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Structure of 2-[1-(2-Carboxyethyl)ethylidene]hydrazinecarbothioamide

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Abstract. $\text{CH}_3\text{C}(\text{=NNHCSNH}_2)(\text{CH}_2)_2\text{CO}_2\text{H}$, $\text{C}_6\text{H}_{11}\text{N}_3\text{O}_2\text{S}$, $M_r = 189.24$, monoclinic, $P2_1/n$, $a = 7.652$ (2), $b = 10.398$ (1), $c = 11.135$ (2) Å, $\beta = 96.80$ (1)°, $V = 879.7$ (3) Å³, $Z = 4$, $D_x = 1.430$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 3.18$ cm⁻¹, $T = 298$ K, $F(000) = 400$, $R = 0.047$ for 1047 $I \geq 3\sigma(I)$ reflections. The thiosemicarbazone exists in the tautomeric thione form; the double-bonded S is H-bonded to two adjacent carboxyl O atoms [$\text{S}\cdots\text{O} = 3.085$ (5), 3.280 (5) Å].

Experimental. Equimolar amounts of levulinic acid and thiosemicarbazide were briefly heated together in ethanol; the cooled mixture furnished slightly tan colored crystals of the title compound. A crystal measuring approximately $0.22 \times 0.22 \times 0.22$ mm was used for the diffraction analysis. The diffractometer was an Enraf–Nonius instrument equipped with Mo $K\alpha$ radiation. Accurate cell dimensions were obtained from the 25 most intense reflections in the $13 \leq \theta \leq 15^\circ$ thin shell. Intensity data were collected up to $2\theta_{\text{max}} = 50^\circ$ ($h, k, \pm l, 9, 12, 13$) by using the ω - 2θ scan technique; 1659 reflections collected; 1417 unique reflections, with 1047 data satisfying the $I \geq 3\sigma(I)$ criterion. The data were corrected for decay (min./max. correction = 1.0000/1.0002); three (008, 245, 404) monitor reflections: negligible variation for the 16 h of collection. The data set was corrected for absorption by using the ψ -scan data (min./max. correction = 0.9751, 0.9995). The structure was solved using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The non-H atoms were refined anisotropically. A difference Fourier synthesis revealed all the H atoms. H atoms

were refined with $B = 5$ Å². The full-matrix least-squares refinement based on F converged with $\Delta/\sigma < 0.01$ at $R = 0.047$, $wR = 0.074$ [$w = [\sigma(F)^2 + (0.02F)^2 + 1]^{-1}$]; $S = 0.830$; 142 variables were refined. The max. $\Delta\rho$ was 0.28 (3) e Å⁻³. Scattering factors were taken from Tables 2.2B and 2.3.1 of *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were performed using the *MolEN* structure determination package (Fair, 1990) on a DEC MicroVAX minicomputer. Table 1* lists the atomic coordinates and Table 2 bond distances and angles; the structure is shown in Fig. 1.

Related literature. The N–N bond length of 1.382 (7) Å in the thiosemicarbazone, which is shorter than the statistical average of 1.401 Å found for planar $R_2\text{N}—\text{NR}_2$ compounds but longer than the statistical average of 1.240 Å found for $R—\text{N}=\text{N}—R$ compounds (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987), suggests double-bond character in the flat $\text{C}=\text{N}—\text{N}—\text{C}$ [$\text{C}—\text{N}—\text{N}—\text{C} = -178.9$ (5)°] fragment. The compound also possesses a carboxylate unit, but the acid does not form a dimer through hydrogen bonding. Instead, the S atom hydrogen bonds to two adjacent [(i) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$] carboxyl O atoms [$\text{S}\cdots\text{O}^{\text{i}} = 3.085$ (5), $\text{S}\cdots\text{O}^{\text{ii}} = 3.280$ (5) Å] to give rise to a hydrogen-bonded network.

* Lists of structure factors, anisotropic thermal parameters and calculated hydrogen positional parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55256 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0572]

Table 1. Positional parameters and their estimated standard deviations

$$B_{eq} = (4/3)[a^2 B_{1,1} + b^2 B_{2,2} + c^2 B_{3,3} + ab(\cos\gamma)B_{1,2} + ac(\cos\beta)B_{1,3} + bc(\cos\alpha)B_{2,3}]$$

	x	y	z	B _{eq} (Å ²)
S	0.1269 (2)	0.2019 (2)	0.1234 (1)	3.24 (3)
O1	0.6948 (6)	0.4927 (5)	0.4250 (4)	4.6 (1)
O2	0.9240 (6)	0.3854 (4)	0.3748 (4)	4.1 (1)
N1	0.2863 (6)	0.3845 (5)	0.2601 (4)	3.5 (1)
N2	0.4310 (6)	0.3181 (5)	0.1027 (4)	2.81 (9)
N3	0.5636 (6)	0.4026 (5)	0.1448 (4)	2.74 (9)
C1	0.8186 (8)	0.4710 (6)	0.3552 (5)	3.2 (1)
C2	0.8153 (8)	0.5643 (6)	0.2530 (6)	3.4 (1)
C3	0.8390 (8)	0.5024 (6)	0.1325 (5)	3.0 (1)
C4	0.6925 (7)	0.4129 (5)	0.0833 (5)	2.5 (1)
C5	0.7147 (8)	0.3431 (6)	-0.0301 (5)	3.6 (1)
C6	0.2920 (7)	0.3078 (5)	0.1660 (5)	2.7 (1)

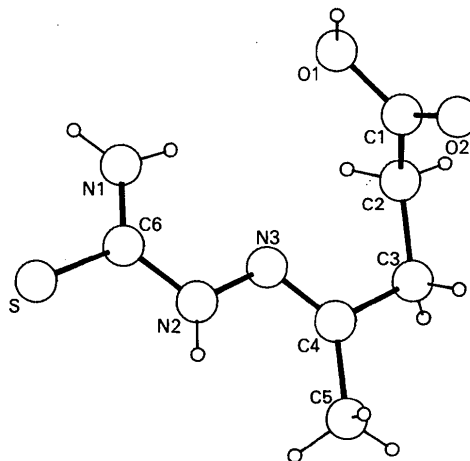


Fig. 1. Atom-numbering scheme.

Table 2. Bond distances (Å) and angles (°)

S—C6	1.700 (6)	N3—C4	1.270 (7)
O1—C1	1.314 (8)	C1—C2	1.492 (9)
O2—C1	1.203 (7)	C2—C3	1.518 (9)
N1—C6	1.322 (8)	C3—C4	1.509 (8)
N2—N3	1.382 (7)	C4—C5	1.484 (9)
N2—C6	1.348 (7)		
S...O1 ⁱ	3.085 (5)	S...O1 ⁱⁱ	3.280 (5)
N3—N2—C6	117.8 (5)	N3—C4—C3	116.6 (5)
N2—N3—C4	117.2 (5)	N3—C4—C5	126.3 (6)
O1—C1—O2	122.3 (6)	C3—C4—C5	117.1 (5)
O1—C1—C2	113.0 (6)	S—C6—N1	121.2 (4)
O2—C1—C2	124.7 (6)	S—C6—N2	120.8 (5)
C1—C2—C3	113.9 (5)	N1—C6—N2	117.9 (5)
C2—C3—C4	114.8 (5)		

Symmetry code: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

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1,3-Dinitropyrrolo[1,2-*b*]isoquinoline-5,10-dione

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Abstract. C₁₂H₅N₃O₆, *M_r* = 287.2, orthorhombic, *Pbca*, *a* = 11.336 (2), *b* = 10.134 (3), *c* = 20.275 (3) Å, *V* = 2329.2 Å³, *Z* = 8, *D_x* = 1.64 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 1.3 cm⁻¹, *F*(000) = 1168, *T* = 195 K, final *R* = 0.063 for 638 observed reflections. The fused ring system is planar and the molecules are disordered between two orientations such that the N

atom is distributed between two sites. The two nitro groups are in the 1,3 sites of the pyrrolo ring and make angles of 44 and 48° with the mean plane of the fused ring nucleus.

Experimental. The compound (I) was obtained by nitration of pyrrolo[1,2-*b*]isoquinoline-5,10-dione