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Acta Cryst. (1990). **C46**, 1178–1179

Structure of 9-Oxo-1-thioxanthencarbonitrile 10,10-Dioxide

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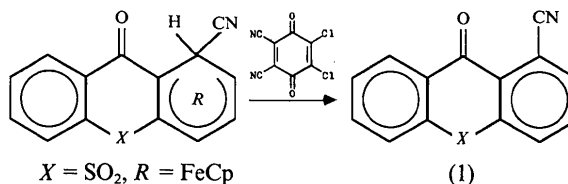
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(Received 27 October 1989; accepted 12 February 1990)

Abstract. $C_{14}H_7NO_3S$, $M_r = 269.3$, triclinic, $P\bar{1}$, $a = 8.069$ (3), $b = 8.654$ (3), $c = 8.973$ (3) Å, $\alpha = 102.12$ (3), $\beta = 91.16$ (3), $\gamma = 108.19$ (3)°, $V = 579.5$ (3) Å³, $Z = 2$, $D_x = 1.543$ g cm⁻³, Mo $K\alpha$ ($\lambda = 0.71073$ Å), $\mu = 2.68$ cm⁻¹, $F(000) = 276$, $T = 298$ K, $R = 0.0429$ and $wR = 0.0494$ for 1854 reflections [$I \geq 3\sigma(I)$]. The bonding between C(9) and C(9a) and C(8a) is asymmetrical. C(9)—C(9a) [1.499 (3) Å] is longer than C(9)—C(8a) [1.475 (3) Å] due to the electron-withdrawing power of the cyano group. A considerable repulsion between the carbonyl and the cyano groups is accommodated by distortion of the cyano group and the geometry around C(1). Angle C(11)—C(1)—C(9a) [122.4 (2)°] is larger than C(11)—C(1)—C(2) [116.2 (2)°]. Additionally, angle C(1)—C(11)—N(11) is 174.0 (2)°, compared to an angle of 180° around an *sp*-hybridized carbon, and the N atom is pointing away from the carbonyl group. The dihedral angle between the two arene rings is 170.1 (2)°.

Experimental. The title compound (1) was synthesized in a demetallation–rearomatization reaction summarized in the scheme below (Sutherland, Chowdhury, Piórko & Lee, 1987). Crystals were grown by slow evaporation from an Me_2SO-d_6 solution and a pale-yellow prism of dimensions $0.28 \times 0.32 \times 0.40$ mm was chosen for X-ray investigation. Data were collected on a Nicolet R3m/V diffractometer equipped with a graphite monochromator utilizing Mo $K\alpha$ radiation. 50 reflections with $15.55 \leq 2\theta \leq 34.99^\circ$ were used to refine the cell parameters. 5108 reflections were collected using the ω -scan method ($h, -10 \rightarrow 10$; $k, -11 \rightarrow 10$; $l, -11 \rightarrow 11$), 2670 unique reflections, $R_{int} = 0.0249$; 2θ range $3 \rightarrow 55^\circ$, $1.2^\circ \omega$ scan at $3-6^\circ \text{ min}^{-1}$, depending upon intensity. Four reflections (020, 112, 0 $\bar{2}$ 0, $\bar{1}\bar{1}\bar{2}$) were

measured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was $< 1.15\%$). Absorption corrections were applied based on measured crystal faces using *SHELXTL-Plus* (Sheldrick, 1987); minimum and maximum transmission 0.9934 and 0.9959.



The structure was solved by direct methods in *SHELXTL-Plus* from which the locations of all non-H atoms were obtained. The structure was refined [*SHELX76* (Sheldrick, 1976)] using full-matrix least squares and the positions of all H atoms were determined from a difference Fourier map. The non-H atoms were treated anisotropically, whereas the H atoms were refined with isotropic thermal parameters. 201 parameters were refined and $\sum w(|F_o| - |F_c|)^2$ was minimized; $w = 1/(\sigma|F_o|)^2$, $\sigma(F_o) = 0.5kI^{-1/2}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$, $I(\text{intensity}) = (I_{peak} - I_{background})(\text{scan rate})$, and $\sigma(I) = (I_{peak} + I_{background})^{1/2}(\text{scan rate})$, k is the correction due to decay and L_p effects, 0.02 is a factor used to down weight intense reflections and to account for instrument instability. An extinction correction $\chi = 0.0153$ (16) [where $F^* = F(1 + 0.002\chi F^2/\sin 2\theta)^{-1/4}$] was also applied (Sheldrick, 1987). Final $R = 0.0429$, $wR = 0.0494$ ($R_{all} = 0.0696$, $wR_{all} = 0.0567$) for 1854 reflections having $I \geq 3\sigma(I)$, and goodness-of-fit = 1.55. Maximum $\Delta/\sigma = 0.001$ in the final refinement cycle and the minimum and maximum peaks in the ΔF map were -0.31 and 0.44 e \AA^{-3} , respectively.

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{eq}(\text{Å}^2)$
S(10)	0.0001 (1)	0.2345 (1)	0.9039 (1)	0.041 (1)
O(9)	0.3912 (2)	0.0076 (3)	0.7327 (3)	0.076 (1)
O(10a)	-0.1285 (2)	0.2091 (3)	1.0129 (2)	0.058 (1)
O(10a)	0.0118 (2)	0.3615 (2)	0.8208 (2)	0.058 (1)
N(11)	0.2530 (3)	-0.3337 (3)	0.5044 (3)	0.075 (1)
C(1)	0.0442 (3)	-0.1702 (3)	0.6080 (3)	0.042 (1)
C(2)	-0.1274 (4)	-0.2434 (4)	0.5385 (3)	0.050 (1)
C(3)	-0.2540 (3)	-0.1690 (4)	0.5860 (3)	0.051 (1)
C(4)	-0.2119 (3)	-0.0274 (4)	0.6998 (3)	0.046 (1)
C(4a)	-0.0405 (3)	0.0448 (3)	0.7686 (3)	0.037 (1)
C(5)	0.2566 (3)	0.3993 (3)	1.1277 (3)	0.046 (1)
C(6)	0.4230 (3)	0.4441 (4)	1.2007 (3)	0.051 (1)
C(7)	0.5385 (3)	0.3601 (4)	1.1425 (3)	0.051 (1)
C(8)	0.4867 (3)	0.2341 (3)	1.0130 (3)	0.047 (1)
C(8a)	0.3209 (3)	0.1871 (3)	0.9363 (3)	0.038 (1)
C(9)	0.2766 (3)	0.0546 (3)	0.7945 (3)	0.044 (1)
C(9a)	0.0914 (3)	-0.0248 (3)	0.7236 (3)	0.038 (1)
C(10a)	0.2069 (3)	0.2731 (3)	0.9974 (3)	0.036 (1)
C(11)	0.1682 (4)	-0.2560 (3)	0.5540 (3)	0.053 (1)

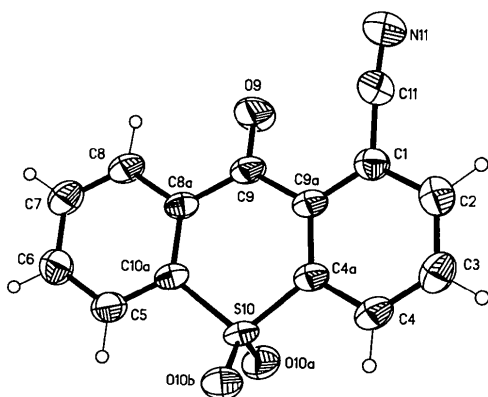


Fig. 1. Molecular structure of (1), with 50% probability ellipsoids, showing the atom-numbering scheme.

The linear absorption coefficient was calculated from values from *International Tables for X-ray Crystallography* (1974). Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with anomalous-dispersion corrections from Cromer & Liberman (1970), while those of H atoms were from Stewart, Davidson & Simpson (1965). The positional parameters and the equivalent isotropic thermal parameters of the non-H atoms are listed in Table 1; * bond lengths and angles are in Table 2. The thermal-ellipsoid drawing (*SHELXTL-Plus*) of the molecule with the atom-labeling scheme is given in Fig. 1.

* Lists of crystallographic data, structure-factor amplitudes, anisotropic thermal parameters, H-atom positional parameters and bond lengths and angles, and a packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52702 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and angles (°) of the non-H atoms

S(10)—O(10b)	1.439 (2)	S(10)—O(10a)	1.433 (2)
S(10)—C(4a)	1.757 (2)	S(10)—C(10a)	1.755 (2)
O(9)—C(9)	1.221 (3)	N(11)—C(11)	1.136 (4)
C(1)—C(2)	1.400 (3)	C(1)—C(9a)	1.394 (3)
C(1)—C(11)	1.456 (4)	C(2)—C(3)	1.398 (5)
C(3)—C(4)	1.364 (4)	C(4)—C(4a)	1.396 (3)
C(4a)—C(9a)	1.405 (4)	C(5)—C(6)	1.384 (4)
C(5)—C(10a)	1.376 (3)	C(6)—C(7)	1.397 (5)
C(7)—C(8)	1.370 (4)	C(8)—C(8a)	1.390 (3)
C(8a)—C(9)	1.475 (3)	C(8a)—C(10a)	1.405 (4)
C(9)—C(9a)	1.499 (3)		
O(10b)—S(10)—O(10a)	118.5 (1)	O(10b)—S(10)—C(4a)	108.0 (1)
O(10a)—S(10)—C(4a)	107.3 (1)	O(10b)—S(10)—C(10a)	108.7 (1)
O(10a)—S(10)—C(10a)	108.5 (1)	C(4a)—S(10)—C(10a)	105.1 (1)
C(2)—C(1)—C(9a)	121.4 (3)	C(2)—C(1)—C(11)	116.2 (2)
C(9a)—C(1)—C(11)	122.4 (2)	C(1)—C(2)—C(3)	119.3 (2)
C(2)—C(3)—C(4)	120.6 (2)	C(3)—C(4)—C(4a)	119.8 (3)
S(10)—C(4a)—C(4)	115.2 (2)	S(10)—C(4a)—C(9a)	123.0 (2)
C(4)—C(4a)—C(9a)	121.6 (2)	C(6)—C(5)—C(10a)	119.5 (3)
C(5)—C(6)—C(7)	119.9 (2)	C(6)—C(7)—C(8)	119.8 (2)
C(7)—C(8)—C(8a)	121.6 (3)	C(8)—C(8a)—C(9)	118.6 (2)
C(8)—C(8a)—C(10a)	117.5 (2)	C(9)—C(8a)—C(10a)	123.9 (2)
O(9)—C(9)—C(8a)	120.2 (2)	O(9)—C(9)—C(9a)	118.8 (2)
C(8a)—C(9)—C(9a)	121.1 (2)	C(1)—C(9a)—C(4a)	117.4 (2)
C(1)—C(9a)—C(9)	120.8 (2)	C(4a)—C(9a)—C(9)	121.8 (2)
S(10)—C(10a)—C(5)	116.6 (2)	S(10)—C(10a)—C(8a)	121.6 (2)
C(5)—C(10a)—C(8a)	121.6 (2)	N(11)—C(11)—C(1)	174.0 (2)

Related literature. For the synthesis of the title compound and related compounds refer to Sutherland *et al.* (1987). For structurally related compounds refer to Chu & Yang (1976), Chu & Napoleone (1982), Longo & Richardson (1982), Chu, Napoleone & Chu (1987).

SHS acknowledges the support of the Robert A. Welch Foundation (grant F-017), and RGS the financial support of the Natural Sciences and Engineering Research Council of Canada.

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