The structure was solved by direct methods and refined on F using the *SHELXTL-Plus* (MicroVAX II) program package (Sheldrick, 1987). H atoms were placed in idealized positions, and constrained to have C—H = 0.96 Å and isotropic displacement parameters U = 0.08 Å^2. All non-H atoms were treated as anisotropic.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1151). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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S-Benzylisothiouronium Hydrogen 2-Oxopentanedioate, $C_8H_{11}N_2S^+.C_5H_5O_5^-$

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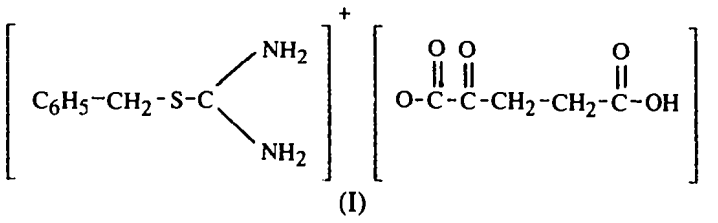
Abstract

The 5-carboxylic acid end of the 2-oxopentanedioate group of *S*-benzylisothiouronium hydrogen 2-oxopentanedioate is linked by a hydrogen bond [O···O = 2.653 (9) Å] across a centre of inversion to give rise to a dianionic species. At the free carboxyl end, the two carboxyl O atoms form hydrogen bonds to the amino groups belonging to four adjacent cations [O···N = 2.757 (9), 2.889 (9), 2.785 (8), 2.844 (7) Å].

Comment

The compound, (I), was synthesized for use in a condensation reaction with triphenyltin hydroxide to yield the $[(C_6H_5)_3SnO_2CCH_2CH_2COCO_2]^-$ anion, following the structural characterization of the $[(C_6H_5)_3SnO_2CCH_2CH_2CO_2]^-$ (Ng, Kumar Das, Xiao, van der Helm, Holecek & Lycka, 1991) and $[(C_6H_5)_3SnO_2CCO_2]^-$ (Ng & Kumar Das, 1993) stannate ions.



(I)

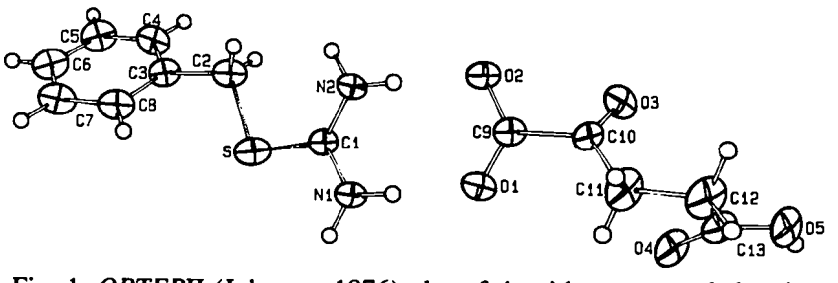


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title compound showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The compound was synthesized from monosodium 2-oxoglutarate and *S*-benzylisothiouronium hydrochloride in water (Furniss, Hannaford, Smith & Tatchell, 1989), and transparent crystals were grown from an ethanol solution of the compound.

Crystal data

$C_8H_{11}N_2S^+.C_5H_5O_5^-$
M_r = 312.35
Monoclinic
$P2_1/n$
a = 16.380 (3) Å
b = 5.5227 (5) Å
c = 17.355 (3) Å
β = 108.78 (8)°
V = 1486.3 (4) Å^3
Z = 4
D_x = 1.396 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 6–10°
μ = 0.229 mm^{-1}
T = 300 K
Platelet
0.25 × 0.25 × 0.07 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.9173, T_{max} = 0.9999
3035 measured reflections
2723 independent reflections
1186 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.025
θ_{max} = 25°
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 6$
$l = -20 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
R = 0.060
wR = 0.069
S = 0.465
1186 reflections
190 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0004|F|^2 + 1]$
$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 0.28 (4) e Å^{-3}
$\Delta\rho_{min}$ = −0.19 (4) e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
S	0.9290 (1)	0.0650 (5)	0.2657 (1)	4.80 (5)
O1	0.7810 (3)	0.774 (1)	0.3682 (3)	4.6 (1)
O2	0.8924 (3)	0.734 (1)	0.4802 (3)	5.0 (1)
O3	0.7767 (3)	0.933 (1)	0.5532 (3)	4.6 (1)
O4	0.5765 (3)	0.978 (1)	0.4563 (3)	5.0 (1)
O5	0.5577 (3)	1.277 (1)	0.5353 (3)	4.5 (1)
N1	0.8316 (3)	0.416 (1)	0.2826 (3)	3.8 (1)
N2	0.9475 (3)	0.349 (1)	0.3957 (3)	3.5 (1)
C1	0.9021 (4)	0.294 (1)	0.3208 (4)	3.0 (2)
C2	1.0326 (4)	−0.038 (2)	0.3327 (4)	3.8 (2)
C3	1.0759 (4)	−0.199 (1)	0.2861 (4)	3.1 (2)
C4	1.1180 (5)	−0.404 (1)	0.3234 (4)	3.5 (2)
C5	1.1623 (4)	−0.547 (2)	0.2858 (4)	4.1 (2)
C6	1.1645 (5)	−0.479 (2)	0.2090 (4)	4.2 (2)
C7	1.1229 (5)	−0.278 (2)	0.1718 (5)	4.1 (2)
C8	1.0775 (4)	−0.135 (1)	0.2093 (4)	3.6 (2)
C9	0.8207 (4)	0.817 (1)	0.4411 (4)	3.2 (2)
C10	0.7742 (4)	0.980 (1)	0.4850 (4)	3.1 (2)
C11	0.7236 (5)	1.187 (2)	0.4381 (5)	4.5 (2)
C12	0.6712 (5)	1.319 (2)	0.4812 (5)	4.9 (2)
C13	0.5970 (5)	1.174 (2)	0.4903 (5)	4.3 (2)

Table 2. *Geometric parameters* (Å, °)

S—C1	1.727 (8)	C3—C4	1.38 (1)
S—C2	1.812 (7)	C3—C8	1.39 (1)
O1—C9	1.243 (8)	C4—C5	1.37 (1)
O2—C9	1.239 (8)	C5—C6	1.39 (1)
O3—C10	1.198 (8)	C6—C7	1.36 (1)
O4—C13	1.23 (1)	C7—C8	1.38 (1)
O5—C13	1.29 (1)	C9—C10	1.53 (1)
N1—C1	1.318 (9)	C10—C11	1.49 (1)
N2—C1	1.308 (8)	C11—C12	1.50 (1)
C2—C3	1.52 (1)	C12—C13	1.51 (1)
C1—S—C2	103.1 (4)	C3—C8—C7	119.3 (8)
S—C1—N1	115.8 (5)	O1—C9—O2	125.6 (8)
S—C1—N2	123.7 (6)	O1—C9—C10	115.8 (6)
N1—C1—N2	120.5 (7)	O2—C9—C10	118.6 (6)
S—C2—C3	110.0 (5)	O3—C10—C9	119.9 (7)
C2—C3—C4	118.7 (7)	O3—C10—C11	122.8 (8)
C2—C3—C8	121.3 (7)	C9—C10—C11	117.2 (7)
C4—C3—C8	119.9 (8)	C10—C11—C12	114.1 (7)
C3—C4—C5	120.8 (8)	C11—C12—C13	113.8 (8)
C4—C5—C6	118.8 (8)	O4—C13—O5	124.5 (9)
C5—C6—C7	120.6 (9)	O4—C13—C12	121.6 (9)
C6—C7—C8	120.5 (8)	O5—C13—C12	113.9 (9)

Table 3. *Contact distances* (Å)

O(1)···N(1)	2.757 (9)	O(2)···N(2^{ii})	2.844 (7)
O(1)···N(1^{i})	2.785 (8)	O(4)···O(5^{iii})	2.653 (9)
O(2)···N(2)	2.889 (9)		

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

Non-H atoms were refined anisotropically; H atoms were generated geometrically and allowed to ride on their parent C or N atoms (C—H = 0.95 Å, B = 5 Å^2).

Data collection: CAD-4 diffractometer software (Enraf–Nonius, 1988). Cell refinement: CAD-4 diffractometer software. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1018). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Phenylhydrazone Form of 2-(4-Chlorophenylazo)-1,3-indandione

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Abstract

In the title compound, 2-(4-chlorophenylhydrazono)-1,3-indandione ($C_{15}H_9ClN_2O_2$), the indandione and phenylazo groups are connected by a C=N bond of 1.307 (3) Å. The molecule is planar and the bond lengths correspond to those expected for a phenylhydrazone. A strong intramolecular N—H···O hydrogen bond was found.