters, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52834 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Molecular dimensions* (Å, °) *for* (+)-*jaboromagellone with e.s.d.'s in parentheses*

O(1)—C(1)	1·200 (4)	C(9)—C(11)	1·542 (5)
O(2)—C(5)	1·444 (5)	C(10)—C(19)	1·527 (6)
O(2)—C(6)	1·441 (5)	C(11)—C(12)	1·498 (4)
O(3)—C(12)	1·206 (5)	C(12)—C(13)	1·533 (5)
O(4)—C(17)	1·432 (5)	C(13)—C(14)	1·547 (5)
O(5)—C(22)	1·452 (5)	C(13)—C(17)	1·589 (4)
O(5)—C(26)	1·348 (4)	C(13)—C(18)	1·540 (6)
O(6)—C(26)	1·198 (6)	C(14)—C(15)	1·523 (4)
C(1)—C(2)	1·502 (6)	C(15)—C(16)	1·513 (5)
C(1)—C(10)	1·542 (5)	C(16)—C(17)	1·538 (5)
C(2)—C(3)	1·536 (6)	C(17)—C(20)	1·565 (5)
C(3)—C(4)	1·515 (8)	C(20)—C(21)	1·527 (6)
C(4)—C(5)	1·493 (5)	C(20)—C(22)	1·524 (4)
C(5)—C(6)	1·465 (5)	C(22)—C(23)	1·500 (5)
C(5)—C(10)	1·541 (6)	C(23)—C(24)	1·518 (6)
C(6)—C(7)	1·481 (4)	C(24)—C(25)	1·342 (7)
C(7)—C(8)	1·524 (6)	C(24)—C(28)	1·496 (6)
C(8)—C(9)	1·529 (5)	C(25)—C(26)	1·468 (6)
C(8)—C(14)	1·530 (4)	C(25)—C(27)	1·503 (6)
C(9)—C(10)	1·565 (4)		

C(5)—O(2)—C(6)	61·0 (2)	C(1)—C(10)—C(9)	106·1 (3)
C(22)—O(5)—C(26)	118·2 (3)	C(1)—C(10)—C(19)	109·7 (3)
O(1)—C(1)—C(2)	123·1 (3)	C(5)—C(10)—C(9)	111·6 (3)
O(1)—C(1)—C(10)	120·9 (3)	C(5)—C(10)—C(19)	108·5 (4)
C(2)—C(1)—C(10)	116·0 (3)	C(9)—C(10)—C(19)	113·0 (3)
C(1)—C(2)—C(3)	112·4 (4)	C(9)—C(11)—C(12)	118·1 (3)
C(2)—C(3)—C(4)	112·6 (4)	O(3)—C(12)—C(11)	120·0 (4)
C(3)—C(4)—C(5)	110·1 (3)	O(3)—C(12)—C(13)	123·5 (3)
O(2)—C(5)—C(4)	116·2 (3)	C(11)—C(12)—C(13)	115·9 (4)
O(2)—C(5)—C(6)	59·4 (3)	C(12)—C(13)—C(14)	109·5 (3)
O(2)—C(5)—C(10)	113·5 (3)	C(12)—C(13)—C(17)	120·6 (3)
C(4)—C(5)—C(6)	119·5 (3)	C(12)—C(13)—C(18)	103·3 (3)
C(4)—C(5)—C(10)	114·6 (3)	C(14)—C(13)—C(17)	102·2 (3)
C(6)—C(5)—C(10)	121·3 (3)	C(14)—C(13)—C(18)	111·3 (4)
O(2)—C(6)—C(5)	59·6 (2)	C(17)—C(13)—C(18)	110·2 (3)
O(2)—C(6)—C(7)	116·1 (4)	C(8)—C(14)—C(13)	114·6 (3)
C(5)—C(6)—C(7)	121·7 (3)	C(8)—C(14)—C(15)	119·5 (3)
C(6)—C(7)—C(8)	113·6 (3)	C(13)—C(14)—C(15)	103·1 (3)
C(7)—C(8)—C(9)	109·7 (3)	C(14)—C(15)—C(16)	102·2 (3)
C(7)—C(8)—C(14)	110·3 (3)	C(15)—C(16)—C(17)	109·9 (3)
C(9)—C(8)—C(14)	108·2 (3)	O(4)—C(17)—C(13)	111·2 (3)
C(8)—C(9)—C(10)	113·4 (3)	O(4)—C(17)—C(16)	106·7 (3)
C(8)—C(9)—C(11)	113·3 (3)	O(4)—C(17)—C(20)	108·0 (3)
C(10)—C(9)—C(11)	110·8 (3)	C(13)—C(17)—C(16)	102·6 (3)
C(1)—C(10)—C(5)	107·7 (3)	C(13)—C(17)—C(20)	114·0 (3)
C(16)—C(17)—C(20)	114·2 (4)	C(23)—C(24)—C(28)	116·1 (5)
C(17)—C(20)—C(21)	115·5 (4)	C(25)—C(24)—C(28)	124·9 (4)
C(17)—C(20)—C(22)	112·5 (3)	C(24)—C(25)—C(26)	119·8 (3)
C(21)—C(20)—C(22)	112·3 (3)	C(24)—C(25)—C(27)	124·3 (4)
O(5)—C(22)—C(20)	104·4 (3)	C(26)—C(25)—C(27)	115·6 (5)
O(5)—C(22)—C(23)	108·5 (3)	O(5)—C(26)—O(6)	118·6 (3)
C(20)—C(22)—C(23)	120·1 (3)	O(5)—C(26)—C(25)	118·4 (4)
C(22)—C(23)—C(24)	110·5 (4)	O(6)—C(26)—C(25)	122·9 (3)
C(23)—C(24)—C(25)	119·0 (4)		

Discussion. Our X-ray analysis unequivocally establishes the molecular structure and relative stereochemistry of the title compound. A view of the molecule is given in Fig. 1 with the crystallographic numbering scheme. Bond lengths, bond angles and important torsion angles are given in Table 2. In the molecule of (+)-jaboromagellone, ring *A* has 1α,4α twist-boat conformation with C(1) and C(4) 0·515 (3) and 0·611 (4) Å, respectively, above the C(2), C(3), C(5), C(10) plane. Ring *B* is *cis* fused to ring *A* and has a half-chair conformation with 8β,9α orientation. Ring *C* adopts a regular chair conformation. The five-membered ring *D*, which is *cis* fused to ring *C*, has a C(14)-envelope conformation with C(14) 0·661 (4) Å above the C(13), C(15), C(16), C(17) plane. The lactone moiety, ring *E*, adopts a half-chair conformation. The methyl groups, C(18) and C(19), the hydroxy group O(4) and the epoxide O(2) are all β-oriented, while the δ-lactone moiety exhibits α orientation.

The molecular dimensions are in accord with accepted values, mean bond lengths being C(sp^3)—C(sp^3) 1·529 (6) [the shortest is 1·465 (5) Å, observed in ring *B* to which the highly constrained epoxide is fused, and the longest is C(13)—C(17), 1·589 (4) Å, in the ring *D*], C(sp^3)—C(sp^2) 1·513 (5), C(sp^2)—C(sp^2) 1·468 (6), C(sp^2)=C(sp^2) 1·412 (7), C(sp^3)—O 1·442 (5), C(sp^2)—O 1·348 (4) and C=O 1·201 (5) Å.

The crystal structure, Fig. 2, consists of discrete molecules separated by normal van der Waals distances with a short hydrogen bond [O(1)···H(O4)^i 2·31 Å, O(1)···H(O4)^i—O(4)^i 115°; (i) = $-x+1$, $\frac{1}{2}+y$, $-z+2$].

This research was supported by NSF grant INT-851226, and by CONICYT and FONDECYT grant 1206.

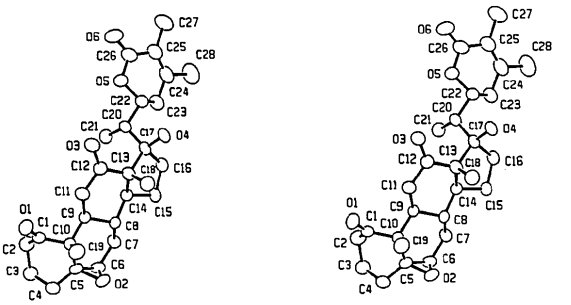


Fig. 1. Stereoview of the molecular structure of (+)-jaboromagellone with the crystallographic numbering scheme.

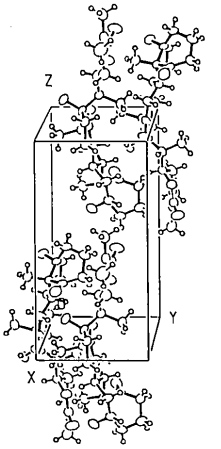


Fig. 2. Unit cell of (+)-jaboromagellone showing the molecular packing.

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Structures of Four Related 4,5,6,7-Tetrahydro-1,2,5-oxadiazolo[3,4-*b*]pyrazines

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Abstract. 4,5,6,7-Tetrahydro-1,2,5-oxadiazolo[3,4-*b*]-pyrazine, (I), $C_4H_6N_4O$, $M_r = 126{\cdot}12$, monoclinic, $P2_1/n$, $a = 6{\cdot}584$ (1), $b = 7{\cdot}105$ (1), $c = 11{\cdot}784$ (2) Å, $\beta = 100{\cdot}86$ (1)°, $V = 541{\cdot}4$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}55$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}11$ cm^{-1}, $F(000) = 264$, $T = 291$ K, $R = 0{\cdot}040$ for 1415 reflections with $|F_o| > 4\sigma(F)$. 4,7-Dinitroso-4,5,6,7-tetrahydro-1,2,5-oxadiazolo[3,4-*b*]pyrazine, (II), $C_4H_4N_6O_3$, $M_r = 184{\cdot}12$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}151$ (1), $b = 9{\cdot}526$ (2), $c = 12{\cdot}573$ (2) Å, $V = 736{\cdot}6$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}66$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 12{\cdot}03$ cm^{-1}, $F(000) = 376$, $T = 291$ K, $R = 0{\cdot}044$ for 901 reflections with $|F_o| > 4\sigma(F)$. 4-Nitroso-4,5,6,7-tetrahydro-1,2,5-oxadiazolo[3,4-*b*]pyrazine, (III), $C_4H_5N_5O_2$, $M_r = 155{\cdot}12$, monoclinic, $P2_1/c$, $a = 6{\cdot}278$ (1), $b = 9{\cdot}902$ (2), $c = 10{\cdot}650$ (2) Å, $\beta = 106{\cdot}02$ (2)°, $V = 636{\cdot}4$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}62$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}25$ cm^{-1}, $F(000) = 320$, $T = 291$ K, $R = 0{\cdot}046$ for 1126 reflections with $|F_o| > 4\sigma(F)$. 4,7-Dinitro-4,5,6,7-tetrahydro-1,2,5-oxadiazolo[3,4-*b*]pyrazine, (IV), $C_4H_4N_6O_5$, $M_r = 216{\cdot}11$, monoclinic, $P2_1/c$, $a = 9{\cdot}743$ (4), $b = 6{\cdot}636$ (2), $c = 12{\cdot}165$ (4) Å, $\beta = 94{\cdot}22$ (3)°, $V = 784{\cdot}4$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}83$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}57$ cm^{-1}, $F(000) = 440$, $T = 291$ K, $R = 0{\cdot}055$ for 1412 reflections with $|F_o| > 4\sigma(F)$. The molecular geometries of all four compounds are very similar. The furazan moiety is planar and shortening of the C—N bonds to the piperazine nitrogen caused by ring fusion is observed. All four molecules exhibit twisting about the bond between the two methylene carbons to achieve a distorted conformation in the six-membered ring. In (I) and (IV) coordination about the piperazine nitrogen is pyramidal whereas in (II) the coordination about the piperazine nitrogen becomes planar owing to the nitrosamine groups. The mononitrosamine (III) exhibits disorder in the rings that may be due to synchronous ring twists as well as exhibiting rotational disorder of the nitrosamine group. (II) exhibits no disorder of the nitrosamine groups. Molecules of (I) link together with hydrogen bonding to form chains. (III) crystallizes as hydrogen-bonded dimers. Only one form of the nitramine (IV) could be obtained as crystals suitable for a structure determination.

Introduction. Although the furazan (1,2,5-oxadiazolo) moiety is well-known in the literature (Boyer, 1986), few studies of compounds with furazan rings fused to saturated heterocyclic rings have been reported. As little is know about molecular geometry and conformation of these types of compounds, four related furazanopiperazines (4,5,6,7-tetrahydro-1,2,5-oxadiazolo[3,4-*b*]pyrazines) (I)–(IV), are reported here. However, the dinitro derivative, (IV), is of particular interest. Empirical density calculations (Cichra, Holden & Dickinson, 1980) and empirical detonation velocity calculations (Rothstein & Petersen, 1979) predict that nitrated derivatives of heterocyclic rings fused to furazan moieties might be of interest as potential explosive or propellant ingredients. A very early structure determination of (IV), crystallized from acetone/water,

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