

of Cu atoms have also been observed in (2,5-DM-DCNQ)₂Cu (Aumüller, Erk, Klebe, Hünig, von Schütz, & Werner, 1986), but with a larger distance between the copper centres (3.88 Å) and tetrahedral CuO₄ arrangement.

Selected interatomic distances and angles in the coordination polyhedra around copper are given in Table 2.

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Structures of Neodymium(III) Complexes with Amino Acids: (I) *catena*-Poly{[triaquatrichloroneodymium(III)]- μ -(β -alanine-*O, O'*)}; (II) Pentaqua-chloro- μ -(L-proline-*O, O'*)-neodymium(III) Hexaqua- μ -(L-proline-*O, O'*)-neodymium(III) Pentachloride

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Abstract. (I) [NdCl₃(H₂O)₃(C₃H₇NO₂)], $M_r = 393.7$, monoclinic, $P2_1/c$, $a = 6.018$ (2), $b = 16.562$ (6), $c = 12.518$ (4) Å, $\beta = 116.18$ (4)°, $V = 1119.7$ Å³, $Z = 4$, $D_m = 2.33$, $D_x = 2.34$ Mg m⁻³. Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 5.36$ mm⁻¹, $F(000) = 756$, $T = 298$ (2) K. Final $R = 0.035$ for 2363 reflections with $F > 6\sigma(F)$. (II) [NdCl(H₂O)₅(C₅H₉NO₂)Cl₂·[Nd(H₂O)₆(C₅H₉NO₂)Cl₃], $M_r = 929.6$, monoclinic, $C2$, $a = 25.076$ (5), $b = 8.132$ (2), $c = 18.361$ (4) Å, $\beta = 120.68$ (5)°, $V = 3220.1$ Å³, $Z = 4$, $D_m = 1.93$, $D_x = 1.92$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 3.76$ mm⁻¹, $F(000) = 1824$, $T = 298$ (3) K. Final $R = 0.040$ for 3540 reflections with $F > 7\sigma(F)$. These are the first known cases of lanthanide carboxylate complexes where the inner sphere contains Cl ions

attached to the metal. In both structures the Nd ions are eight coordinate. There are three Cl ions bonded to the Nd^{III} centre in (I) while in (II) one Cl ion is attached to Nd(1) and Nd(2) is coordinated exclusively by O atoms. The structure of (I) is that of a linear polymer while that of (II) consists of dinuclear species of two distinct types. The carboxylate group acts as a bridging ligand in both structures.

Introduction. Our investigation of lanthanide complexes with L- α -alanine (Dao, Głowiak, Huskowska & Legendziewicz, 1988, and references therein) revealed that when the stoichiometric ratio Ln^{III}:amino acid is 1:2 the structures consist of pseudosymmetric dinuclear units. However, when

Table 1. Atomic coordinates and equivalent isotropic thermal parameters (\AA^2) with e.s.d.'s in parentheses
$$B_{eq} = (1/3) \sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

(I)	x	y	z	B_{eq}
Nd	0.16114 (5)	0.14777 (2)	0.24738 (2)	0.98 (1)
Cl(1)	0.1615 (3)	0.0345 (1)	0.4176 (2)	2.7 (1)
Cl(2)	-0.1746 (3)	0.0279 (1)	0.1117 (2)	2.3 (1)
Cl(3)	-0.0867 (3)	0.2494 (1)	0.0473 (1)	2.2 (1)
O(1)	-0.1900 (8)	0.2002 (3)	0.2660 (4)	2.0 (3)
O(2)	-0.5495 (8)	0.2460 (3)	0.2487 (4)	1.7 (3)
O(3)	0.5797 (9)	0.0890 (3)	0.3622 (4)	2.1 (3)
O(4)	0.3101 (9)	0.2339 (4)	0.4319 (4)	3.2 (5)
O(5)	0.3147 (9)	0.0979 (3)	0.1009 (4)	2.3 (4)
N	-0.6755 (12)	0.4074 (4)	0.1929 (6)	2.8 (5)
C(1)	-0.3338 (11)	0.2570 (4)	0.2578 (5)	1.3 (4)
C(2)	-0.2586 (14)	0.3440 (4)	0.2555 (7)	2.1 (5)
C(3)	-0.4123 (14)	0.4047 (5)	0.2828 (6)	2.2 (5)
(II)				
Nd(1)	0.42755 (2)	0.5	0.06977 (3)	2.56 (2)
Nd(2)	0.06959 (2)	0.45005 (8)	0.42406 (3)	2.16 (2)
Cl(1)	0.4807 (2)	0.4324 (5)	0.2417 (2)	5.3 (2)
Cl(2)	0.1970 (2)	0.5012 (6)	0.0108 (4)	9.0 (2)
Cl(3)	0.1393 (1)	0.1805 (5)	0.7155 (2)	4.4 (1)
Cl(4)	0.1392 (2)	0.2698 (4)	0.2151 (2)	4.2 (1)
Cl(5)	0.3020 (2)	0.4432 (5)	0.4926 (3)	5.8 (2)
Cl(6)	0	0.5121 (8)	0	6.1 (2)
Cl(7)	0.5	0.4303 (6)	0.5	5.3 (2)
O(1)	0.0717 (3)	0.5025 (11)	0.5498 (4)	3.5 (3)
O(2)	0.0346 (3)	0.5732 (10)	0.6309 (4)	3.1 (3)
O(3)	0.5263 (3)	0.4442 (14)	0.0925 (4)	4.5 (3)
O(4)	0.6004 (4)	0.3787 (11)	0.0668 (4)	3.6 (3)
O(5)	0.3727 (4)	0.6961 (12)	0.1185 (5)	4.6 (4)
O(6)	0.4939 (4)	0.7549 (16)	0.1233 (7)	6.2 (5)
O(7)	0.3197 (4)	0.4031 (13)	0.0105 (6)	5.0 (4)
O(8)	0.3756 (5)	0.7198 (12)	-0.0377 (5)	5.2 (4)
O(9)	0.4331 (5)	0.1765 (19)	0.0838 (7)	8.2 (6)
O(10)	0.0205 (4)	0.5107 (13)	0.2600 (4)	4.5 (4)
O(11)	0.1190 (6)	0.2635 (16)	0.3709 (7)	8.2 (6)
O(12)	0.1310 (4)	0.2398 (12)	0.5328 (5)	4.2 (3)
O(13)	0.1765 (4)	0.5520 (15)	0.4743 (7)	6.4 (5)
O(14)	0.0727 (3)	0.7547 (9)	0.4235 (5)	3.6 (3)
O(15)	0.0016 (4)	0.2159 (11)	0.3847 (7)	5.7 (4)
N(1)	0.1198 (5)	0.8006 (15)	0.7185 (7)	4.8 (5)
N(2)	0.6506 (5)	0.1374 (15)	0.1775 (6)	4.5 (4)
C(1)	0.0745 (4)	0.5871 (11)	0.6106 (5)	2.1 (3)
C(2)	0.1262 (5)	0.7063 (15)	0.6522 (6)	3.2 (4)
C(3)	0.1900 (5)	0.6280 (21)	0.6975 (8)	5.4 (6)
C(4)	0.2061 (10)	0.6246 (27)	0.7844 (11)	9.8 (10)
C(5)	0.1724 (10)	0.7389 (27)	0.8005 (8)	8.7 (10)
C(6)	0.5719 (5)	0.3596 (16)	0.1047 (6)	3.4 (4)
C(7)	0.6004 (5)	0.2372 (16)	0.1793 (7)	3.5 (4)
C(8)	0.6352 (8)	0.3214 (22)	0.2679 (8)	6.7 (7)
C(9)	0.7031 (9)	0.2946 (60)	0.2920 (17)	20.0 (25)
C(10)	0.7114 (7)	0.1690 (31)	0.2630 (16)	12.0 (12)

the Ln^{III} :amino acid ratio is 1:1, a linear polymer is formed (Głowiak & Dao, 1988). The structures of the title complexes are of interest because (i) Cl ions are involved in the coordination sphere of the Ln ions, and (ii) both polymer and dimer structures are formed at an Ln^{III} :amino acid ratio of 1:1.

Experimental. The following applies to both (I) and (II) unless stated otherwise. Rose-coloured crystals obtained from aqueous solutions of NdCl_3 and β -alanine (I) or NdCl_3 and L-proline (II). The crystals show limited stability in air. Parallelepiped $0.30 \times 0.30 \times 0.50$ mm (I) and $0.35 \times 0.40 \times 0.50$ mm (II) were cut from larger crystals for intensity measurement. D_m by flotation in $\text{C}_2\text{H}_4\text{Br}_2/\text{CHBr}_3$ (I) and $\text{CHCl}_3/\text{C}_2\text{H}_4\text{Br}_2$ (II), monoclinic from Weissenberg photographs; Syntex $P2_1$ four-circle diffractometer,

Table 2. Bond distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

(I)			
Nd—Cl(1)	2.838 (2)	Nd—O(3)	2.482 (5)
Nd—Cl(2)	2.807 (2)	Nd—O(4)	2.519 (6)
Nd—Cl(3)	2.835 (2)	Nd—O(5)	2.529 (5)
Nd—O(1)	2.389 (5)	Nd—O(2)	2.378 (5)
C(1)—O(1)	1.252 (8)	C(1)—O(2)	1.266 (9)
C(1)—C(2)	1.514 (10)	C(2)—C(3)	1.505 (12)
C(3)—N	1.485 (11)		
O(1)—C(1)—O(2)	123.0 (6)	C(1)—C(2)—C(3)	114.4 (6)
O(1)—C(1)—C(2)	121.0 (6)	N—C(3)—C(2)	113.9 (7)
O(2)—C(1)—C(2)	116.0 (6)		
(II)			
Nd(1)—O(3)	2.335 (9)	Nd(2)—O(1)	2.321 (7)
Nd(1)—O(4 ⁱⁱ)	2.440 (7)	Nd(2)—O(2 ⁱⁱⁱ)	2.481 (9)
Nd(1)—O(5)	2.547 (11)	Nd(2)—O(10)	2.655 (7)
Nd(1)—O(6)	2.523 (12)	Nd(2)—O(11)	2.453 (15)
Nd(1)—O(7)	2.474 (11)	Nd(2)—O(12)	2.479 (8)
Nd(1)—O(8)	2.482 (9)	Nd(2)—O(13)	2.491 (12)
Nd(1)—O(9)	2.640 (16)	Nd(2)—O(14)	2.479 (7)
Nd(1)—Cl(1)	2.784 (3)	Nd(2)—O(15)	2.407 (10)
C(1)—O(1)	1.28 (1)	C(6)—O(3)	1.25 (2)
C(1)—O(2)	1.24 (1)	C(6)—O(4)	1.24 (2)
C(1)—C(2)	1.48 (2)	C(6)—C(7)	1.54 (2)
C(2)—C(3)	1.52 (2)	C(7)—C(8)	1.56 (2)
C(2)—N(1)	1.52 (2)	C(7)—N(2)	1.51 (2)
C(3)—C(4)	1.43 (2)	C(8)—C(9)	1.54 (4)
C(4)—C(5)	1.39 (3)	C(9)—C(10)	1.22 (5)
C(5)—N(1)	1.50 (2)	C(10)—N(2)	1.55 (3)
O(1)—C(1)—O(2)	121.3 (9)	O(3)—C(6)—O(4)	124.6 (11)
O(1)—C(1)—C(2)	116.4 (9)	O(3)—C(6)—C(7)	118.8 (11)
O(2)—C(1)—C(2)	122.3 (9)	O(4)—C(6)—C(7)	116.2 (11)
C(1)—C(2)—C(3)	114.2 (10)	C(6)—C(7)—C(8)	113.7 (11)
C(1)—C(2)—N(1)	108.7 (10)	C(6)—C(7)—N(2)	110.1 (10)
C(2)—C(3)—C(4)	103.8 (14)	C(7)—C(8)—C(9)	100.6 (17)
C(3)—C(4)—C(5)	111.6 (18)	C(8)—C(9)—C(10)	113.7 (28)
C(4)—C(5)—N(1)	109.3 (17)	C(9)—C(10)—N(2)	106.4 (24)
C(5)—N(1)—C(2)	103.8 (12)	C(10)—N(2)—C(7)	106.8 (13)

Symmetry code: (i) $1+x, y, z$; (ii) $1-x, y, -z$; (iii) $-x, y, 1-z$.

graphite monochromator, scintillation counter; cell parameters by least-squares fitting of setting angles of 15 reflections with $20 < 2\theta < 29^\circ$ measured on diffractometer, variable $\theta/2\theta$ scan, scan rate 2.0 to $29.3^\circ \text{ min}^{-1}$ depending on intensity; $2\theta_{\text{max}} = 55.0^\circ$ for (I) and (II), two standards (252 and 360) for (I) and (352 and $6,2, \bar{1}0$) for (II) measured every 50 reflections, mean intensity variation 6.0%. Independent reflections measured: (I) 2580, index range $h\ 0 \rightarrow 7, k\ 0 \rightarrow 21, l\ -16 \rightarrow 16$ and (II) 3815, $h\ 0 \rightarrow 32, k\ 0 \rightarrow 10, l\ -21 \rightarrow 20$; correction for Lorentz and polarization effects; for (I) an absorption correction applied (Walker & Stuart, 1983) to isotropically refined data, max. and min. transmission factors 1.38, 0.79. Calculations performed with programs supplied by Syntex (1976); scattering factors for neutral atoms Nd and Cl corrected for anomalous dispersion, from *International Tables for X-ray Crystallography* (1974, Vol. IV); Nd atoms located from a Patterson map, remaining non-H atoms and H atoms of waters and NH_3^+ group from subsequent difference syntheses, C-bonded H atoms placed geometrically. 2363 amplitudes with $F > 6\sigma(F)$ (I) and 3540 amplitudes with $F > 7\sigma(F)$ (II) for structure solution and refinement, full-matrix least-squares refinement for non-H atoms

with anisotropic thermal parameters, minimizing $\sum w(|F_o| - |F_c|)^2$; $w = 1/\sigma^2(F)$; coordinates and isotropic temperature factors for H atoms were fixed. Final parameters: (I) $\Delta/\sigma = 0.03$, $\Delta\rho = \pm 0.35 \text{ e } \text{Å}^{-3}$, $R = 0.035$, $wR = 0.049$, $S = 4.63$ for 118 variable parameters; (II) $\Delta/\sigma = 0.08$, $\Delta\rho$ between -0.63 and $0.90 \text{ e } \text{Å}^{-3}$, highest peaks around Nd atoms, $R = 0.040$, $wR = 0.047$, $S = 4.21$ for 316 variable parameters. L-Proline was used as an internal chirality standard.

Discussion. Final atomic parameters are given in Table 1. The geometries of the Nd coordination spheres and amino acid molecules are shown in Table 2.*

Structure (I). The carboxyl O(1) atom coordinates to the Nd ion, whereas O(2) is coordinated to Ndⁱ [(i): $1+x, y, z$] to form polymeric chains along the *a* axis. Similar chains occur in [Ho(L-threonine)(H₂O)₅]Cl₃ (Głowiak & Dao, 1988) and [Dy(L- α -alanine)(H₂O)₆]Cl₃ (Głowiak, Legend-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53354 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

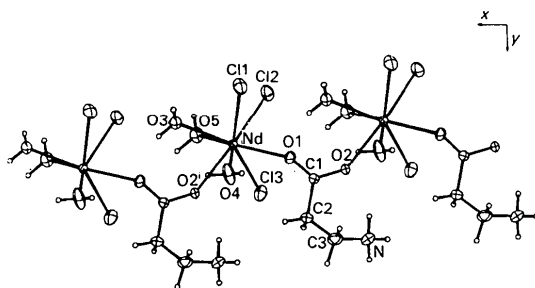


Fig. 1. An ORTEP view of the [NdCl₃(C₃H₇NO₂)(H₂O)₃] structure.

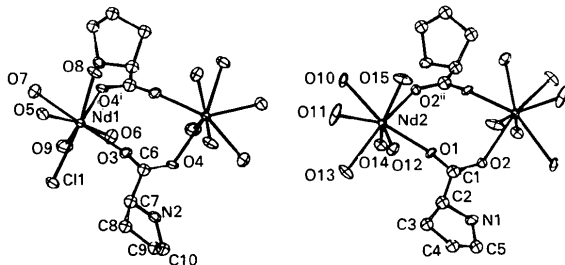


Fig. 2. Perspective view of a characteristic fragment of the [Nd(C₅H₉NO₂)Cl(H₂O)₅]Cl₂·[Nd(C₅H₉NO₂)(H₂O)₆]Cl₃ structure. The atoms of the crystallographic asymmetric unit are labelled as in the text.

Table 3. Hydrogen bonds (Å, °)

(I)	D...A	H...A	D—H...A
O(4)—H(10)···Cl(3 ^{iv})	3.303 (6)	2.44	145
O(4)—H(11)···Cl(3 ^{iv})	3.273 (6)	2.40	158
O(5)—H(12)···Cl(2 ^{iv})	3.186 (5)	2.29	155
N—H(6)···(1 ^v)	3.283 (7)	2.48	143

Symmetry code: (iii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$; (iv) $-x, -y, -z$; (v) $-1+x, \frac{1}{2}-y, -\frac{1}{2}+z$.

(II)	D...A	H...A	D—H...A
O(5)—H(19)···Cl(2 ^{iv})	3.257 (11)	2.40	170
O(5)—H(20)···Cl(3 ^{iv})	3.215 (10)	2.30	165
O(6)—H(21)···Cl(4 ^v)	3.139 (13)	2.01	159
O(6)—H(22)···Cl(6 ^{iv})	3.142 (13)	2.06	178
O(7)—H(23)···Cl(2)	3.181 (13)	2.11	179
O(7)—H(24)···Cl(2 ^{iv})	3.293 (11)	2.11	179
O(8)—H(25)···Cl(2 ^{iv})	3.117 (12)	1.96	177
O(8)—H(26)···Cl(4 ^{iv})	3.112 (10)	2.08	179
O(9)—H(27)···Cl(6 ^{iv})	3.100 (14)	2.10	171
O(9)—H(28)···Cl(2 ^{iv})	3.147 (15)	2.26	159
O(11)—H(32)···Cl(4)	3.150 (14)	2.10	179
O(12)—H(33)···Cl(3)	3.288 (9)	2.27	175
O(12)—H(34)···Cl(5 ^v)	3.107 (11)	2.05	180
O(13)—H(35)···Cl(5)	3.118 (13)	2.09	179
O(13)—H(36)···Cl(5 ^v)	3.232 (13)	2.16	179
O(14)—H(37)···Cl(7 ^v)	3.154 (9)	2.05	174
O(14)—H(38)···Cl(5 ^v)	3.108 (10)	2.23	146
O(15)—H(39)···Cl(7 ^v)	3.157 (10)	2.11	178
O(15)—H(40)···Cl(3 ^v)	3.052 (12)	2.02	177
N(1)—H(8)···Cl(1 ^v)	3.148 (14)	2.40	132
N(1)—H(9)···Cl(3 ^{iv})	3.133 (13)	2.13	173
N(2)—H(18)···Cl(4 ^{iv})	3.113 (13)	2.16	158

Symmetry code: (iii) $\frac{1}{2}-x, \frac{1}{2}+y, -z$; (iv) $\frac{1}{2}-x, \frac{1}{2}+y, 1-z$; (v) $\frac{1}{2}+x, \frac{1}{2}+y, z$; (vi) $-x, y, -z$; (vii) $\frac{1}{2}-x, -\frac{1}{2}+y, -z$; (viii) $-x, -1+y, -z$; (ix) $\frac{1}{2}-x, -\frac{1}{2}+y, 1-z$; (x) $-1-x, y, -z$; (xi) $-1-x, -1+y, -z$; (xii) $x, 1+y, z$; (xiii) $\frac{1}{2}+x, -\frac{1}{2}+y, z$.

ziewicz, Dao & Huskowska, 1990). The torsion angles Nd—O(1)—C(1)—C(2) and Ndⁱ—O(2)—C(1)—C(2) are -19.7 (10) and -177.4 (8)°, respectively, showing that the carboxyl group coordinates in the *syn-anti* bridging mode. Each Nd ion is eight coordinate with three Cl ions, three water molecules and two O atoms from β -alanine molecules. This coordination differs significantly from that observed in the similar dysprosium complex with L- α -alanine (Głowiak, Legendziewicz, Dao & Huskowska, 1990). The nature of the lanthanide coordination changes as the result either of the position of the NH₂ group in the amino acid (β) and/or of the size of the ionic radius of the lanthanide (Nd and Dy). It is interesting that all β -alanine molecules lie on one side of the Nd ion (see Fig. 1). Up to now, such an arrangement of carboxylic acid ligands in lanthanide complex structures has not been found.

Structure (II). The crystal contains dinuclear units of two types involving Nd(1) and Nd(2) respectively. Both types of unit have diad symmetry and in both the Nd ions are linked by the carboxyl groups of a pair of L-proline ligands. However, the Nd(1) ion is attached to a Cl ion while in the coordination of Nd(2) this is replaced by a water molecule. ORTEP views (Johnson, 1976) of these dinuclear units are given in Fig. 2. They involve Nd...Nd distances [Nd(1)···Nd(1ⁱⁱⁱ) = 5.410 (1) and Nd(2)···Nd(2ⁱⁱ) =

5.467 (1) Å; (ii) $1 - x, y, -z$, (iii) $-x, y, 1 - z$] which are shorter than the Nd...Nd distance in the polymer of structure (I) [6.018 (2) Å]. The presence of two carboxylate bridges in (II) may explain this. The conformations of the two independent proline rings in (II) are defined as E, C , [$Q = 0.21$ (2), 0.27 (3) and $\varphi = 212$ (6)°, 250 (4)°, respectively] (Cremer & Pople, 1975). The high temperature factors of atoms C(9) and C(10) and some anomalous interatomic distances [especially C(9)—C(10) = 1.23 (5) Å] suggests slight disorder of the proline rings.

It is interesting to note that, in both complexes, the coordination sphere involves both Cl and O atoms. This is not observed in other lanthanide complexes with carboxylic acids. The greater length of the Nd—Cl bonds compared with the Nd—O bonds causes rather large distortions of the coordination polyhedra around the Nd ions. The C—C, C—O and C—N bond lengths are within normal

ranges. For both complexes the crystal structures are stabilized by hydrogen bonds of type O—H...Cl and N—H...Cl (Table 3).

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Structure of a Five-Coordinate Copper(II) Complex: (2,2'-Bipyridine)[4,4,4-trifluoro-1-(2-thienyl)butane-1,3-dionato-O][4,4,4-trifluoro-1-(2-thienyl)butane-1,3-dionato-O,O']copper(II)

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Abstract. $[\text{Cu}(\text{C}_8\text{H}_4\text{F}_3\text{O}_2\text{S})_2(\text{C}_{10}\text{H}_8\text{N}_2)]$, $M_r = 662.08$, triclinic, $P\bar{1}$, $a = 10.108$ (2), $b = 14.455$ (3), $c = 9.068$ (2) Å, $\alpha = 96.11$ (1), $\beta = 90.92$ (1), $\gamma = 96.12$ (1)°, $V = 1309.4$ Å³, $Z = 2$, $D_x = 1.68$ g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 11.06$ cm⁻¹, $T = 298$ K, $F(000) = 666$, $R = 0.059$, $wR = 0.072$ for 2900 unique observed [$I > 3\sigma(I)$] reflections. The title complex contains Cu^{II} in a square-pyramidal environment; the axial ligand is an O atom from the 4,4,4-trifluoro-1-(2-thienyl)butane-1,3-dionato-O moiety.

Introduction. Complexes of β -diketones and their derivatives with N bases have been extensively investigated in recent years, since they are extracting agents, chemiluminescent materials and chemical shift agents. It is important to determine the molecular structure and to explore the relationship between the structures and properties of these compounds. We have studied the structures of bis[4,4,4-trifluoro-1-(2-thienyl)butane-1,3-dionato-O,O']copper(II), and its adducts with pyridine, dimethyl sulfoxide and quinoline (Yu, Xu, You, Lu, Shi, Liu & Lin, 1988; Liu, Lin, Xu, Yu & You, 1986; Li You, Yao, Huang & Wang, 1987; Yu, Zhu, Xu, Gou, You, Liu & Lin,

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