

Fig. 1. The molecular structure of  $C_{10}H_{14}N_2O_2$  showing the numbering scheme of the atoms.

block-diagonal least squares, H atoms at calculated positions not refined, fixed isotropic temperature parameter of  $4.0 \text{ \AA}^2$ , anisotropic thermal parameters for heavy atoms, weighting scheme  $w^{-1} = \sigma^2(F) + (0.02|F_o|)^2$ ,  $\sigma(F)$  from counting statistics,  $(\Delta/\sigma)_{\max} = 0.09$ ,  $R = 0.053$  and  $0.063$ ,  $wR = 0.067$  and  $0.069$  for observed and all reflections, respectively,  $S = 2.01$ ,  $(\Delta\rho)_{\max} = 0.13$ ,  $(\Delta\rho)_{\min} = -0.19 \text{ e \AA}^{-3}$ . Scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974). All calculations with XRC83 (Pavelčík, Kettmann & Majer, 1985) on an ES-1045 computer. Final atomic coordinates are given in Table

*Acta Cryst.* (1989), **C45**, 957–959

## Structure of Recinine\*

BY M. SORIANO-GARCÍA,† M. JIMÉNEZ E., R. REYES VACA AND R. A. TOSCANO

*Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán 04510, Mexico DF*

(Received 28 June 1988; accepted 17 November 1988)

**Abstract.** 4-Methoxy-1-methyl-2-oxo-3-pyridine-carbonitrile,  $C_{16}H_{16}N_4O_4$ ,  $M_r = 328.3$ , monoclinic,  $P2_1/n$ ,  $a = 7.357$  (3),  $b = 14.620$  (6),  $c = 14.838$  (6)  $\text{\AA}$ ,  $\beta = 94.15$  (3) $^\circ$ ,  $V = 1592$  (1)  $\text{\AA}^3$ ,  $Z = 8$ ,  $D_x = 1.37 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.7107 \text{ \AA}$ ,  $\mu = 0.094 \text{ mm}^{-1}$ ,  $F(000) = 688$ ,  $T = 293 \text{ K}$ ,  $R = 0.059$  for 1787 observed reflections. The X-ray study confirms that the structure of recinine is as inferred from chemical evidence. There are two crystallographically independent molecules and in both cases the six-membered ring is planar to within  $0.012 \text{ \AA}$  and the  $-\text{C}-\text{C}\equiv\text{N}$

fragments are linear. The largest bond-length discrepancy between molecules *A* and *B* is  $0.016 \text{ \AA}$  ( $4\sigma$ ) for C(4)–O(2). The two C=O carbonyl distances [mean  $1.232$  (4)  $\text{\AA}$ ] are both slightly longer than normal. The methoxy groups at C(4) are coplanar with the six-membered ring. The molecules in the crystal are packed at normal van der Waals distances. Five C–H...O/N intermolecular contacts  $< 3.4 \text{ \AA}$  are noted.

**Experimental.** In the state of Oaxaca, Mexico, castor oil is obtained by crushing the seeds of the *Recinus communis* or castor-oil plant using stone mills. A by-product of this process is a paste that is sold at a low

1.\* Interatomic distances, bond angles and torsion angles are presented in Table 2. Fig. 1 shows the molecule and the atom-numbering scheme.

**Related literature.** An overview of the conformations of pyrrolidine derivatives has been published by Pfafferott, Oberhammer, Boggs & Caminati (1985).

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51600 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CHI 2HU, England.

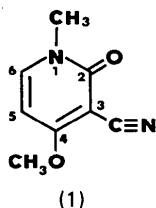
## References

- HALADOVÁ, M., EISENREICHOVÁ, E., BUČKOVÁ, A., TOMKO, J. & UHRIN, D. (1988). *Collect. Czech. Chem. Commun.* **53**, 157–160.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- PAVELČÍK, F., KETTMANN, V. & MAJER, J. (1985). *Chem. Pap.* **39**, 467–472.
- PFÄFFEROTT, G., OBERHAMMER, H., BOGGS, J. E. & CAMINATTI, W. (1985). *J. Am. Chem. Soc.* **107**, 2305–2309.

\* Contribution No. 957 of the Instituto de Química, UNAM.

† To whom correspondence should be addressed.

price and used as a fertilizing product. Recinine (1) was extracted from the by-product paste with petroleum ether. Subsequent recrystallization from ethanol gave



deep-red crystals, m.p. 474 K. Crystal size  $0.23 \times 0.34 \times 0.40$  mm. Nicolet R3 four-circle diffractometer. Lattice parameters from 25 machine-centred reflections with  $3.9 < 2\theta < 17.4^\circ$ . Mo K $\alpha$  radiation. 2819 independent reflections with  $3 < 2\theta < 50^\circ$ , 1787 with  $I > 2.5\sigma(I)$ , index range  $h \pm 8, k 0 \rightarrow 17, l 0 \rightarrow 17$ ,  $2\theta/\theta$  scan mode, variable scan speed, scan width  $1.0^\circ$  ( $\theta$ ), two standard reflections (212, 040) monitored every 50 measurements, Lp correction, absorption ignored and  $R_{int} = 0.043$ . Structure solved by direct methods using *SHELXTL* (Sheldrick, 1985). Least-squares refinement of all non-H atoms with anisotropic

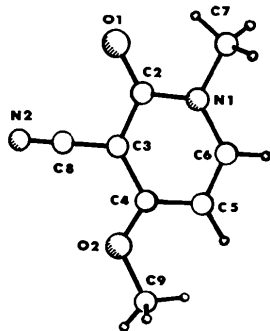


Fig. 1. Structure of molecule A of (1) showing the atom labelling.

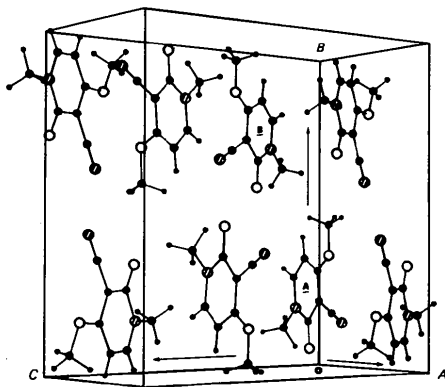


Fig. 2. A perspective drawing of the packing arrangement.

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )

$$U_{eq} = (U_{11} \times U_{22} \times U_{33})^{1/3}$$

	x	y	z	$U_{eq}$
N(1A)	4988 (4)	1967 (2)	3011 (2)	45 (1)
C(2A)	4261 (4)	1537 (2)	2228 (2)	43 (1)
C(3A)	3584 (4)	2145 (2)	1522 (2)	39 (1)
C(4A)	3662 (4)	3090 (2)	1611 (2)	43 (1)
C(5A)	4377 (5)	3462 (2)	2429 (2)	52 (1)
C(6A)	5001 (5)	2893 (2)	3092 (2)	49 (1)
C(7A)	5752 (5)	1391 (3)	3761 (2)	59 (1)
O(1A)	4244 (4)	694 (1)	2171 (2)	63 (1)
C(8A)	2830 (4)	1746 (2)	701 (2)	43 (1)
N(2A)	2225 (4)	1425 (2)	40 (2)	58 (1)
O(2A)	3018 (4)	3573 (2)	898 (1)	60 (1)
C(9A)	3089 (7)	4561 (2)	943 (3)	78 (2)
N(1B)	5967 (4)	1682 (2)	8937 (2)	46 (1)
C(2B)	6350 (4)	2536 (2)	9343 (2)	44 (1)
C(3B)	7309 (4)	2490 (2)	10218 (2)	41 (1)
C(4B)	7811 (4)	1670 (2)	10627 (2)	42 (1)
C(5B)	7398 (5)	843 (2)	10169 (2)	50 (1)
C(6B)	6492 (5)	886 (2)	9351 (2)	50 (1)
C(7B)	4923 (5)	1663 (3)	8061 (2)	63 (1)
O(1B)	5875 (4)	3243 (2)	8945 (2)	64 (1)
C(8B)	7711 (5)	3340 (2)	10670 (2)	50 (1)
N(2B)	8038 (5)	4018 (2)	11034 (2)	73 (1)
O(2B)	8636 (3)	1718 (1)	11466 (1)	52 (1)
C(9B)	9176 (5)	881 (2)	11920 (2)	60 (1)

Table 2. Molecular geometry of (1)

E.s.d.'s are given in parentheses.

	Molecule A	Molecule B
(a) Bond lengths ( $\text{\AA}$ )		
N(1)—C(2)	1.394 (4)	1.406 (4)
N(1)—C(6)	1.358 (4)	1.358 (4)
N(1)—C(7)	1.474 (4)	1.462 (4)
C(2)—C(3)	1.434 (4)	1.433 (4)
C(2)—O(1)	1.236 (4)	1.229 (4)
C(3)—C(4)	1.390 (4)	1.381 (4)
C(3)—C(8)	1.426 (4)	1.434 (4)
C(4)—C(5)	1.397 (4)	1.410 (4)
C(4)—O(2)	1.330 (4)	1.346 (4)
C(5)—C(6)	1.344 (4)	1.342 (5)
C(8)—N(2)	1.148 (4)	1.145 (4)
O(2)—C(9)	1.447 (4)	1.439 (4)
(b) Valence angles ( $^\circ$ )		
C(2)—N(1)—C(6)	121.5 (3)	121.7 (3)
C(2)—N(1)—C(7)	118.3 (3)	118.2 (3)
C(6)—N(1)—C(7)	120.2 (3)	120.0 (3)
N(1)—C(2)—C(3)	114.9 (3)	114.6 (3)
N(1)—C(2)—O(1)	120.5 (3)	120.0 (3)
C(3)—C(2)—O(1)	124.5 (3)	125.4 (3)
C(2)—C(3)—C(4)	122.5 (3)	122.5 (3)
C(2)—C(3)—C(8)	117.6 (3)	117.0 (3)
C(4)—C(3)—C(8)	119.9 (3)	120.4 (3)
C(3)—C(4)—C(5)	118.7 (3)	119.3 (3)
C(3)—C(4)—O(2)	116.2 (3)	116.7 (3)
C(5)—C(4)—O(2)	125.1 (3)	123.9 (3)
C(4)—C(5)—C(6)	118.8 (3)	118.1 (3)
N(1)—C(6)—C(5)	123.6 (3)	123.7 (3)
C(3)—C(8)—N(2)	180.0 (5)	179.8 (5)
C(4)—O(2)—C(9)	118.9 (3)	118.6 (2)
(c) Intermolecular approaches ( $\text{\AA}$ ) $< 3.4 \text{\AA}$		
C(5B) ... N(2A)	(1-x, -y, 1-z)	3.34 (1)
C(6B) ... O(1A)	(1-x, -y, 1-z)	3.25 (1)
C(9A) ... O(1B)	(1-x, 1-y, 1-z)	3.30 (1)
C(7B) ... N(2B)	(-0.5+x, 0.5-y, -0.5+z)	3.37 (1)
C(6A) ... N(2A)	(0.5+x, 0.5-y, 0.5+z)	3.37 (1)

thermal parameters; H atoms of CH and CH<sub>3</sub> groups riding on bonded C with a fixed isotropic temperature factor  $U = 0.06 \text{ \AA}^2$ ,  $\sum w(\Delta F)^2$  minimized,  $w = [\sigma^2(F_o) + 0.00224(F_o)^2]^{-1}$ , where  $\sigma$  is the standard deviation of observed amplitudes based on counting statistics. In the last cycle ( $\Delta/\sigma$ )<sub>max</sub> = 0.102;  $\Delta\rho$  from -0.26 to 0.24 e  $\text{\AA}^{-3}$ ; final  $R = 0.059$ ,  $wR = 0.076$  and  $S = 1.16$ . Scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a Nova 4S computer and plots drawn on a Tektronix plotter with the *SHELXTL* system of programs.

Atomic coordinates are given in Table 1.\* A perspective view of the molecule and the atom labelling are shown in Fig. 1. Bond distances, angles and intermolecular approaches are listed in Table 2. The arrangement of the molecules in the unit cell is shown in Fig. 2.

\* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51627 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Related literature.** Recinine was first isolated from the seeds of *Recinus communis* by Tuson (1864) and was chemically characterized by Späth & Koller (1923, 1925). It was of interest to determine the crystal structure of (1) to ascertain its conformation and molecular geometry.

Financial support from the Consejo Nacional de Ciencia y Tecnología de México, CONACYT (Project No. PCEXECNA-040603) is acknowledged. We also thank Mr Abelardo Cuellar for technical assistance.

#### References

- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)  
 SHELDRICK, G. M. (1985). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Version 5. Univ. of Göttingen, Federal Republic of Germany.  
 SPÄTH, E. & KOLLER, G. (1923). *Ber. Dtsch. Chem. Ges.* **56**, 2454–2460.  
 SPÄTH, E. & KOLLER, G. (1925). *Ber. Dtsch. Chem. Ges.* **58**, 2124–2126.  
 TUSON, R. V. (1864). *J. Chem. Soc.* **17**, 195–197.

*Acta Cryst.* (1989), **C45**, 959–961

## Structures of Sulfur Analogues of Precocenes. I. 2,2,6-Trimethyl-2H-1-benzothiopyran 1,1-Dioxide

BY E. E. CASTELLANO, C. A. DE SIMONE\* AND J. ZUKERMAN-SCHPECTOR\*

*Instituto de Física e Química de São Carlos, Universidade de São Paulo, Caixa Postal 369, 13560 São Carlos, SP, Brazil*

AND J. TERCIO B. FERREIRA AND V. CATANI

*Departamento de Química, Universidade Federal de São Carlos, Caixa Postal 676, 13560 São Carlos, SP, Brazil*

(Received 9 September 1988; accepted 28 November 1988)

**Abstract.** C<sub>12</sub>H<sub>14</sub>O<sub>2</sub>S,  $M_r = 222.30$ , orthorhombic,  $P2_12_12_1$ ,  $a = 7.357$  (2),  $b = 10.186$  (3),  $c = 15.253$  (2)  $\text{\AA}$ ,  $V = 1143.0$  (8)  $\text{\AA}^3$ ,  $Z = 4$ ,  $D_x = 1.292$  g  $\text{cm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71073$   $\text{\AA}$ ,  $\mu = 2.48$   $\text{cm}^{-1}$ ,  $F(000) = 472$ ,  $T = 298$  K,  $R = 0.050$  for 663 observed reflections. The S atom is tetrahedral with a dihedral angle between planes C–S–C and O–S–O of 89.4 (4)°. The S–C(sp<sup>2</sup>) and S–C(sp<sup>3</sup>) bond distances are 1.751 (8) and 1.799 (9)  $\text{\AA}$  respectively.

\* Permanent address: Laboratório de Cristalografia e Físico-Química dos Materiais, CCEN, Universidade Federal de Alagoas, 57000 Maceió, AL, Brazil.

**Experimental.** The synthesis of compound (1) has been reported previously (Ferreira & Catani, 1987). The data-collection and refinement parameters are summarized in Table 1. The structure was solved using standard direct methods and difference Fourier techniques. In final cycles of full-matrix least-squares refinement all non-H atoms anisotropic. H atoms included at positions found in difference synthesis, all with a common isotropic temperature factor that refined to  $U = 0.11$   $\text{\AA}^2$ . Scattering factors for non-H atoms from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970), for H from Stewart, Davidson & Simpson