Characterization of two new zinc(II) complexes with saccharinate and imidazole or benzimidazole as ligands

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The crystal structure of the complexes $[Zn(sac)_2(im)_2]$ (1) and $[Zn(sac)_2(bzim)_2]_2 \cdot 2Et-OH \cdot H_2O$ (2) (sac = saccharinate anion; im = imidazole; bzim = benzimidazole; EtOH = ethanol) was determined by single crystal X-ray diffractometry. Complex 1 crystallizes in the monoclinic $P2_1/n$ space group with a = 9.1585(5), b = 16.4409(6), c = 15.0249(5) Å, $\beta = 94.079(1)^\circ$, and Z = 4, whereas complex 2 belongs to the triclinic space group P1 with a = 10.8500(2), b = 12.4860(2), c = 13.5640(3) Å, $\alpha = 115,696(1)$, $\beta = 100.086(1)$, $\gamma = 102.169(1)^\circ$, and Z = 1. In both complexes, the Zn(II) cations are in a slightly distorted tetrahedral ZnN₄ environment, coordinated to two saccharinate anions and to two imidazole (1) and to two benzimidazole (2) molecules. The complexes were also characterized by means of infrared spectroscopy and their thermal behavior investigated by means of thermogravimetric and differential thermal analytical techniques.

KEY WORDS: Zinc(II); saccharinate; imidazole; benzimidazole; IR-spectra; thermal behavior.

Introduction

The coordination chemistry of saccharin (Hsac, *o*-sulphobenzimide) has proved to be very interesting and versatile. Besides, as confirmed by numerous recent examples, the saccharinate anion is able to produce a variety of mixed-ligand complexes, in which it shares the coordination sphere of metal centers with numerous other simple and complex ligands (cf., for example, Refs.^{1–9}).

As a continuation of our own research on this type of coordination compounds^{10–14} we have now prepared and thoroughly characterized two new tetrahedral zinc(II) complexes containing the saccharinate anion together with imidazole or benz-imidazole as the second ligand.

Experimental

Synthesis of the complexes

The starting material for the synthesis was tetraquabis(saccharinato)-zinc(II) dihydrate, obtained according to literature procedures.^{15,16}

For the preparation of $[Zn(sac)_2(im)_2]$ (1), 0.537 g (1 mmol) of this precursor was dissolved in 10 mL of distilled water and this solution was added dropwise to 10 mL of an aqueous

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solution containing 0.136 g (2 mmol) of imidazole. The reaction mixture was stirred for 1 h and, after filtration, evaporated slowly at room temperature. After a few days, colorless crystals were collected by filtration, washed with small portions of cold distilled water, and left to dry at room temperature. The other complex, $[Zn(sac)_2(bzim)_2]_2 \cdot 2EtOH \cdot H_2O$ (2), was prepared by same procedure, but working in ethanolic instead of in aqueous solutions, and employing 0.236 g (2 mmol) of benzimidazole. The obtained crystals were only stable in the mother liquor, becoming unstable in air because of the loss of the ethanol molecules.

Instrumentation

The IR spectra were recorded on a Perkin Elmer 580 B spectrophotometer using the KBr pellet technique.

Thermogravimetric (TG) and differential thermal analysis (DTA) were performed on a Shimadzu thermoanalytical system (models TG 50 and DTA 50, respectively), between room temperature and 800° C, working in Pt-crucibles, at a heating rate of 10° C·min⁻¹ and under a constant oxygen flow of 50 mL min⁻¹. Alumina was used as DTA standard.

X-ray structure determinations

Relevant crystallographic data and details of the refinement are presented in Table 1. All hydrogen atoms of $[Zn(sac)_2(im)_2]$ (1) were found in a difference Fourier map. However, they were positioned stereochemically and refined with the riding model. Most H-atoms of $[Zn(sac)_2(bzim)_2]_2 \cdot 2EtOH \cdot H_2O$ (2), including the ones of the water molecule, were also found in a difference Fourier map. The saccharinato and benzimidazole H-atoms were positioned and refined as described for (1). The water hydrogen atoms were refined with O—H distances restrained to a target value of 0.82(2) Å. The ethanol hydroxyl hydrogen was positioned at a tetrahedral location and the corresponding torsion angle refined to maximize the observed electron density at the calculated position. The methyl hydrogen atoms were treated in the refinement as rigid bodies and allowed to rotate along the corresponding C-C bond such as to maximize the sum of the observed electron density at the three calculated H-positions. The structures were resolved by direct and Fourier methods and the final molecular models obtained by anisotropic full-matrix leastsquares refinement of the non-hydrogen atoms. Programs used were DENZO and SCALEPACK¹⁷ for data reduction and corrections and SHELXS-9718 and SHELXL-9719 for structure solution and refinement, respectively. Intensity date were corrected for Lorentz, polarization, and experimental absorption effects.²⁰ Unit cell dimensions were obtained by least-square refinement of the angular settings for 17287 (1) and 20714 (2) reflections in the $1.84 < \theta < 25^{\circ}$ (1) and $1.75 < \theta < 26.00^{\circ}$ (2) ranges.

Results and discussion

Description of the structures

Figures 1 and 2 show the ORTEP²¹ plots of both structures including the atom labeling of the non-hydrogen atoms and their vibrational ellipsoids at 30% probability. Intramolecular bond distances and angles around the Zn(II) centers are presented in Tables 2 and 3.

In both complexes the saccharinate anion coordinates through its N-atom and the Zn(II) cation shows a slightly distorted ZnN₄ tetrahedral environment. In [Zn(sac)₂(im)₂] (1) the two Zn—N distances, involving the saccharinate ligands, are of 2.033(3) and 1.971(3) Å, whereas the bonds to the N-atom of the imidazole ligands are of 1.987(3) and 2.010(3) Å.

In $[Zn(sac)_2(bzim)_2]_2 \cdot 2EtOH \cdot H_2O$ (2) the two independent complexes can be related to each other through a noncrystallographic improper rotation. The homologous atom of all but one of the saccharinate ligands can be brought into a close superposition. The corresponding rms deviation from the best least-squares fit of the partial

Zn(II)-Saccharinate complexes

| | Complex 1 | Complex 2 | |
|--|---|--|--|
| CCDC deposit no. | 225621 | 225622 | |
| Chemical formula | $C_{20}H_{16}N_6O_6S_2Zn$ | C ₆₀ H ₅₄ N ₁₂ O ₁₅ S ₄ Zn ₂ | |
| Formula weight | 565.88 | 1442.13 | |
| Crystal size, mm | $0.20 \times 0.20 \times 0.20$ | $0.24 \times 0.20 \times 0.09$ | |
| Temperature, K | ature, K 120(2) | | |
| Diffractometer/scan | Kappa CCD/ φ and ω | Kappa CCD/ φ and ω | |
| Radiation, graph. Monochr. | graph. Monochr. Mo K α : $\lambda = 0.71073 \text{ Å}$ | | |
| Crystal system | Monoclinic | Triclinic | |
| Space group | $P2_{1}/c$ | P1 | |
| Unit cell dimensions | a = 9.1585(3) Å | a = 10.8500(2) Å | |
| | b = 16.4409(6) Å | b = 12.4860(2) Å | |
| | c = 15.0249(5) Å | c = 13.5640(3) Å | |
| | 0 1010213(0)11 | $\alpha = 115.696(1)^{\circ}$ | |
| | $\beta = 94.079(1)^{\circ}$ | $\beta = 100.086(1)^{\circ}$ | |
| | F | $\nu = 102.169(1)^{\circ}$ | |
| Volume, Å ³ | 2256.6(1) | 1542.76(5) | |
| Z | 4 | 1 | |
| Density (calcd.), mg/m^3 | 1.666 | 1.552 | |
| Absorption correction: | | | |
| Max. and mini. transm. | 0.796 and 0.554 | 0.916 and 0.797 | |
| Absorption coefficient, mm ⁻¹ | 1.325 | 0.991 | |
| θ range for data collection | 2.48–25.00° | 1.75–26.00° | |
| F(000) | 1152 | 742 | |
| Reflections collected | 17,287 | 20,714 | |
| Independent reflections | $3958 (R_{int} = 0.085)$ | $10,733 \ (R_{\rm int} = 0.047)$ | |
| Observed reflections $[I > 2\sigma(I)]$ | 3401 | 10,372 | |
| Data/restraints/parameters | 3958/0/316 | 10,733/5/850 | |
| Flack parameter | | 0.016(8) | |
| Goodness of fit on F^2 | 1.067 | 1.037 | |
| Final R indices $[I > 2\sigma(I)]$ | $R_1 = 0.045, wR_2 = 0.109$ | $R_1 = 0.042, wR_2 = 0.115$ | |
| R indices (all data) | $R_1 = 0.055, wR_2 = 0.118$ | $R_1 = 0.044, wR_2 = 0.117$ | |
| Largest peak and hole, $e \cdot A^{-3}$ | 0.80 and -0.64 | 0.69 and -0.80 | |

Table 1. Summary of Crystal Data, X-Ray Measurements, and Structure Refinements of $[Zn(sac)_2(im)_2](1)$ and $[Zn(sac)_2(bzim)_2]_2 \cdot 2EtOH \cdot H_2O(2)$

structures (calculated following Kabsch²²) is 0.11 Å. The remaining pair of homologous saccharinate groups is rotated from each other in about 180° around the corresponding Zn-N(sac) bond. In this complex, the Zn-N(sac) distances for the two independent complexes ranged from 2.014(4) to 2.034(4) Å and the Zn-N(bzim) bonds between 2.024(4) and 2.043(3) Å.

Average N—Zn—N bond angles are $109(5)^{\circ}$ in 1 and $109(4)^{\circ}$ in 2.

As expected, the molecular skeleton of the saccharinate ligands in both complexes are nearly planar (rms deviation of atoms from the mean plane less than 0.027 Å). The imidazole (in complex 1) and the benzimidazole (in complex 2) lig-

ands are also planar within experimental accuracy. The metal cation departs from the ligand planes in less than 0.434 Å in complex **1** and 0.293 Å in complex **2**.

It is worth commenting that the determined saccharinate N–S and N–C bond lengths (mean values of 1.642(6) and 1.369(8) Å, respectively, in complex **1** and 1.66(1) and 1.39(2) Å in complex **2**) are closer to the corresponding distances for neutral saccharin (1.663 and 1.375 Å, respectively²³) than for the anionic moiety (for example, 1.596 and 1.354 Å, respectively, in sodium saccharinate²⁴). On the other hand, the C=O bond distances are somewhat shorter in the complexes than in sodium saccharinate.



Fig. 1. Plot of the structure of $[Zn(sac)_2(im)_2]$ displaying the non-H labeling and their displacement ellipsoids at 30% probability. Full lines indicate zinc-ligand bonds.

The crystals are further stabilized by a net of intermolecular $N-H\cdots O$ bonds involving the imidazole (in 1) and benzimidazole (in 2) ligands of a complex and the saccharinate carbonyl and sulfonyl oxygen atoms of neighboring complexes. In complex 2, the EtOH and H₂O molecules act as acceptors in $N-H\cdots O$ bonds and the water molecule is a donor in $Ow-H\cdots O$ bonds with a carbonyl and a sulfonyl oxygen atoms.

Infrared spectra

Some of the most representative and characteristic vibrational modes of the two new



Fig. 2. Plot showing one of the complexes in $[Zn(sac)_2 (bzim)_2]_2 \cdot 2EtOH \cdot H_2O$ showing the non-H labeling and their displacement ellipsoids at 30% probability. Full lines indicate zinc-ligand bonds.

| Table 2. Bond Distances (Å) and Bond Angles (°) |
|---|
| Around $Zn(II)$ in $[Zn(sac)_2(im)_2](1)$ |

| Bond distance | |
|--------------------|------------|
| Zn-N(21) | 1.971(3) |
| Zn-N(31) | 1.987(3) |
| Zn-N(41) | 2.010(3) |
| Zn-N(11) | 2.033(3) |
| Bond angle | |
| N(21) - Zn - N(31) | 110.50(11) |
| N(21) - Zn - N(41) | 115.98(11) |
| N(31) - Zn - N(41) | 105.80(12) |
| N(21) - Zn - N(11) | 115.78(11) |
| N(31) - Zn - N(11) | 105.44(11) |
| N(41) - Zn - N(11) | 102.28(11) |
| | |

complexes are compared with those of the respective bands of sodium saccharinate¹⁰ and free imidazole^{25,26} and benzimidazole^{13,27} in Table 4.

Some aspects of the performed assignments are briefly commented, as follows.

- The high-frequency region is relatively complicated because of the superimposition of ν(CH) and ν(NH) vibrations and bands originating from nonfundamental modes (cf. also²⁸). In the case of complex 2, the ν(OH) band of the water molecule could be identified. Bands related to the
- Table 3. Bond Distances (Å) and Bond Angles (°) Around

 Zn(II) in [Zn(sac)₂(bzim)₂]₂·2EtOH·H₂O (2)

| Bond distance | |
|-------------------------|------------|
| Zn(1) - N(121) | 2.014(4) |
| Zn(1) - N(111) | 2.016(3) |
| Zn(1) - N(131) | 2.032(3) |
| Zn(1) - N(141) | 2.043(4) |
| Zn(2) - N(211) | 2.018(3) |
| Zn(2) - N(241) | 2.024(4) |
| Zn(2)-N(231) | 2.033(3) |
| Zn(2) - N(221) | 2.034(4) |
| Bond angle | |
| N(121) - Zn(1) - N(111) | 108.87(14) |
| N(121) - Zn(1) - N(131) | 106.93(14) |
| N(111) - Zn(1) - N(131) | 110.94(13) |
| N(121) - Zn(1) - N(141) | 111.70(14) |
| N(111) - Zn(1) - N(141) | 111.31(15) |
| N(131) - Zn(1) - N(141) | 107.00(14) |
| N(211) - Zn(2) - N(241) | 111.92(15) |
| N(211) - Zn(2) - N(231) | 110.47(13) |
| N(241) - Zn(2) - N(231) | 104.71(14) |
| N(211) - Zn(2) - N(221) | 108.79(14) |
| N(241) - Zn(2) - N(221) | 115.55(14) |
| N(231) - Zn(2) - N(221) | 105.06(14) |

| Na(sac)·H ₂ O | im | Complex 1 ^{<i>a</i>} | bzim | Complex 2 ^b | Assignments |
|--------------------------|----------|-------------------------------|---------|------------------------|--|
| | | | | 3517(s) | v(OH) |
| | 3125(w) | 3143(m) | 3115(w) | 3127(s) | V(OII) |
| 1642(vs) | 5120(11) | 1681(vs) | 5110() | 1696(sh) | v(C=O) |
| 1012(15) | | | | 1672(vs) | .(0 0) |
| | 1672(m) | 1671(vs) | | | $\nu(C-C)$ |
| | () | () | 1619(m) | 1630(m) | $\nu(C-C)$ |
| 1590(s) | 1579(w) | 1590(w) | 1588(m) | 1591(m) | $\nu(C-C) +$ |
| | 1545(m) | 1542(w) | | 1497(m) | $\delta_{ip}(NH)$ |
| | 1499(m) | 1499(w) | | | ······································ |
| | 1481(m) | ~ / | | | |
| 1460(m) | | | 1456(s) | 1462(m) | $\nu(C-C)$ |
| | | 1416(w) | 1353(s) | 1415(m) | v(C-C) |
| 1336(m) | | 1336(m) | | 1337(sh) | $v_{s}(C-N-S)$ |
| | 1326(s) | 1296(vs) | 1280(s) | 1291(vs) | $\delta_{ip}(CH)$ |
| | 1263(s) | | | | r · · |
| 1258(vs) | | 1250(s) | | 1248(vs) | $v_{\rm as}({\rm SO}_2)$ |
| 1150(vs) | | 1169(vs) | | 1150(vs) | $v_{\rm s}({\rm SO}_2)$ |
| | 1102(m) | 1119(sh) | 1115(m) | 1115(sh) | |
| | 1056(s) | 1103(sh) | | | $\delta_{ip}(CH)$ |
| | | 1060(s) | | | r · · |
| | 936(s) | 961(vs) | 933(m) | 964(vs) | v(ring) |
| | 661(s) | 664(m) | | 648(w) | |
| 610(m) | | 599(s) | | 601(s) | $\delta(SO_2)$ |
| | | 415(w) | 425(s) | 430(m) | τ-ring |
| | | 336(w) | | 341(w) | $\nu(Zn-N)$ |

 Table 4. Assignment of Some Characteristic IR Bands (in cm⁻¹) of Sodium Saccharinate, Imidazole, Benzimidazole, and the Two Investigated Complexes

Note. vs: very strong, s: strong, m: medium, w: weak, sh: shoulder.

a[Zn(sac)₂(im)₂].

^b[Zn(sac)₂(bzim)₂]₂·2EtOH·H₂O.

EtOH molecules could not be found. These molecules are probably lost during the preparation of the KBr pellets for measurement.

- In both complexes, the saccharinate C=O stretching mode is observed at higher frequencies than in the free saccharinate anion, in agreement with the fact that these bonds are slightly stronger in the complexes.
- The characteristic SO₂ and $\nu_s(C-N-S)$ vibrational modes remain practically unchanged after coordination.
- The behavior of the 936 cm⁻¹ ring mode of free imidazole (corresponding probably to the medium-intensity 933 cm⁻¹ band in benzimidazole) which, as known,^{29,30} is strongly dependent on the coordination

geometry of the complexes, is very interesting. In the present cases, this band is displaced to higher energies after coordination and its intensity is strongly enhanced. Most of the other skeletal vibrations are scarcely affected by the coordination.

- The ν (Zn-N) bands are located in the 350–280 cm⁻¹ region, as expected by comparison with other similar systems.^{13,25,31,32} These stretchings lie at comparable energies in agreement with the not very different Zn-N bond distances in both complexes (cf. Tables 2 and 3).

Thermal behavior

Both complexes show a comparable thermal behavior as derived from the analysis of its TG

and DTA data. $[Zn(sac)_2(im)_2]$ (1) presents a fourstep degradation pattern with steps between 20 and 320°C, 320 and 460°C, 460 and 550°C, and 550 and 700°C. The first three weight losses are accompanied by some weak DTA signals located at 287°C (endo), 400°C (exo), and 536°C (exo), respectively. The final pyrolysis process is related to two very strong exothermic signals at 607 and 619°C. Weight constancy is attained at around 700°C and the final solid pyrolysis residue was ZnO, as confirmed by IR spectroscopy. The total experimental weight loss of 85.70% is in excellent agreement with the calculated one (85.60%).

In the case of $[Zn(sac)_2(bzim)_2]_2 \cdot 2Et$ - $OH \cdot H_2O$ (2) it was observed that the weakly bonded ethanol molecules are lost during the crushing and weighing of the crystals. The TGA trace of this complex shows that in the temperature range between 20 and 180°C only the water molecule is given off (experimentally found mass loss 1.50%; calculated value = 1.33%) and this process is accompanied by a weak endothermic DTA signal at 58°C. The degradation of the anhydrous complex also occurs in four steps, as in the case of complex 1: between 180 and 380°C, 380 and 490°C, 490 and 562°C, and 562 and 700°C. Corresponding DTA signals are seen at 200 and 287°C (very weak, endo), 427°C (medium, exo), 556°C (strong, exo), and 630°C (very strong, exo).

Weight constancy is attained at around 650° C and the final solid pyrolysis residue was also in this case, ZnO. The total experimental weight loss of 88.00% practically coincides with the calculated value (87.95%).

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