

1982) so that neither compound falls into the group of C-nitroso compounds that exhibit weak intra- or intermolecular bonding.

The N(10)—O(11) bond in the nitroso group [1.213 (5) Å] is the shortest yet to be reported in a C-nitroso compound, just significantly shorter than that found in ethyl *trans-m*-nitrosocinnamate [1.229 (3) Å] (Talberg, 1978). The cinnamate, like (I), is a compound in which the nitroso group is not involved in 'extended mutual conjugation' (Talberg, 1978). The C(6)—N(10) bond, 1.419 (5) Å, is not as long as would be predicted from Talberg's (1979*a*) regression relationship between N—O and C—N bond lengths. The endocyclic angle at C(6), 123.7 (5)°, is greater than in the cinnamate, but in benzothiophene without substituents at C(6) (Pascard & Pascard-Billy, 1972) the endocyclic angle is already greater than 120°, so that the large angle probably represents the summation of the benzothiophene distortion and the effect of the nitroso substituent.

The dimensions of the rest of the molecule are unexceptional.

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## 2-Methyl-6-nitroso-7-phenethyl-1,3-benzothiazole

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**Abstract.** C<sub>16</sub>H<sub>14</sub>N<sub>2</sub>OS, *M<sub>r</sub>* = 282.37, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 9.355 (4), *b* = 7.403 (4), *c* = 20.665 (6) Å, β = 87.14 (3)°, *U* = 1429.4 Å<sup>3</sup>, *Z* = 4, *D<sub>c</sub>* = 1.31 Mg m<sup>-3</sup>, Mo *K*α radiation (λ = 0.71069 Å), μ = 0.2219

mm<sup>-1</sup>, 534 independent reflections, *R* = 0.043. The asymmetric unit contains one molecule of a nitroso monomer; bond lengths C—NO 1.41 (2), CN—O 1.23 (1) Å.

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**Introduction.** The structure was determined as part of a study of nitroso compounds [see Prout & Miao (1982), and references therein].

The experimental procedures were closely similar to those used previously (Prout & Miao, 1982). 2231 reflections were obtained from a crystal  $0.1 \times 0.2 \times 0.3$  mm; 534 reflections with  $I > 3\sigma(I)$  were used in the refinement. These were corrected for Lorentz and polarization effects, but not absorption. The structure was solved with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1979) from 160 reflections with  $1.69 < E < 4.03$  and refined by full-matrix least squares. All H atoms except H(181) were located from a series of difference syntheses at  $R$  values between 0.068 and 0.054. The refinement converged at  $R = 0.043$ ,  $R_w = 0.046$ . All calculations were performed with *CRYSTALS* (Carruthers & Watkin, 1980). Final atomic parameters are given in Table 1;\*

\* Lists of structure factors and anisotropic thermal parameters for the non-hydrogen atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36354 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional parameters ( $\times 10^4$ ) and isotropic temperature factors ( $\times 10^4$ , for H  $\times 10^3$ ) (equivalent values for heavy atoms, mean e.s.d.  $0.008 \text{ \AA}^2$ )

$U_{eq} = (U_1U_2U_3)^{1/3}$ , where  $U_1$ ,  $U_2$ ,  $U_3$  are the mean-square displacements ( $\text{\AA}^2$ ) along the principal axes of the thermal ellipsoid.

	$x$	$y$	$z$	$U_{eq}/U$ ( $\text{\AA}^2$ )
S(1)	2055 (3)	-237 (4)	4886 (2)	733
C(2)	2962 (15)	-1846 (15)	4400 (6)	728
N(3)	3956 (11)	-2728 (11)	4678 (5)	673
C(4)	4065 (14)	-2170 (13)	5323 (6)	548
C(5)	5005 (11)	-2862 (13)	5761 (6)	611
C(6)	5003 (13)	-2174 (15)	6379 (6)	713
C(7)	4020 (15)	-798 (13)	6569 (6)	652
C(8)	3047 (11)	-67 (14)	6148 (6)	570
C(9)	3106 (11)	-807 (13)	5523 (5)	490
C(10)	2525 (15)	-2084 (18)	3724 (6)	1070
C(11)	2013 (13)	1393 (13)	6340 (6)	707
C(12)	2644 (12)	3294 (12)	6170 (6)	669
C(13)	1526 (12)	4760 (14)	6338 (7)	662
C(14)	1546 (12)	5698 (15)	6909 (6)	675
C(15)	522 (18)	6979 (17)	7059 (6)	906
C(16)	-526 (14)	7307 (17)	6654 (8)	878
C(17)	-564 (16)	6390 (22)	6091 (8)	1062
C(18)	475 (16)	5109 (17)	5930 (6)	845
N(19)	3975 (12)	-54 (13)	7195 (5)	821
O(20)	4922 (13)	-594 (12)	7541 (5)	1173
H(101)	2335 (77)	-890 (42)	3537 (15)	94 (17)
H(102)	3305 (45)	-2661 (74)	3468 (13)	117 (21)
H(103)	3305 (45)	-2661 (74)	3468 (13)	117 (21)
H(111)	1765 (85)	1342 (50)	6814 (16)	72 (15)
H(112)	1134 (64)	1216 (59)	6104 (35)	128 (16)
H(121)	3553 (41)	3626 (69)	6361 (37)	85 (14)
H(122)	2796 (68)	3227 (87)	5698 (15)	92 (15)
H(141)	2349 (72)	5521 (111)	7199 (30)	93 (13)
H(151)	540 (80)	7642 (123)	7477 (30)	90 (16)
H(161)	-1337 (57)	8092 (102)	6796 (23)	58 (15)
H(171)	-1358 (72)	6578 (111)	5808 (30)	66 (14)
H(181)	452 (73)	4462 (117)	5518 (23)	79 (14)
H(51)	5735 (55)	-3733 (94)	5616 (16)	54 (14)
H(61)	5738 (78)	-2589 (101)	6685 (22)	66 (14)

Table 2. Bond distances ( $\text{\AA}$ ) and interbond angles ( $^\circ$ )

S(1)-C(2)	1.75 (2)	C(7)-N(19)	1.41 (2)
C(2)-N(3)	1.29 (2)	N(19)-O(20)	1.23 (1)
N(3)-C(4)	1.40 (2)	C(8)-C(11)	1.49 (2)
C(4)-C(5)	1.39 (2)	C(11)-C(12)	1.56 (2)
C(5)-C(6)	1.38 (2)	C(12)-C(13)	1.54 (2)
C(6)-C(7)	1.42 (2)	C(13)-C(14)	1.37 (2)
C(7)-C(8)	1.40 (2)	C(14)-C(15)	1.37 (2)
C(8)-C(9)	1.40 (2)	C(15)-C(16)	1.34 (2)
C(9)-C(4)	1.40 (2)	C(16)-C(17)	1.35 (2)
C(9)-S(1)	1.73 (1)	C(17)-C(18)	1.39 (2)
C(2)-C(10)	1.49 (2)	C(18)-C(13)	1.35 (2)
C(2)-S(1)-C(9)	89.6 (6)	S(1)-C(9)-C(4)	109.3 (10)
S(1)-C(2)-N(3)	115.3 (10)	C(8)-C(11)-C(12)	111.2 (11)
S(1)-C(2)-C(10)	118.2 (10)	C(11)-C(12)-C(13)	109.9 (10)
N(3)-C(2)-C(10)	126.6 (13)	C(12)-C(13)-C(14)	121.0 (11)
C(2)-N(3)-C(4)	111.2 (12)	C(12)-C(13)-C(18)	120.1 (12)
N(3)-C(4)-C(5)	126.0 (12)	C(14)-C(13)-C(18)	118.9 (10)
N(3)-C(4)-C(9)	114.6 (11)	C(13)-C(14)-C(15)	120.3 (9)
C(5)-C(4)-C(9)	119.4 (11)	C(14)-C(15)-C(16)	120.5 (9)
C(4)-C(5)-C(6)	119.8 (9)	C(15)-C(16)-C(17)	119.8 (10)
C(5)-C(6)-C(7)	119.7 (8)	C(16)-C(17)-C(18)	120.2 (9)
C(6)-C(7)-C(8)	122.7 (11)	C(13)-C(18)-C(17)	120.3 (9)
C(6)-C(7)-N(19)	121.8 (11)	C(7)-N(19)-O(20)	114.7 (12)
C(8)-C(7)-N(19)	115.5 (12)		
C(7)-C(8)-C(9)	115.2 (11)		
C(7)-C(8)-C(11)	123.2 (11)		
C(9)-C(8)-C(11)	121.6 (10)		
C(8)-C(9)-S(1)	127.4 (9)		
C(4)-C(9)-C(8)	123.3 (10)		

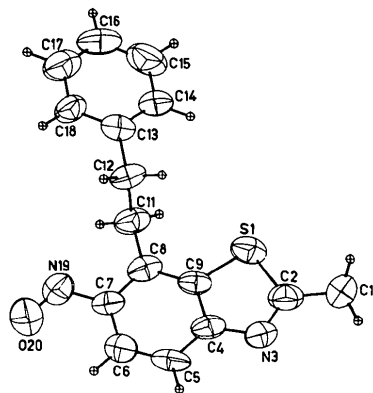


Fig. 1. A three-dimensional 50% probability thermal-ellipsoid plot (Davies, 1981) showing the atom numbering.

Table 2 lists interatomic distances and interbond angles with e.s.d.'s calculated from the variance-covariance matrix.

**Discussion.** The crystal structure confirms that the molecule is a C-nitroso monomer (Fig. 1). The nitroso group, like that in 5-nitroso-4-phenethylbenzo[b]thiophene (Prout & Miao, 1982), does not take part in weak bonding interactions and makes no intra- or intermolecular contacts significantly less than the sum of the van der Waals radii. Its shortest intramolecular non-bonded contacts are N(19)···H(111) 2.48 and O(20)···H(61) 2.40  $\text{\AA}$ , and the shortest intermolec-

ular contact is 2.71 Å from O(20) to H(102) of a neighbouring molecule. The molecular dimensions are unexceptional.

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## Riolozatrione

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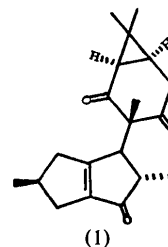
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**Abstract.** C<sub>20</sub>H<sub>26</sub>O<sub>3</sub>,  $M_r = 314.42$ , orthorhombic,  $P2_12_12_1$ ,  $a = 11.404$  (3),  $b = 21.090$  (8),  $c = 7.506$  (3) Å,  $V = 1805$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $d_c = 1.157$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54178$  Å. A full-matrix least-squares refinement (nonhydrogen atoms anisotropic, H atoms isotropic) based on 1586 reflexions led to a final  $R$  of 0.053. Riolozatrione represents a new diterpene structural type whose skeleton was named riolozane. The structure is composed of two five-membered rings sharing a common double bond. One five-membered ring exhibits a flattened envelope conformation while the other containing an  $\alpha, \beta$ -unsaturated ketone moiety is more planar. The double bond deviates from planarity by 6.5°. A cyclohexanedione moiety containing a fused cyclopropane ring is attached to the five-membered ring containing the keto function. The six-membered ring exhibits a 1,2-diplanar conformation.

**Introduction.** *Jatropha dioica* var. *sessiliflora* (Hook) McVaugh (*J. spathulata*) (Standley, 1923; Vines, 1960) is a shrub found in the arid regions of Northeastern Mexico. It is known as 'Sangre-drago' or 'Sangre de Drago' and was called 'tlapelex patli' by the Aztecs. The red root juice is used in folk medicine and the roots are chewed to relieve toothache (Martinez, 1959). Root extracts exhibit antibiotic activity against *Staphylococcus aureus* and the new diterpene riolozatrione (1) was isolated from the active

fraction (Dominguez, Cano, Franco, Villarreal, Watson & Zabel, 1980). The structure was determined by X-ray diffraction techniques and represents a new diterpene structural type for which the name riolozane was proposed.



A crystal of dimensions 0.7 × 0.12 × 0.35 mm was used to collect intensity data on a Syntex P2<sub>1</sub> diffractometer system by the  $\theta:2\theta$  scanning technique using a variable scan speed and a graphite monochromator. Room-temperature lattice parameters were refined by a least-squares procedure utilizing 15 reflexions whose angles were measured by a centering routine associated with the diffractometer. Systematic absences were consistent with space group  $P2_12_12_1$ . A periodically monitored reflexion showed no significant change in intensity. Of the 1838 independent reflexions measured, 250 had intensities less than  $2\sigma(I)$ . Two additional reflexions showed significant secondary extinction and were dropped from the final refinement. Lorentz and polarization corrections were applied, but no absorption corrections were made.

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