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Two novel zinc(II) complexes of *N*′-[1-(2-pyridyl)ethylidene]morpholine-4-carbothiohydrazide

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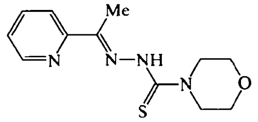
Abstract

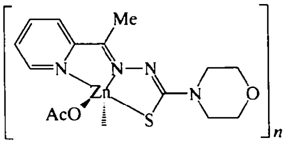
Two novel zinc(II) complexes of a morpholine derivative of 2-acetylpyridine N^4-substituted thiosemicarbazone, *i.e.* *N*′-[1-(2-pyridyl)ethylidene]morpholine-4-carbothiohydrazide (HL), have been studied. In the acetato–zinc(II) complex, *catena*-poly[[{*N*′-[1-(2-pyridyl-κ*N*)ethylidene]morpholine-4-carbothiohydrazide-κ^2N',S}zinc(II)]-μ-(acetato-$\kappa^2O{:}O'$)], [$Zn(C_{12}H_{15}N_4OS)(C_2H_3O_2)$], a deprotonated L ligand coordinates to the Zn^{II} atom through its three donor atoms (N, N′ and S), and the two O atoms of the acetato group bridge two Zn^{II} atoms in a twofold screw axis along **b** to form a linear polymer, *catena*-poly[Zn^{II}(L)-μ-(OAc-*O*,*O*′)]$_n$. The acetato group connects the Zn^{II} atoms in an *anti–syn* arrangement. In the nitrato complex, bis(nitrato-κ*O*){*N*′-[1-(2-pyridyl-κ*N*)ethylidene]morpholine-4-carbothiohydrazide-κ^2N',S}zinc(II), [$Zn(NO_3)_2(C_{12}H_{16}N_4OS)$], a neutral HL and two NO_3^- ligands coordinate to the Zn^{II} atom and the complex is monomeric. In both complexes, the coordination geometry around the five-coordinate Zn^{II} atom is described as an intermediate between regular square pyramidal and trigonal bipyramidal, due to the planar tridentate thiosemicarbazone ligand.

Comment

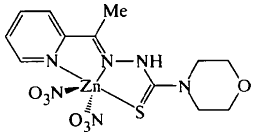
Currently, there is considerable interest in thiosemicarbazone complexes because 2-acetylpyridine thiosemicarbazones are found to be versatile compounds showing wide ranges of antibacterial, antifungal, antimalarial and antitumour activities (Klayman *et al.*, 1979; Dobek *et al.*, 1980; Scovill *et al.*, 1982; Liberta & West, 1992). This biological activity is considered to be due to their ability to form tridentate chelates with biologically important heavy metal ions. Thiosemicarbazones can coordinate to the metal either as a neutral bidentate ligand or as a deprotonated ligand through the N, N′ and S atoms. The structures of their Zn complexes are reported to be influenced significantly by the reaction conditions (nature of the solvent, pH, stoichiometry and temperature) and the complex can have five- and six-coordinate structures such as [$Zn^{II}X_2$(HL)] and [$Zn^{II}(L)_2$] (Ferrari *et al.*, 1992). N^4-Substituted 2-acetylpyridine thiosemicarbazones and their transition metal complexes were studied extensively by West *et al.* (1993, 1996), however, only a few structural studies have been reported for complexes of N^4-heterocyclic substituted thiosemicarbazones; for instance, a morpholine-substituted thiosemicarbazone, *N*′-[1-(2-pyridyl)ethylidene]morpholine-4-carbothiohydrazide (HL), has antimicrobial activities against selected bacteria, moulds and yeasts (Kasuga *et al.*, 1997). The reaction of HL with zinc(II) acetate or nitrate hydrates (Zn^{II}:HL = 1:1) in mixed organic solvents ($EtOH/CHCl_3$) at room temperature affords two novel complexes with different structures, namely the acetato complex, (1), and the nitrato complex, (2), which are described here.



HL



(1)



(2)

The molecular structure of the acetato complex, (1), is depicted in Fig. 1. The deprotonated L ligand coordinates to the Zn^{II} atom through three donor atoms (N, N′ and S) and the two O atoms of the acetato groups are bonded to the metal ion. These acetato groups bridge two Zn^{II} atoms in a twofold screw axis along **b** to form a linear polymer, *catena*-poly[Zn^{II}(L)-μ-(OAc-*O*,*O*′)]$_n$, as shown in Fig. 2. Three coordination arrangements are known for $RCOO^-$ when it bridges metal ions *via* the O atoms [*M*—O—C(*R*)-O—*M*′], namely, *syn–syn* (both *M*—O—C—*R* and *R*—C—O—*M*′ are *trans*), *anti–anti* (both *M*—O—C—*R* and *R*—C—O—*M*′ are *cis*) and *anti–syn* (*M*—O—C—*R* is *trans* and *R*—C—O—*M*′ is *cis*) (Inoue & Kubo, 1970). The Zn—O3—C13—C14 and C14—C13—O2—Zn^{ii} torsion angles are −176.7 (3) and 10.9 (4)°, respectively [symmetry code: (ii) $\frac{1}{2}-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$], hence the Zn—O—C—O—Zn^{ii} linkage in complex (1) is in the *anti–syn* arrangement.

In the nitrato complex, (2), one neutral HL and two NO_3^- ligands coordinate to the Zn^{II} atom to form a monomeric complex, as depicted in Fig. 3. Other crystals of complex (2) having the same molecular

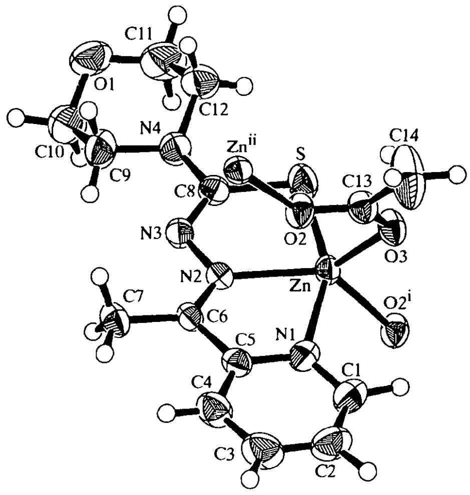


Fig. 1. An *ORTEP*II drawing (Johnson, 1976) of the acetato complex, (1), with 50% probability displacement ellipsoids. H atoms are shown as spheres of arbitrary radii [symmetry codes: (i) $\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$; (ii) $\frac{1}{2}-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$].

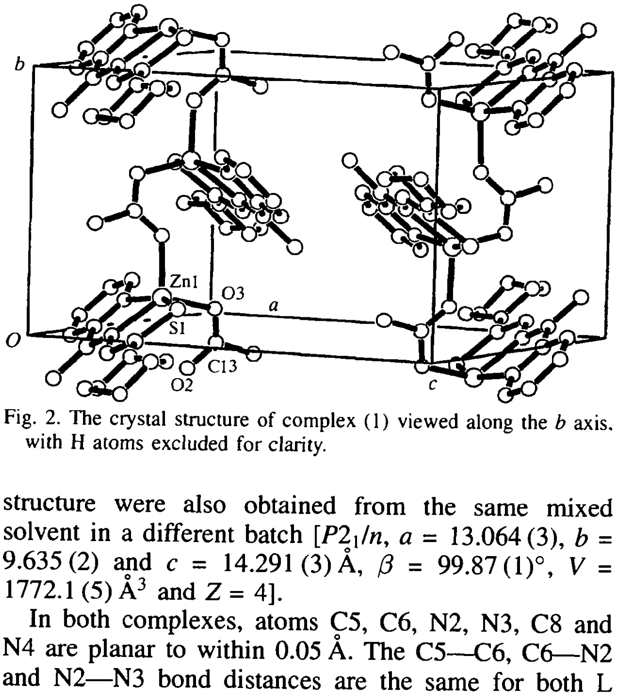


Fig. 2. The crystal structure of complex (1) viewed along the *b* axis, with H atoms excluded for clarity.

structure were also obtained from the same mixed solvent in a different batch [$P2_1/n$, a = 13.064 (3), b = 9.635 (2) and c = 14.291 (3) Å, β = 99.87 (1)°, V = 1772.1 (5) Å^3 and Z = 4].

In both complexes, atoms C5, C6, N2, N3, C8 and N4 are planar to within 0.05 Å. The C5—C6, C6—N2 and N2—N3 bond distances are the same for both L and HL. The differences due to the thiosemicarbazone moiety (N3—C8, C8—S and C8—N4) in L and HL [1.316 (4), 1.739 (3) and 1.373 (4) Å, respectively, in L; 1.365 (5), 1.702 (4) and 1.333 (5) Å, respectively, in HL] resulted in the formation of thioenolate in L.

In both complexes, the Zn^{II} atom is five-coordinate to N, N′, S, O and O′. The Zn—S distance in complex (1) [2.3723 (9) Å] is slightly longer than the sum of the covalent radii for Zn and S (2.35 Å). The Zn—O and Zn—N bond lengths in complex (1) are normal [Zn—N1 = 2.186 (2), Zn—N2 = 2.101 (2), Zn—O2^i = 2.016 (2) and Zn—O3 = 2.002 (2) Å; symmetry code: (i) $\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$]. The bond lengths about Zn in complex (2) are similar to those in complex (1) [Zn—S = 2.374 (1), Zn—N1 = 2.132 (3), Zn—N2 = 2.146 (3), Zn—O2 = 2.056 (3) and Zn—O5 = 2.028 (3) Å].

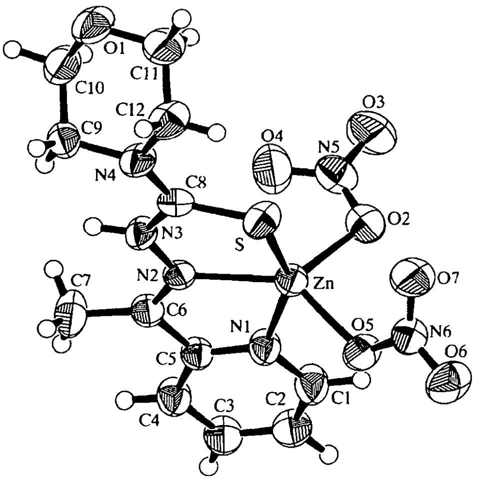


Fig. 3. A perspective view of the nitrato complex, (2), with 50% probability displacement ellipsoids. H atoms are shown as spheres of arbitrary radii.

The bond angles around the central metal (Tables 1 and 2) show values intermediate between a regular trigonal bipyramid (90, 120 and 180°) and a square pyramid (88.4, 100 and 160°). Therefore, the coordination geometry around the Zn^{II} atom can be described as intermediate between regular square pyramidal and trigonal bipyramidal, which is similar to that found by Ferrari *et al.* (1992) in [ZnCl$_2$(Hept)] (Hept is ethyl pyruvate thiosemicarbazone). This geometry about the Zn^{II} atom would be due to the planarity of the tridentate ligand.

The significant structural differences between complexes (1) and (2) result from the nature of the OAc$^-$ and NO$_3^-$ ligands, although the geometry about the Zn^{II} atom in both complexes is similar.

Experimental

HL was prepared according to the procedure published by Scovill (1991). The acetato complex, (1), was obtained by mixing zinc acetate dihydrate (1.10 g, 5 mmol) dissolved in EtOH (30 ml) with HL (1.32 g, 5 mmol) dissolved in CHCl$_3$ (15 ml) and stirring the mixture for 3 h at room temperature. The yellow precipitate that formed was filtered and recrystallized from a mixed solvent (EtOH:CHCl$_3$ = 1:2). After two weeks, single crystals suitable for X-ray diffraction studies were grown and mounted on glass capillaries (yield 15%). The remaining crystals were collected in a Büchner funnel, washed with cold EtOH and dried *in vacuo*. The nitrato complex, (2), was prepared by mixing zinc nitrate hexahydrate (1.49 g, 5 mmol) dissolved in EtOH (30 ml) with HL (1.32 g, 5 mmol) dissolved in CHCl$_3$ (30 ml) and stirring the mixture for 3 h at room temperature. The pale-yellow precipitate that gradually formed was filtered, and the pale-yellow solid obtained was recrystallized from a mixed solvent (EtOH:$CHCl_3$ = 1:1) at room temperature. About three days later, single-crystal needles were obtained and a few samples suitable for diffraction analysis were selected. The remaining crystals were collected in a Büchner funnel, washed with cold EtOH and dried *in vacuo* (combined yield 30%). The thiosemicarbazone ligand and its complexes were characterized by elemental analysis, IR, ^{1}H and ^{13}C NMR, and UV spectroscopy. For the acetato complex, (1): analysis calculated for $C_{14}H_{18}N_4O_3SZn$: C 43.37, H 4.68, N 14.45, S 8.27%; found: C 43.40, H 4.71, N 14.47, S 7.81%; IR (KBr, cm^{-1}): 1552 (*s*), 1413 (*s*), 1356 (*m*), 1259 (*m*), 1220 (*m*), 1208 (*m*), 1151 (*m*), 1084 (*m*), 888 (*m*); ^{1}H NMR (400 MHz, $CDCl_3$ at room temperature, p.p.m.): 1.87 (*s*, 3H), 2.52 (*s*, 3H) 3.76 (*m*, 4H), 4.07 (*m*, 4H), 7.46 (*d*, 1H), 7.59 (*d*, 1H), 7.89 (*t*, 1H), 8.81 (*d*, 1H); ^{13}C NMR (100 MHz, $CDCl_3$ at room temperature, p.p.m.): 13.44, 23.79, 46.48, 66.88, 120.87, 124.67, 139.52, 145.26, 149.23, 150.66, 178.62, 179.95; UV [$CHCl_3$; λ_{max}/nm (ε M^{-1} cm^{-1})]: 401 (19 900), 309 (11 200). For the nitrato complex, (2): analysis calculated for $C_{12}H_{16}N_6O_7SZn$: C 31.77, H 3.55, N 18.52, S 7.07%; found: C 31.81, H 3.24, N 18.60, S 6.78%; IR (KBr, cm^{-1}): 1384 (*s*), 1300 (*m*), 1550 (*m*), 1262 (*m*), 1086 (*m*), 778 (*m*); ^{1}H NMR (400 MHz, DMSO-d_6 at room temperature, p.p.m.): 2.55 (*s*, 3H), 3.64 (*t*, 4H), 3.95 (*t*, 4H), 7.61 (*br*, 1H), 7.98 (*d*, 1H), 8.13 (*t*, 1H), 8.45 (*br*, 1H); ^{13}C NMR (100 MHz, DMSO-d_6 at room temperature, p.p.m.): 13.71, 48.11, 66.02, 123.08, 126.00, 141.04, 148.00, 148.90, 178.95; UV [EtOH; λ_{max}/nm (ε M^{-1} cm^{-1})]: 402 (22 400), 307 (12 000).

Compound (1)

Crystal data

[$Zn(C_{12}H_{15}N_4OS)(C_2H_3O_2)$]
M_r = 387.76
Monoclinic
$P2_1/n$
a = 13.093 (2) Å
b = 8.836 (1) Å
c = 14.724 (1) Å
β = 92.913 (8)°
V = 1701.4 (3) Å^3
Z = 4
D_x = 1.51 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 14.6–15.0°
μ = 1.585 mm^{-1}
T = 296 (5) K
Plate
0.3 × 0.2 × 0.1 mm
Yellow

Data collection

Rigaku AFC-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (North *et al.*, 1968)
T_{min} = 0.730, T_{max} = 0.853
6647 measured reflections
3989 independent reflections
2762 reflections with $I > 2\sigma(I)$

R_{int} = 0.035
θ_{max} = 27.49°
h = −17 → 16
k = −11 → 11
l = 0 → 19
3 standard reflections every 150 reflections intensity decay: 2.29%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.109

$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

S = 1.029
3898 reflections
257 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0610P)^2 + 0.2681P]$ where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: Zachariasen (1967)
Extinction coefficient: 0.0065 (9)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °) for (1)*

Zn—O3	2.002 (2)	N2—N3	1.370 (3)
Zn—O2^i	2.016 (2)	N3—C8	1.316 (4)
Zn—N2	2.101 (2)	N4—C8	1.373 (4)
Zn—N1	2.186 (2)	N4—C12	1.458 (4)
Zn—S	2.3723 (9)	N4—C9	1.462 (4)
S—C8	1.739 (3)	C5—C6	1.481 (4)
O1—C10	1.420 (5)	C6—C7	1.489 (4)
O1—C11	1.421 (5)	C9—C10	1.507 (6)
O2—C13	1.241 (3)	C11—C12	1.499 (5)
O3—C13	1.255 (3)	C13—C14	1.521 (4)
N2—C6	1.289 (3)		
O3—Zn—O2^i	98.81 (8)	C6—N2—N3	117.9 (2)
O3—Zn—N2	129.94 (8)	C8—N3—N2	113.9 (2)
O2^i—Zn—N2	128.43 (8)	C12—N4—C9	112.1 (3)
O3—Zn—N1	92.78 (9)	N1—C5—C6	116.4 (2)
O2^i—Zn—N1	88.63 (9)	N2—C6—C5	114.6 (2)
N2—Zn—N1	75.06 (9)	N2—C6—C7	124.7 (2)
O3—Zn—S	105.25 (7)	N3—C8—N4	115.6 (3)
O2^i—Zn—S	103.85 (7)	N3—C8—S	126.8 (2)
N2—Zn—S	81.11 (6)	N4—C9—C10	110.0 (3)
N1—Zn—S	155.94 (7)	O1—C10—C9	112.2 (4)
C8—S—Zn	95.85 (10)	O1—C11—C12	111.3 (4)
C10—O1—C11	109.7 (3)	O2—C13—O3	122.3 (3)
C13—O2—Zn^{ii}	130.9 (2)	O3—C13—C14	117.4 (3)

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

Compound (2)

Crystal data

[$Zn(NO_3)_2(C_{12}H_{16}N_4OS)$]
M_r = 453.74
Triclinic
$P\bar{1}$
a = 9.5284 (15) Å
b = 10.829 (2) Å
c = 8.863 (3) Å
α = 91.94 (3)°
β = 94.00 (2)°
γ = 101.698 (13)°
V = 892.2 (3) Å^3
Z = 2
D_x = 1.69 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 10.7–13.8°
μ = 1.543 mm^{-1}
T = 293 (5) K
Needle
0.3 × 0.1 × 0.1 mm
Yellow

Data collection

Rigaku AFS-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (North *et al.*, 1968)
T_{min} = 0.638, T_{max} = 0.857
4437 measured reflections
4111 independent reflections
2283 reflections with $I > 2\sigma(I)$

R_{int} = 0.026
θ_{max} = 27.5°
h = 0 → 12
k = −14 → 13
l = −11 → 11
3 standard reflections every 150 reflections intensity decay: 1.37%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.120$
$S = 0.959$
4111 reflections
244 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.5031P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Zn—O5	2.028 (3)	O7—N6	1.219 (4)
Zn—O2	2.056 (3)	N1—C5	1.335 (5)
Zn—N1	2.132 (3)	N1—C1	1.341 (5)
Zn—N2	2.146 (3)	N2—C6	1.282 (5)
Zn—S	2.3744 (13)	N2—N3	1.373 (4)
S—C8	1.702 (4)	N3—C8	1.365 (5)
O1—C10	1.408 (5)	N4—C8	1.333 (5)
O1—C11	1.418 (6)	N4—C12	1.464 (5)
O2—N5	1.246 (5)	N4—C9	1.465 (5)
O3—N5	1.231 (5)	C5—C6	1.496 (5)
O4—N5	1.221 (5)	C6—C7	1.474 (6)
O5—N6	1.295 (4)	C9—C10	1.497 (7)
O6—N6	1.227 (4)	C11—C12	1.490 (7)
O5—Zn—O2	94.00 (13)	C8—N4—C9	126.7 (3)
O5—Zn—N1	86.35 (12)	C12—N4—C9	110.3 (3)
O2—Zn—N1	97.04 (13)	O4—N5—O3	120.9 (4)
O5—Zn—N2	135.52 (12)	O4—N5—O2	118.3 (4)
O2—Zn—N2	127.10 (13)	O3—N5—O2	120.7 (4)
N1—Zn—N2	73.76 (12)	O7—N6—O6	123.8 (4)
O5—Zn—S	105.59 (9)	O7—N6—O5	119.1 (4)
O2—Zn—S	107.43 (10)	O6—N6—O5	117.1 (4)
N1—Zn—S	151.63 (10)	N1—C5—C6	115.2 (3)
N2—Zn—S	80.01 (9)	C7—C6—C5	119.9 (4)
C10—O1—C11	110.5 (3)	N3—C8—S	121.4 (3)
C5—N1—C1	117.9 (3)	O1—C10—C9	112.2 (4)
C6—N2—N3	120.4 (3)	O1—C11—C12	111.9 (4)
C8—N3—N2	118.1 (3)	N4—C12—C11	108.1 (4)
C8—N4—C12	122.4 (4)		

H atoms except those in methyl groups of (1) were refined isotropically. H atoms in methyl groups of (1) and the H atoms of (2) were located geometrically by *TEXSAN* (Molecular Structure Corporation, 1995) and were not refined.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN*; program(s) used to solve structures: *TEXSAN*, *DIRDIF* (Beurskens *et al.*, 1992), *DIRDIF*94 (Beurskens *et al.*, 1994) and *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1085). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1267–1270

Tetrakis(methylamine-*N*)(oxalato-O^1,O^2)-cobalt(III) perchlorate and tetrakis(ethylamine-*N*)(oxalato-O^1,O^2)cobalt(III) perchlorate monohydrate

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Abstract

In the title compounds, $[Co(C_2O_4)(CH_5N)_4]ClO_4$, (1), and $[Co(C_2O_4)(C_2H_7N)_4]ClO_4\cdot H_2O$, (2), the Co atom has a distorted octahedral coordination composed of four amine ligands and one oxalato ligand. The coordination