2708 reflections
292 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00042F_o^2]$

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)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Au(1)	0.2782 (1)	0.2152 (1)	0.2508 (1)	0.063 (1)
Au(2)	0	0	0	0.051 (1)
Au(3)	1/2	1/2	1/2	0.076 (1)
S(1)	0.1317 (3)	0.1174 (2)	0.0590 (3)	0.072 (1)
S(2)	0.0309 (3)	0.0763 (2)	−0.1641 (2)	0.072 (1)
S(3)	0.5391 (4)	0.4228 (3)	0.3422 (3)	0.097 (2)
S(4)	0.3302 (4)	0.6326 (3)	0.4339 (3)	0.094 (1)
P(1)	0.0615 (3)	0.3148 (3)	0.3069 (3)	0.074 (1)
P(2)	0.4860 (3)	0.0952 (3)	0.2187 (3)	0.079 (1)
C(1)	0.1871 (12)	0.1890 (8)	−0.0537 (10)	0.067 (5)
C(2)	0.1448 (11)	0.1718 (8)	−0.1497 (9)	0.063 (4)
C(3)	0.2758 (14)	0.2660 (10)	−0.0499 (12)	0.085 (6)
C(4)	0.3212 (14)	0.3200 (10)	−0.1364 (14)	0.091 (7)
C(5)	0.2828 (15)	0.3038 (10)	−0.2287 (13)	0.098 (7)
C(6)	0.1912 (14)	0.2291 (10)	−0.2358 (10)	0.091 (6)
C(7)	−0.0667 (12)	0.2277 (11)	0.3410 (11)	0.091 (6)
C(8)	−0.0249 (16)	0.1369 (13)	0.4145 (15)	0.144 (10)
C(9)	0.0749 (17)	0.3924 (14)	0.4175 (14)	0.140 (9)
C(10)	0.1665 (22)	0.3442 (17)	0.4956 (14)	0.181 (13)
C(11)	−0.0259 (18)	0.4269 (13)	0.2207 (18)	0.158 (11)
C(12)	−0.0534 (22)	0.4001 (16)	0.1291 (14)	0.170 (12)
C(13)	0.5756 (18)	0.1037 (15)	0.0851 (14)	0.145 (9)
C(14)	0.6390 (17)	0.1988 (13)	0.0609 (15)	0.144 (10)
C(15)	0.4500 (19)	−0.0373 (12)	0.2093 (15)	0.156 (10)
C(16)	0.3605 (21)	−0.0848 (14)	0.2832 (20)	0.216 (15)
C(17)	0.6135 (20)	0.0949 (19)	0.3003 (17)	0.207 (14)
C(18)	0.5730 (19)	0.1065 (16)	0.4027 (13)	0.143 (10)
C(19)	0.4123 (13)	0.5062 (9)	0.2698 (11)	0.079 (6)
C(20)	0.3242 (13)	0.5931 (9)	0.3078 (10)	0.075 (5)
C(21)	0.2263 (14)	0.6533 (10)	0.2459 (13)	0.084 (6)
C(22)	0.2158 (16)	0.6286 (11)	0.1478 (13)	0.095 (7)
C(23)	0.3089 (16)	0.5424 (13)	0.1074 (12)	0.100 (7)
C(24)	0.4044 (15)	0.4795 (10)	0.1683 (13)	0.091 (6)

Table 2. *Selected geometric parameters* (Å, °)

Au(1)—P(1)	2.315 (3)	Au(2)—S(2)	2.311 (3)
Au(1)—P(2)	2.311 (3)	Au(3)—S(3)	2.319 (4)
Au(2)—S(1)	2.317 (3)	Au(3)—S(4)	2.324 (3)
P(1)—Au(1)—P(2)	169.9 (1)	S(3)—Au(3)—S(4)	89.6 (1)
S(1)—Au(2)—S(2)	90.0 (1)		

Computations were performed using the *SHELXTL* program package (Sheldrick, 1986).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms, and a packing diagram have been deposited with the IUCr (Reference: MU1100). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Monomeric (Dipropionato-*O*)(dithiourea-*S*)-zinc(II)

KIMMO SMOLANDER AND MARKKU AHLGRÈN

Department of Chemistry, University of Joensuu, PO Box 111, SF 80101 Joensuu, Finland

MILAN MELNÍK

Department of Inorganic Chemistry, Slovak Technical University, Jânska 1, 81237 Bratislava, Slovakia

JOZEF SKORSEPA AND KATARINA GYÖRYOVÀ

Department of Inorganic Chemistry, Faculty of Natural Science, University of Upjs, Kosice, Slovakia

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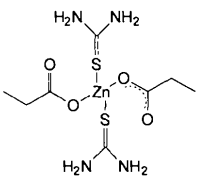
Abstract

Zinc(II) propionate reacts with thiourea in water to form the title monomeric colourless zinc(II) complex, $[Zn(C_3H_5O_2)_2(CH_4N_2S)_2]$. Two thiourea S atoms and two propionate O atoms form a distorted tetrahedron around the Zn^{II} atom with Zn—S bonds of 2.320 (1) and 2.341 (2) Å and Zn—O bonds of 1.959 (3) and 1.988 (2) Å. One carboxylate group forms two intermolecular hydrogen bonds to two different amino groups and these, together with other intermolecular hydrogen bonds, distort the tetrahedron.

Comment

Both anhydrous zinc(II) acetate (Capilla, 1979) and zinc(II) propionate (Goldschmied, Rae & Stephenson, 1977) form polymeric structures, the former a three-

dimensional and the latter a two-dimensional network. The zinc(II) ions lie in distorted tetrahedral environments with mean Zn—O distances of 1.934 (12) and 1.952 (18) Å, respectively. In $[Zn(acetate)_2(thiourea)_2]$ (Cavalca, Gasparri, Andreetti & Domiano, 1967), the metal is tetrahedrally coordinated by two S atoms from two thiourea molecules [Zn—S distances of 2.261 (4) and 2.326 (4) Å] and by two O atoms from two acetate groups [Zn—O distances of 1.954 (8) and 1.973 (8) Å]. Two further O atoms from the same acetate groups are involved in weaker interactions with the Zn^{II} ion at distances of 2.891 (8) and 2.996 (8) Å. Here we report the structure of $[Zn^{II}(propionate)_2(thiourea)_2]$, (I), which is being studied because of its possible antibacterial and anti-inflammatory activity.



(I)

The structure consists of discrete four-coordinate monomeric complexes (Fig. 1). The distorted coordination tetrahedron is formed by one O atom from each carboxylate ligand and by one S atom from each thiourea ligand. Intramolecular hydrogen bonds exist between one of the carboxylate groups and its two neighboring amino groups (Table 3), causing the two carboxylate C—O distances to be different. Together with the other intermolecular hydrogen bonds, they distort the tetrahedron.

The average Zn—S bond length of 2.333 (10) Å is 0.039 (11) Å longer than that in $[Zn(acetate)_2(thiourea)_2]$ (Cavalea *et al.*, 1967), whereas the average Zn—O bond distance of 1.97 (2) Å in the present compound is almost identical to that of 1.96 (2) Å in $[Zn(acetate)_2(thiourea)_2]$.

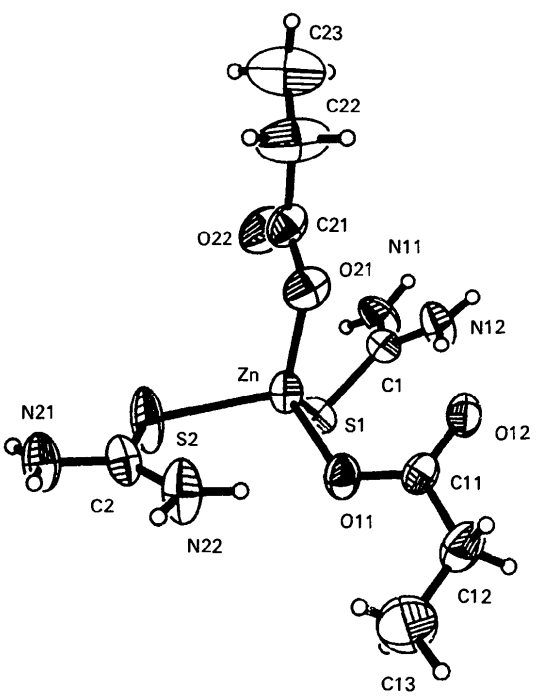


Fig. 1. Structure and numbering scheme of the title complex. The probability level of the displacement ellipsoids is 50%.

Experimental

The title compound was prepared by dissolving 0.01 mmol of zinc(II) propionate in 100 ml of water followed by addition of 0.02 mmol of thiourea. The precipitate that formed was filtered off and recrystallized from water. The colourless crystals were dried at room temperature.

Crystal data

$[Zn(C_3H_5O_2)_2(CH_4N_2S)_2]$	Mo $K\alpha$ radiation
M_r = 363.8	λ = 0.71073 Å
Trigonal	Cell parameters from 25 reflections
$R3c$ (hexagonal axes)	θ = 8–14°
a = 25.058 (4) Å	μ = 1.787 mm^{-1}
c = 13.585 (2) Å	T = 293 K
V = 7387 (2) $Å^3$	Prism
Z = 18	0.30 × 0.28 × 0.22 mm
D_x = 1.471 Mg m^{-3}	Colourless

Data collection

Siemens $R3$ diffractometer	R_{int} = 0.0252
ω scans	θ_{max} = 25°
Absorption correction: empirical	$h = 0 \rightarrow 32$
T_{min} = 0.506, T_{max} = 0.805	$k = -28 \rightarrow 28$
11 365 measured reflections	$l = -17 \rightarrow 17$
3784 independent reflections	2 standard reflections monitored every 98 reflections
3031 observed reflections [$I \geq 3\sigma(I)$]	intensity variation: 3%

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.005(F)^2]$
R = 0.0292	$(\Delta/\sigma)_{max}$ = 0.002
wR = 0.0343	$\Delta\rho_{max}$ = 0.47 e $Å^{-3}$
S = 0.95	$\Delta\rho_{min}$ = −0.56 e $Å^{-3}$
3031 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
195 parameters	
H-atom parameters not refined	

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Zn	0.3880 (1)	0.1730 (1)	0.2500	0.036 (1)
S(1)	0.4008 (1)	0.2224 (1)	0.1002 (1)	0.037 (1)
S(2)	0.4085 (1)	0.0931 (1)	0.2179 (1)	0.063 (1)

O(11)	0.4533 (1)	0.2301 (1)	0.3422 (2)	0.041 (1)
O(12)	0.4089 (1)	0.2873 (1)	0.3620 (2)	0.045 (1)
O(21)	0.3142 (1)	0.1398 (1)	0.3327 (2)	0.048 (1)
O(22)	0.2592 (1)	0.1012 (1)	0.1978 (2)	0.060 (1)
N(11)	0.3143 (2)	0.2378 (2)	0.0199 (3)	0.052 (2)
N(12)	0.3314 (2)	0.2635 (2)	0.1816 (3)	0.051 (2)
N(21)	0.4422 (2)	0.0324 (2)	0.3356 (3)	0.052 (2)
N(22)	0.4642 (2)	0.1261 (2)	0.3940 (3)	0.059 (2)
C(1)	0.3442 (3)	0.2427 (2)	0.1019 (3)	0.036 (1)
C(2)	0.4408 (2)	0.0844 (2)	0.3244 (3)	0.043 (2)
C(11)	0.4519 (2)	0.2762 (1)	0.3781 (2)	0.037 (1)
C(12)	0.5050 (2)	0.3184 (2)	0.4426 (3)	0.057 (2)
C(13)	0.5582 (3)	0.3083 (3)	0.4440 (6)	0.115 (4)
C(21)	0.2629 (2)	0.1075 (2)	0.2882 (3)	0.046 (2)
C(22)	0.2063 (2)	0.0752 (3)	0.3522 (4)	0.093 (3)
C(23)	0.1489 (3)	0.0478 (4)	0.3107 (6)	0.112 (4)

Table 2. *Selected geometric parameters* (Å, °)

Zn—S(1)	2.320 (1)	Zn—S(2)	2.341 (2)
Zn—O(11)	1.988 (2)	Zn—O(21)	1.959 (3)
S(1)—C(1)	1.731 (5)	S(2)—C(2)	1.723 (4)
O(11)—C(11)	1.269 (5)	O(12)—C(11)	1.260 (6)
O(21)—C(21)	1.278 (4)	O(22)—C(21)	1.236 (5)
N(11)—C(1)	1.312 (5)	N(12)—C(1)	1.309 (6)
N(21)—C(2)	1.331 (7)	N(22)—C(2)	1.310 (5)
C(11)—C(12)	1.499 (5)	C(12)—C(13)	1.477 (10)
C(21)—C(22)	1.508 (6)	C(22)—C(23)	1.369 (8)
S(1)—Zn—S(2)	105.2 (1)	O(11)—Zn—O(21)	101.9 (1)
S(1)—Zn—O(11)	108.9 (1)	S(2)—Zn—O(11)	106.2 (1)
S(1)—Zn—O(21)	125.4 (1)	S(2)—Zn—O(21)	107.9 (1)
Zn—S(1)—C(1)	103.3 (1)	Zn—S(2)—C(2)	105.6 (2)
Zn—O(11)—C(11)	120.7 (3)	Zn—O(21)—C(21)	115.7 (2)
S(1)—C(1)—N(11)	118.3 (3)	S(1)—C(1)—N(12)	121.8 (3)
N(11)—C(1)—N(12)	119.9 (5)	N(21)—C(2)—N(22)	118.4 (4)
S(2)—C(2)—N(21)	117.7 (3)	S(2)—C(2)—N(22)	123.9 (4)
O(11)—C(11)—O(12)	123.3 (3)	O(21)—C(21)—O(22)	122.6 (3)
O(11)—C(11)—C(12)	116.7 (4)	O(21)—C(21)—C(22)	116.4 (3)
O(12)—C(11)—C(12)	120.0 (4)	O(22)—C(21)—C(22)	120.9 (3)
C(11)—C(12)—C(13)	116.3 (5)	C(21)—C(22)—C(23)	120.2 (5)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N22—H8···O22^{i}	0.90 (7)	1.92 (7)	2.802 (4)	168 (2)
N22—H7···O11	0.95 (6)	1.92 (6)	2.842 (4)	165 (3)
N11—H2···O12^{ii}	0.88 (7)	1.99 (7)	2.857 (5)	171 (2)
N12—H4···O12	0.77 (6)	2.23 (6)	2.996 (4)	172 (2)
N11—H1···O21^{iii}	0.86 (6)	2.15 (6)	2.997 (4)	167 (2)
N21—H6···O12^{iv}	0.85 (6)	2.23 (6)	3.028 (4)	156 (2)
N21—H5···O22^{i}	0.77 (6)	2.32 (7)	3.029 (5)	155 (2)

Symmetry codes: (i) $\frac{2}{3}+y-x, \frac{1}{3}-x, \frac{1}{3}+z$; (ii) $\frac{1}{3}+y-x, \frac{2}{3}-x, z-\frac{1}{3}$; (iii) $x, x-y, z-\frac{1}{2}$; (iv) $\frac{2}{3}-y, \frac{1}{3}-x, z-\frac{1}{6}$.

The structure was solved by heavy-atom methods and Fourier techniques and refined by blocked-cascade full-matrix least squares with anisotropic displacement parameters for all non-H atoms. The H atoms attached to C atoms were included at calculated positions with fixed bond lengths (0.96 Å) and constrained angles; displacement parameters were set at 0.080 Å^2. The H atoms of the amino groups were located from a $\Delta\rho$ map. The calculations were performed with the *SHELXTL-Plus* program package (Sheldrick 1990). The figures were drawn with the same package.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and hydrogen-bonding parameters have been deposited with the IUCr (Reference: AB1149). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(thiourea-κS)bis(trichloroacetato-κO)-zinc(II) Monohydrate

IVAN POTOČŇÁK AND MICHAL DUNAJ-JURČO

Department of Inorganic Chemistry, Slovak Technical University, Radlinského 12, 812 37 Bratislava, Slovakia

VÁCLAV PETŘÍČEK

Physical Institute of Czech Academy of Sciences, Na Slovance 2, 180 40 Praha, Czech Republic

JURAJ ČERNÁK

Department of Inorganic Chemistry, P. J. Šafárik University, Moyzesova 11, 041 54 Košice, Slovakia

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Abstract

The crystal structure of the complex $[Zn(C_2Cl_3O_2)_2(CH_4N_2S)_2].H_2O$ is reported. The structure consists of discrete molecules bridged by hydrogen bonds to form an infinite three-dimensional network. The first coordination sphere of the Zn atom is best described as a deformed tetrahedron composed of two Zn—O bonds [2.012 (5) and 2.000 (5) Å] and two Zn—S bonds [2.316 (2) and 2.291 (2) Å]. The distances from the Zn atom to the non-bonded carboxylate O atoms are beyond the normal bonding distance.

Comment

As part of our study on the synthesis, crystallochemistry, properties and biological activity of zinc carboxylates, either with or without additional ligands, the title complex, (I), was isolated and its crystal structure determined. The complex was prepared by mixing a freshly prepared suspension of zinc hydroxide with an aqueous solution of trichloroacetic acid and an aqueous solution of thiourea in a 1:2:2 molar ratio. After several days colourless needles of the title complex were filtered off,