

Table 2. Bond lengths (Å) and valence angles (°)

C(1)—C(7)	1.450 (3)	C(1)—C(2)	1.425 (3)
C(1)—C(6)	1.387 (3)	C(7)—O(7)	1.214 (3)
C(2)—O(2)	1.287 (3)	C(2)—C(3)	1.424 (3)
C(3)—C(8)	1.452 (3)	C(3)—C(4)	1.381 (4)
C(8)—O(8)	1.207 (3)	C(4)—C(5)	1.378 (3)
C(5)—C(9)	1.507 (4)	C(5)—C(6)	1.372 (3)
N(10)—C(11)	1.481 (3)	N(10)—C(18')	1.486 (3)
C(11)—C(12)	1.496 (4)	C(12)—O(13)	1.413 (2)
O(13)—C(14)	1.423 (4)	C(14)—C(15)	1.485 (3)
C(15)—O(16)	1.417 (3)	O(16)—C(17)	1.413 (2)
C(17)—C(18)	1.480 (4)	C(18)—N(10')	1.486 (3)
C(7)—C(1)—C(2)	120.6 (2)	C(7)—C(1)—C(6)	119.4 (2)
C(2)—C(1)—C(6)	120.0 (2)	C(1)—C(7)—O(7)	125.3 (3)
C(1)—C(2)—O(2)	122.8 (2)	C(1)—C(2)—C(3)	116.2 (2)
O(2)—C(2)—C(3)	121.1 (2)	C(2)—C(3)—C(8)	119.7 (2)
C(2)—C(3)—C(4)	120.4 (2)	C(8)—C(3)—C(4)	119.8 (2)
C(3)—C(8)—O(8)	125.6 (3)	C(3)—C(4)—C(5)	123.4 (2)
C(4)—C(5)—C(9)	122.3 (2)	C(4)—C(5)—C(6)	116.2 (2)
C(9)—C(5)—C(6)	121.5 (2)	C(1)—C(6)—C(5)	123.8 (2)
C(11)—N(10)—C(18')	114.8 (2)	N(10)—C(11)—C(12)	110.1 (2)
C(11)—C(12)—O(13)	108.3 (2)	C(12)—O(13)—C(14)	111.9 (2)
O(13)—C(14)—C(15)	109.4 (2)	C(14)—C(15)—O(16)	109.7 (2)
C(15)—O(16)—C(17)	113.0 (2)	O(16)—C(17)—C(18)	107.8 (2)
C(17)—C(18)—N(10')	109.7 (2)		

Computer programs for Desktop 30 Microclipline and Nova 4/C configuration (Nicolet Instrument Corporation, 1986); scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic positional parameters and  $U_{eq}$  values while Table 2 gives bond lengths and bond angles. Fig. 1 is a drawing of the title compound.\*

\* Lists of H-atom coordinates, anisotropic thermal parameters, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44923 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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### Structure of (Z)-Methyl 3-[(E)-(2-Cyano-1-phenylethylidene)-amino]-3-methylthiopropenoate

BY J. L. BALCÁZAR

*Department of Geology, University of Alcalá de Henares, Spain*

AND F. FLORENCIO AND S. GARCÍA-BLANCO

*X-ray Department, Institute Rocasolano, CSIC, 28006-Madrid, Spain*

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**Abstract.** C<sub>14</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub>S,  $M_r = 242.277$ , triclinic,  $P\bar{1}$ ,  $a = 8.019$  (3),  $b = 9.858$  (3),  $c = 10.295$  (3) Å,  $\alpha = 115.26$  (3),  $\beta = 99.24$  (3),  $\gamma = 90.63$  (2)°,  $V = 723.6$  (5) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.259$ ,  $D_x = 1.111$  Mg m<sup>-3</sup>,

**Related literature.** The interactions between crown macrocycles and organic host molecules have been of increasing interest during the past decade and have been the subject of several reviews (Vögtle, Sieger & Müller, 1981; Vögtle, Müller & Watson, 1984; Atwood, Davies & MacNicol, 1984). The structures of ionic complexes of diaza-18-crown-6 with tropolone and 4-hydroxy-3-methoxybenzaldehyde have been reported (Watson, Vögtle & Müller, 1988). The conformations of complexed crown ethers in the solid state have been investigated and are the subject of a number of reports (e.g. Elbasyouny *et al.*, 1983; Watson, Galloy, Grossie, Vögtle & Müller, 1984).

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Mo  $K\alpha$  radiation,  $\lambda = 0.7107$  Å,  $\mu = 0.21$  mm<sup>-1</sup>, room temperature,  $F(000) = 288$ , final  $R = 0.065$  and  $wR = 0.085$  for 3318 observed reflections. There is no electronic conjugation in the molecular chain with a

torsion angle  $\angle \text{C}=\text{N}-\text{C}=\text{C} <$  of  $-82.0(3)^\circ$ . The  $\text{H}_3\text{C}-\text{S}-\text{C}(\text{CH}_3)=\text{N}-\text{C}=\text{C}$  part is planar and the dihedral angle between this plane and that of the benzene ring is  $51.3(4)^\circ$ . The conformation is *E* with respect to the  $\text{C}=\text{N}$  and *Z* with respect to the  $\text{C}=\text{C}$  bond. Intermolecular distances correspond to van der Waals contacts.

**Experimental.** The title compound was prepared by Dr A. Lorente (Department of Organic Chemistry, University of Alcalá de Henares, Spain) as part of an investigation that we initiated in order to determine the influence of different functional groups on the central chain, and to discover its conformation and electron distribution. The structural study by X-ray diffraction was therefore undertaken on a transparent prismatic crystal  $0.15 \times 0.23 \times 0.47$  mm. Cell dimensions were obtained by least-squares refinement of 25 reflections,  $12 < \theta < 27^\circ$ , measured on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated  $\text{Mo K}\alpha$  radiation. Intensity measurements were made using the  $\omega-2\theta$  scan mode up to  $2 < \theta < 30^\circ$ ,  $h(11,-11)$ ,  $k(13,-13)$ ,  $l(0,14)$ . Two standard reflections were measured every 100 reflections with no intensity variation. 4647 unique reflections, 3318 independent reflections measured, 3308 considered as observed with  $I > 2\sigma(I)$ .  $R_{\text{int}} = 0.002$ . Lorentz, polarization and absorption corrections (transmission factors 1.418–0.517) (Walker & Stuart, 1983) were made.

Structure solved by direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); refined by full-matrix least squares with anisotropic and isotropic temperature factors for the non-H and H atoms (located by difference synthesis), respectively.  $\sum w(|F_o| - |F_c|)^2$  minimized.  $w = 1$  because an applied weighting scheme (Martínez-Ripoll & Cano, 1975) gave no trends in  $w\Delta^2$  vs  $\langle |F_o| \rangle$  or  $\langle \sin\theta/\lambda \rangle$ .

Final  $R = 0.065$ ,  $wR = 0.085$ ,  $S = 3.19$  for 172 variables, max. residual density  $0.17 \text{ e } \text{Å}^{-3}$ , max. shift  $0.056\sigma$ . Scattering factors from *International Tables*

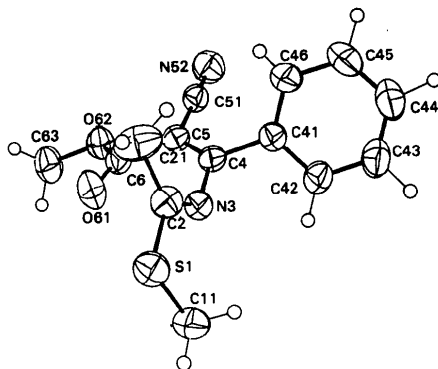


Fig. 1. A view of the molecule with the atomic labelling.

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j \cos(a_i, a_j).$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}} (\text{Å}^2 \times 10^4)$
S1	0.9734 (1)	0.2780 (1)	0.4996 (1)	609 (24)
C11	0.8519 (5)	0.3011 (4)	0.3495 (3)	696 (29)
C2	0.8616 (3)	0.3734 (3)	0.6397 (2)	488 (20)
C21	0.9368 (4)	0.3740 (5)	0.7837 (4)	767 (34)
N3	0.7296 (2)	0.4365 (2)	0.6168 (2)	473 (19)
C4	0.6361 (3)	0.5161 (2)	0.7222 (2)	418 (17)
C41	0.6666 (3)	0.6817 (2)	0.7772 (2)	463 (19)
C42	0.6633 (3)	0.7437 (3)	0.6787 (3)	560 (24)
C43	0.6948 (4)	0.8975 (3)	0.7278 (4)	698 (31)
C44	0.7315 (6)	0.9898 (3)	0.8723 (4)	920 (40)
C45	0.7349 (9)	0.9293 (4)	0.9710 (4)	1342 (61)
C46	0.7025 (6)	0.7761 (4)	0.9244 (3)	961 (42)
C5	0.5160 (2)	0.4491 (2)	0.7620 (2)	422 (18)
C51	0.4088 (3)	0.5400 (2)	0.8581 (2)	469 (20)
N52	0.3236 (3)	0.6099 (3)	0.9369 (3)	676 (28)
C6	0.4842 (3)	0.2834 (2)	0.7033 (2)	441 (18)
O61	0.5734 (3)	0.1927 (2)	0.6368 (2)	678 (28)
O62	0.3385 (2)	0.2460 (2)	0.7334 (2)	543 (22)
C63	0.2895 (4)	0.0865 (3)	0.6739 (4)	692 (29)

Table 2. *Bond distances (Å), bond angles (°) and torsion angles (°)*

S1–C11	1.792 (4)	S1–C2	1.746 (2)
C2–C21	1.505 (5)	C2–N3	1.273 (3)
N3–C4	1.379 (3)	C4–C41	1.484 (3)
C4–C5	1.370 (3)	C41–C42	1.387 (4)
C41–C46	1.377 (3)	C42–C43	1.383 (4)
C43–C44	1.352(5)	C44–C45	1.377 (7)
C45–C46	1.383 (5)	C5–C51	1.433 (3)
C5–C6	1.482 (3)	C51–N52	1.144 (3)
C6–O61	1.196 (3)	C6–O62	1.342 (3)
O62–C63	1.445 (3)		
C11–S1–C2	101.9 (2)	S1–C2–N3	120.7 (2)
S1–C2–C21	114.1 (2)	C21–C2–N3	125.3 (2)
C2–N3–C4	124.2 (2)	N3–C4–C5	123.3 (2)
N3–C4–C41	113.1 (2)	C41–C4–C5	123.3 (2)
C4–C41–C46	121.7 (2)	C4–C41–C42	119.6 (2)
C42–C41–C46	118.7 (3)	C41–C42–C43	120.5 (3)
C42–C43–C44	120.8 (3)	C43–C44–C45	119.2 (4)
C44–C45–C46	121.1 (3)	C41–C46–C45	119.8 (3)
C4–C5–C6	122.7 (2)	C4–C5–C51	119.8 (2)
C51–C5–C6	117.4 (2)	C5–C51–N52	178.0 (3)
C5–C6–O62	110.9 (2)	C5–C6–O61	125.8 (2)
O61–C6–O62	123.3 (2)	C6–O62–C63	115.8 (2)
C11–S1–C2–C21	–179.6 (3)	C11–S1–C2–N3	–0.2 (3)
S1–C2–N3–C4	–178.8 (2)	C21–C2–N3–C4	0.4 (4)
C2–N3–C4–C41	103.8 (3)	C2–N3–C4–C5	–82.0 (3)
N3–C4–C5–C51	–171.8 (2)	N3–C4–C5–C6	4.8 (4)
N3–C4–C41–C42	47.8 (3)	N3–C4–C41–C46	–130.5 (3)
C41–C4–C5–C51	1.8 (4)	C41–C4–C5–C6	178.4 (2)
C5–C4–C41–C46	55.3 (4)	C5–C4–C41–C42	–126.5 (3)
C4–C41–C46–C45	178.1 (4)	C4–C41–C42–C43	–178.7 (3)
C42–C41–C46–C45	–0.2 (5)	C46–C41–C42–C43	–0.3 (4)
C41–C42–C43–C44	0.9 (5)	C42–C43–C44–C45	–0.9 (6)
C43–C44–C45–C46	0.4 (7)	C44–C45–C46–C41	–0.2 (7)
C4–C5–C6–O61	12.9 (4)	C4–C5–C6–O62	–166.6 (2)
C51–C5–C6–O61	–170.4 (3)	C51–C5–C6–O62	10.1 (3)
C5–C6–O62–C63	176.5 (2)	O61–C6–O62–C63	–3.0 (4)

for X-ray Crystallography (1974). Computations by *XRAY70* system (Stewart, Kundell & Baldwin, 1970), *PARST* (Nardelli, 1983) and *PESOS* (Martínez-Ripoll & Cano, 1975), on a VAX 11/750 computer.

The structure of the title compound with the atomic labelling is shown in Fig. 1. Positional parameters and

the equivalent *U* values of the anisotropic temperature factors are given in Table 1.\* Bond lengths and angles and selected torsion angles are given in Table 2.

**Related literature.** Bond distances are comparable with those found in 2-[ $\alpha$ -(methylthio)benzylideneamino]-2-phenyl-1,1-ethylenedicarbonitrile (Balcázar, Florencio & García-Blanco, 1985), (3*E*,5*Z*,7*Z*)-8-(diphenylmethylene)amino-3,5,7-octatrien-2-one (Wong, 1978), and *N*-[bis(methylthio)methylene]cinnamamide (Kamath & Venkatesan, 1984).

\* 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 44906 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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