except those on the methyl groups in (1), were located from difference electron density (Δ) maps. In (2), ten H atoms, including the water H atoms, were found from the Δ map. The remaining H atoms, and all H atoms in (3), were given geometrically assumed positions (C—H = 1.00 Å) which were recalculated after each refinement cycle. Refinement of the water O atom in (3) with full site occupancy gave rise to unacceptably high displacement parameters (U_{iso} = 0.205 Å^2) for that atom. Moreover, there is an unusually short O(*W*)···O(*W*) contact distance of 2.40 (1) Å, indicating possible disorder for the water O atom. Subsequent refinement of the site occupation factor and the displacement parameter of O(*W*) in consecutive cycles yielded an approximately 50% occupancy for each O(*W*) position in the unit cell and a decrease of the vibrational parameter to U_{iso} = 0.088 Å^2. The partially occupied water H-atom positions, however, could not be located. In the last stage of the refinement the positions of the non-H atoms were refined together with their anisotropic displacement parameters, and isotropic vibrational parameters were refined for the O(*W*) position. The methyl groups were treated as rigid with free rotation around the O—C_{methyl} bond (Sheldrick, 1976). In the final calculation the mean shift/e.s.d. of the three rotation parameters of the methyl C(11) group of complex (3) was 0.51 (3). Six and 15 low θ reflections with F_o systematically lower than F_c, indicating extinction, were excluded from the last refinements of (1) and (3), respectively. The geometric features were calculated with the program *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry, as well as stereo drawings of the structures, have been deposited with the IUCr (Reference: AB1160). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(benzoato-*O*)bis(thiourea-*S*)zinc(II)

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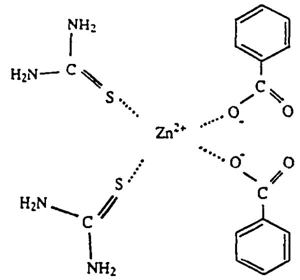
Abstract

The title complex crystallizes from an aqueous solution containing sodium benzoate, zinc sulfate and thiourea in the ratio 2:1:2. The structure is molecular and contains two crystallographically different $[Zn(C_6H_5COO)_2\{CS(NH_2)_2\}_2]$ molecules. The coordination geometry of each Zn atom, by one O atom from each benzoate anion and by one S atom from each thiourea ligand, is that of a deformed tetrahedron [Zn—O 1.964 (2) and 1.963 (2) Å; Zn—S 2.368 (1) and 2.366 (1) Å; angles 103.25 (9)–126.6 (2)°]. The Zn···O distances to the non-coordinated O atoms are long: 2.957 (3) (×2) and 2.955 (3) Å (×2). Most of the H atoms of the amidic groups are involved in hydrogen bonds of the N—H···O type.

Comment

As a part of our study of zinc carboxylates with or without additional ligands (Potočňák, Dunaj-Jurčo & Černák, 1993; Potočňák, Dunaj-Jurčo, Petříček & Černák, 1994), we prepared the title complex, (I). The

IR spectrum of this complex [FT-IR in KBr (cm^{-1}): 3473*m*, 3451*m*, 3326*s*, 3178*m*, 3147*m*, 3077*w*, 3027*w*, 1666*m*, 1623*vs*, 1599*s*, 1568*vs*, 1490*w*, 1497*w*, 1447*m*, 1418*m*, 1373*vs*, 722*m*, 686*m*, 636*m*, 614*m*, 498*m*] contains two peaks at 3473 and 3451 cm^{-1}. These peaks would usually indicate the presence of water molecules, but they remained after recrystallization from ethanol. The result of analysis (calculated C 41.79, H 3.95, N 12.18, Zn 14.19%; found C 41.68, H 3.68, N 11.91, Zn 13.95%) corresponds to the anhydrous complex. In order to find an explanation and to compare the structure with that of similar complexes, the structure determination of the title complex was undertaken.



(I)

The structure is molecular (Fig. 1). There are two crystallochemically different molecules in the unit cell. These molecules are chiral and both enantiomorphs are present. The Zn atoms are coordinated tetrahedrally in the ZnO_2S_2 form by two benzoate anions and two thiourea molecules; the ZnO_2S_2 tetrahedra are rather deformed. The bond lengths in the coordination polyhedra of the Zn atoms are normal (Table 2). Two additional Zn···O interactions to the non-coordinated O atoms of the carboxylate groups are long [2.957 (3) (×2) and 2.955 (3) Å (×2)] and the highest values for the 'tetrahedral' angles *X*—Zn—*Y* are 124.2 (2) and 126.6 (2)°. This is in line with the corresponding values found in similar complexes containing the ZnO_2S_2 unit: [$Zn(CH_3COO)_2(tu)_2$] (tu = thiourea) [2.891 (9) and 2.996 (5) Å, 119.3 (3)°; Cavalca, Gasparri, Andreetti & Domiano, 1967] and [$Zn(Cl_3CCOO)_2(tu)_2$].H_2O [2.916 (5) and 3.282 (7) Å, 123.23 (9)°; Potočňák, Dunaj-Jurčo, Petříček & Černák, 1994]. On the other hand, in complexes containing a ZnO_2O_2 unit, the additional interactions are relatively short and the tetrahedral deformations are greater: [Zn(2-HO-$C_6H_4COO)_2(H_2O)_2$] [2.523 (4) Å (×2), 131.6 (2)°; Rissanen, Valkonen, Kakkonen & Leskelä, 1987], [Zn(4-$ClC_6H_4COO)_2(H_2O)_2$] [2.456 (5) Å (×2), 136.9 (2)°; Potočňák, Dunaj-Jurčo & Černák, 1993] and [Zn(4-$O_2NC_6H_4COO)_2(H_2O)_2$] [2.511 (6) and 2.522 (6) Å, 136.7 (3)°; Guseinov, Musaev, Amiraslanov, Usubaliev & Mamedov, 1983]. In the complex [Zn-$(C_6H_5COO)_2(H_2O)_2]_2.C_6H_5COOH$ this distance is even shorter [2.36 Å (×2)], so we consider this coordination

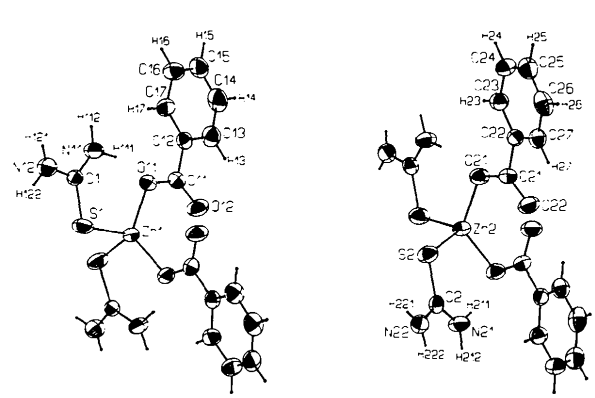


Fig. 1. *ORTEP* (Johnson, 1965) view of the two molecules of [$Zn(C_7H_5O_2)_2(CH_4N_2S)_2$] showing the atom-numbering scheme. Displacement ellipsoids are plotted at the 40% probability level.

geometry to be deformed octahedral. If, for the sake of comparison, one considers this coordination sphere as deformed tetrahedral, the largest value of the *X*—Zn—*Y* angle is 143.1 (2)° (Usubaliev, Guliev, Musaev, Ganbarov, Ashurova & Movsumov, 1992). So this complex represents an intermediate between a monodentately coordinated and a chelate-bonded Zn atom.

The complexes with a ZnO_2N_2 unit exhibit one short and one long additional interaction, *e.g.* [$Zn(CH_3COO)_2$-$(im)_2$] (im = imidazole) [2.645 (2) and 3.034 (2) Å, 118.5 (1)°; Horrocks, Ishley & Whittle, 1982] and [$Zn(CH_3CH_2COO)_2(im)_2$] [2.692 (2) and 3.151 (2) Å, 116.3 (1)°; Horrocks, Ishley & Whittle, 1982], [Zn(4-$H_2NC_6H_4COO)_2$].1.5H_2O [2.494 (8) and 3.395 (8) Å, 122.0 (3)°; Amiraslanov, Nadzhafov, Usubaliev, Musaev, Movsumov & Mamedov, 1980] and [Zn(4-HO-$C_6H_4COO)_2(py)_2$].2py (py = pyridine) [2.50 (1) and 3.18 (1) Å, 126.0 (6)°; Nadzhafov, Usubaliev, Amiraslanov, Movsumov & Mamedov, 1981], the tetrahedral deformation being approximatively the same as for the complexes containing S donor atoms. These experimental data indicate a correlation between the type of donor atom and the distance to the second O atom of a carboxylate group, as well as the degree of the tetrahedral deformation, but the reasons for the observed differences between different donor atoms are not clear at the moment. The question arises as to what the Zn···O limiting distance is for a bonding interaction.

As expected, the C—O bond lengths of the coordinated O atoms are somewhat longer than the others. The average C—C distances in the phenyl rings are somewhat shorter [1.379 (23) and 1.381 (4) Å] than the expected mean value of 1.395 Å. The planes formed by the phenyl rings and the carboxylate groups form angles of 11.8 (5) and 17.3 (4)°, hindering the delocalization of π electrons, which manifests itself in the observed C—C bond distances of 1.500 (5) and 1.505 (5) Å, respectively. The thiourea molecule containing S2 is planar within experimental error but that containing S1 is not. The bond distances and angles in both molecules are normal. A similar situation was found in the zinc trichloroacetate complex with thiourea (Potočňák, Dunaj-Jurčo, Petříček & Černák, 1994).

There are eight independent amidic hydrogen atoms in the structure. Five of them form strong hydrogen bonds (Table 3), two of them (H121 and H222) form weak hydrogen bonds and H atom H122 is not involved in hydrogen bonding at all; its shortest contact is to S1, with a length of 2.66 (5) Å and an angle N12—H122···S1 of 77 (4)°. These data may serve as an explanation of the fact that in the IR sprectrum of the complex, absorption bands corresponding to the ν(N—H) vibrations were found in the wide range 3473–3147 cm^{-1}.

Experimental

The title compound was prepared by first adding 1.22 g of solid benzoic acid to a solution of 0.4 g of NaOH in 25 ml of water (pH in the range 6.0–6.5). Successive solutions of $ZnSO_4.7H_2O$ (1.435 g in 10 ml of water) and of thiourea (0.762 g in 10 ml of water) were then added. After some days the product separated out in the form of very thin colourless plates. Monocrystals suitable for X-ray analysis were prepared by recrystallization from ethanol. The phase identity of the studied monocrystal with the polycrystalline product was checked by comparison of the experimental powder diffraction pattern with the calculated one by using the *DIFK*91 (Smrčok & Weiss, 1993) program.

Crystal data

$[Zn(C_7H_5O_2)_2(CH_4N_2S)_2]$
$M_r = 459.83$
Orthorhombic
$Ccc2$
$a = 24.667$ (4) Å
$b = 10.573$ (3) Å
$c = 15.736$ (4) Å
$V = 4104.0$ (17) $Å^3$
$Z = 8$
$D_x = 1.488$ Mg m^{-3}
$D_m = 1.44$ (2) Mg m^{-3}
D_m measured by flotation
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 10.93$–$16.72°$
$\mu = 1.457$ mm^{-1}
$T = 293$ (2) K
Pseudohexagonal prism
0.55 × 0.42 × 0.22 mm
Colourless

Data collection

Enraf–Nonius CAD-4L four-circle diffractometer
ω scans
Absorption correction: none
6087 measured reflections
3090 independent reflections
2118 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.0255$
$\theta_{max} = 29.90°$
$h = -34 \rightarrow 34$
$k = 0 \rightarrow 14$
$l = -22 \rightarrow 0$
2 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0286$
$wR(F^2) = 0.0664$
$\Delta\rho_{max} = 0.318$ e $Å^{-3}$
$\Delta\rho_{min} = -0.482$ e $Å^{-3}$
Extinction correction: none
$S = 1.075$
3090 reflections
273 parameters
$w = 1/[\sigma^2(F_o^2) + (0.037P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.041$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)

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

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

	x	y	z	U_{eq}
Zn1	0	0	0.25113 (3)	0.04212 (12)
Zn2	1/4	−1/4	0.15561 (3)	0.04530 (13)
S1	0.04630 (4)	−0.14165 (12)	0.34240 (7)	0.0642 (3)
S2	0.20469 (4)	−0.09755 (10)	0.07153 (7)	0.0582 (3)
O11	0.05627 (10)	0.0986 (2)	0.1927 (2)	0.0478 (6)
O21	0.30720 (10)	−0.1515 (3)	0.2116 (2)	0.0514 (6)
O12	−0.00445 (11)	0.1701 (3)	0.1021 (2)	0.0681 (8)
O22	0.24763 (11)	−0.0808 (3)	0.3050 (2)	0.0647 (8)
N11	0.1366 (2)	−0.0314 (4)	0.2845 (3)	0.0535 (9)
N12	0.1472 (2)	−0.1823 (4)	0.3852 (3)	0.0579 (10)
N21	0.1147 (2)	−0.2254 (4)	0.1134 (3)	0.0568 (10)
N22	0.1041 (2)	−0.0546 (4)	0.0277 (3)	0.0518 (9)
C1	0.11456 (14)	−0.1162 (3)	0.3347 (2)	0.0407 (7)
C11	0.04148 (14)	0.1718 (3)	0.1327 (2)	0.0427 (7)
C13	0.0677 (2)	0.3598 (4)	0.0454 (3)	0.0621 (10)
C14	0.1056 (3)	0.4478 (5)	0.0168 (3)	0.073 (2)
C15	0.1589 (2)	0.4362 (5)	0.0390 (3)	0.0744 (14)
C16	0.1743 (2)	0.3415 (5)	0.0899 (3)	0.0689 (12)
C17	0.1375 (2)	0.2528 (4)	0.1219 (3)	0.0532 (10)
C12	0.0832 (2)	0.2626 (3)	0.0995 (2)	0.0427 (8)
C2	0.13647 (15)	−0.1309 (3)	0.0719 (2)	0.0440 (7)
C21	0.29278 (13)	−0.0767 (3)	0.2716 (2)	0.0436 (8)
C23	0.3880 (2)	0.0100 (5)	0.2803 (3)	0.0522 (10)
C24	0.4239 (2)	0.1025 (5)	0.3065 (3)	0.0645 (11)
C25	0.4051 (2)	0.2046 (4)	0.3520 (3)	0.0685 (12)
C26	0.3510 (3)	0.2163 (5)	0.3714 (4)	0.069 (2)
C27	0.3150 (2)	0.1229 (4)	0.3466 (3)	0.0539 (9)
C22	0.3336 (2)	0.0200 (4)	0.3003 (2)	0.0424 (8)

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

Zn1—O11	1.964 (2)	O12—C11	1.231 (4)
Zn1—S1	2.3686 (11)	O22—C21	1.232 (4)
Zn2—O21	1.963 (2)	N11—C1	1.312 (5)
Zn2—S2	2.3659 (11)	N12—C1	1.330 (5)
S1—C1	1.710 (4)	N21—C2	1.309 (5)
S2—C2	1.719 (4)	N22—C2	1.331 (5)
O11—C11	1.275 (4)	C11—C12	1.500 (5)
O21—C21	1.281 (4)	C21—C22	1.505 (5)
O11ⁱ—Zn1—O11	124.2 (2)	N11—C1—N12	117.9 (4)
O11ⁱ—Zn1—S1	106.79 (8)	N11—C1—S1	123.9 (3)
O11—Zn1—S1	106.18 (8)	N12—C1—S1	118.2 (3)
S1ⁱ—Zn1—S1	105.34 (7)	O12—C11—O11	123.0 (3)
O21ⁱⁱ—Zn2—O21	126.6 (2)	O12—C11—C12	120.3 (3)
O21ⁱⁱ—Zn2—S2	105.86 (8)	O11—C11—C12	116.7 (3)
O21—Zn2—S2	103.25 (9)	N21—C2—N22	118.5 (4)
S2ⁱⁱ—Zn2—S2	112.00 (6)	N21—C2—S2	124.0 (3)
C1—S1—Zn1	109.42 (12)	N22—C2—S2	117.5 (3)
C2—S2—Zn2	108.73 (13)	O22—C21—O21	122.9 (3)
C11—O11—Zn1	117.8 (2)	O22—C21—C22	120.0 (4)
C21—O21—Zn2	117.3 (2)	O21—C21—C22	117.1 (3)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N11—H111···O11	0.89 (5)	1.96 (5)	2.811 (5)	158 (5)
N11—H112···O22	0.90 (5)	1.95 (5)	2.808 (5)	158 (4)
N12—H121···O22	0.86 (5)	2.40 (5)	2.980 (5)	125 (4)
N21—H211···O21ⁱ	0.79 (4)	2.02 (4)	2.792 (5)	165 (4)

N21—H212···O12^{ii}	0.84 (4)	2.03 (4)	2.788 (5)	151 (4)
N22—H221···N12^{iii}	0.82 (4)	2.74 (4)	3.526 (6)	161 (4)
N22—H222···O12^{ii}	0.75 (4)	2.28 (4)	2.984 (5)	156 (4)

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

The intensity data were collected for a monoclinic unit cell with a = 13.419 (3), b = 15.733 (4), c = 10.573 (4) Å, β = 113.19 (2)°. During refinement the unit cell was transformed to higher orthorhombic symmetry; the transformation matrix was M = (201/001/0$\bar{1}$0). The position of the H atoms of thiourea were refined with a common isotropic temperature factor U_{iso}, whereas for the H atoms of the phenyl rings, a riding model was used while refining common U_{iso}'s.

Data collection: CAD-4L diffractometer software. Cell refinement: CAD-4L diffractometer software. Data reduction: *LOPOTRI* (Gravereau, 1982). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1071). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Sodium Bumetanide Trihydrate

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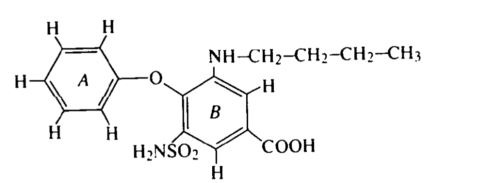
Abstract

The structure of sodium 3-(aminosulfonyl)-5-(butylamino)-4-phenoxybenzoate trihydrate (sodium bumetanide trihydrate), $Na^+.C_{17}H_{19}N_2O_5S^-.3H_2O$, consists of a layer of sodium ions and water molecules between two layers of bumetanide molecules. Six-coordinate Na^+ ions linked by water molecules are arranged in columns parallel to **b**.

Comment

The loop diuretic bumetanide is a powerful inhibitor of the Na^+, K^+, Cl^- cotransport system in a variety of epithelial cells (Petzinger, Muller, Follmann, Deutscher & Kinne, 1989). Bumetanide, (I), is a competitive inhibitor of sodium-dependent taurocholate uptake into hepatocytes (Blitzer, Tatoosh, Donovan & Boyer, 1982). The solid-state structures of the bile acids and taurocholate have been reported (Campanelli, Candeloro, Giglio & Scaramuzza, 1987). We undertook the X-ray analysis of sodium bumetanide trihydrate, reported here, as part of our studies of the structures of bioactive molecules.



(I)