

Fig. 1. Molecular structure and labeling scheme for $[\text{Nb}(\text{Cl})(\text{O})(\text{C}_5\text{H}_5)_2]$.

Atomic and equivalent isotropic thermal parameters are given in Table 1 and selected bond lengths in Table 2. Fig. 1 shows the molecular structure of the compound. A unit-cell packing diagram is shown in Fig. 2.*

Related literature. The title compound was previously prepared by the reaction of niobocene

* Lists of structure factors, anisotropic thermal parameters and full lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54012 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of uns-*cis-mer* (Pyridine-2-carboxylato-*N,O*)(ethylenediaminediacetato-*N,N',O,O'*)cobalt(III)

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Abstract. $\text{C}_{12}\text{H}_{14}\text{CoN}_3\text{O}_6$, $M_r = 355.19$, orthorhombic, $Pccn$, $a = 8.838(3)$, $b = 24.221(5)$, $c = 12.553(2)$ Å, $V = 2687.03$ Å³, $Z = 8$, $D_x = 1.756$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.7107$ Å, $\mu = 12.4$ cm⁻¹,

$F(000) = 1456$, room temperature, final $R = 0.052$ for 2044 unique reflections. The geometry about the Co atom is roughly octahedral with the edda (ethylenediaminediacetato) ligand assuming an uns-*cis* configuration such that there is one out-of-plane acetate (*R*) ring and one in-plane amino acidate (*G*) ring.

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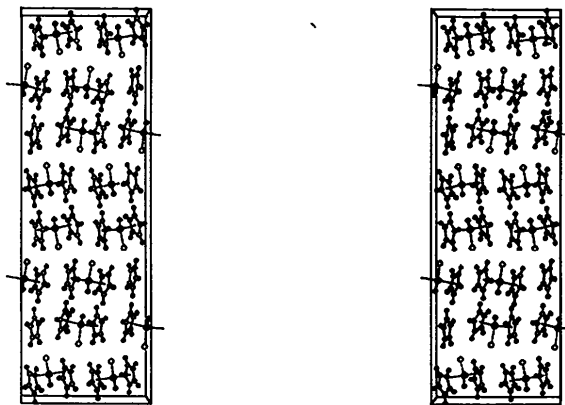


Fig. 2. Unit-cell packing diagram as viewed down the c axis.

dichloride with various organolithium compounds followed by oxidation with atmospheric oxygen (Baukova, Knizhnikov, Lemenovskii, Nesmeyanov & Perevalova, 1976). A similar compound, μ -oxo-bis[bis(η^5 -cyclopentadienyl)]chloroniobium(V) bis(tetrafluoroborate), has been crystallographically characterized (Cameron, Critchley, Denton, Forder, Prout & Rees, 1974).

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Table 1. Fractional atomic coordinates ($\times 10^4$, Co $\times 10^5$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$, Co $\times 10^4$) for Co(edda)(pic)

	x	y	z	U_{eq}
Co	16136 (13)	13706 (5)	16288 (10)	229 (2)
O1	3312 (7)	1841 (2)	1763 (5)	30 (1)
O2	5795 (7)	1806 (2)	2115 (5)	39 (2)
O3	-53 (6)	889 (2)	1515 (5)	29 (1)
O4	-1369 (8)	393 (3)	348 (6)	59 (2)
O5	1482 (7)	1341 (2)	3144 (4)	32 (1)
O6	161 (9)	1639 (3)	4482 (5)	55 (2)
N1	3111 (8)	793 (3)	1564 (6)	27 (2)
N2	1461 (8)	1453 (3)	88 (6)	27 (2)
HN2	2518 (8)	1485 (3)	-339 (6)	26 (6)*
N3	283 (8)	1993 (3)	1749 (6)	21 (2)
HN2	864 (8)	2378 (3)	1986 (6)	26 (6)*
C1	4598 (11)	1580 (4)	1875 (7)	24 (2)
C2	4522 (10)	984 (3)	1694 (7)	22 (2)
C3	5766 (10)	636 (3)	1668 (8)	32 (2)
H3	6886 (10)	808 (3)	1765 (8)	26 (6)*
C4	5551 (12)	76 (4)	1508 (8)	34 (3)
H4	6519 (12)	-196 (4)	1514 (8)	26 (6)*
C5	4068 (12)	-133 (4)	1371 (7)	31 (3)
H5	3963 (12)	-572 (4)	1240 (7)	26 (6)*
C6	2830 (11)	239 (3)	1405 (7)	29 (2)
H6	1679 (11)	98 (3)	1304 (7)	26 (6)*
C7	-341 (11)	721 (4)	562 (8)	34 (2)
C8	659 (11)	951 (4)	-314 (7)	40 (2)
H81	-14 (11)	1048 (4)	-1007 (7)	26 (6)*
H82	1488 (11)	641 (4)	-517 (7)	26 (6)*
C9	623 (11)	1985 (3)	-109 (8)	29 (2)
H91	42 (11)	1959 (3)	-864 (8)	26 (6)*
H92	1406 (11)	2328 (30)	-126 (8)	26 (6)*
C10	-529 (11)	2064 (3)	729 (7)	28 (2)
H101	-1432 (11)	1766 (3)	656 (7)	26 (6)*
H102	-994 (11)	2476 (3)	689 (7)	26 (6)*
C11	-590 (12)	1913 (4)	2706 (7)	30 (2)
H111	-980 (12)	2303 (4)	3019 (7)	26 (6)*
H112	-1551 (12)	1656 (4)	2514 (7)	26 (6)*
C12	378 (12)	1615 (3)	3534 (8)	36 (2)

* Isotropic temperature factors.

Table 2. Selected bond lengths (\AA) and angles ($^\circ$) for Co(edda)(pic)

O1—Co	1.892 (6)	O3—Co	1.884 (5)
O5—Co	1.908 (5)	N1—Co	1.928 (7)
N2—Co	1.948 (7)	N3—Co	1.917 (7)
O3—Co—O1	178.6 (3)	O5—Co—O1	88.9 (3)
O5—Co—O3	90.2 (3)	N1—Co—O1	84.1 (3)
N1—Co—O3	94.8 (3)	N1—Co—O5	93.2 (3)
N2—Co—O1	94.7 (3)	N2—Co—O3	86.2 (3)
N2—Co—O5	171.6 (3)	N2—Co—N1	94.6 (3)
N3—Co—O1	90.3 (3)	N3—Co—O3	90.8 (3)
N3—Co—O5	85.0 (3)	N3—Co—N1	174.2 (3)
N3—Co—N2	87.5 (3)		

The pic (picolinato, pyridine-2-carboxylato) ligand bonds to the remaining coordination sites such that the three nitrogen donors are meridional.

Experimental. The title compound was prepared by the reaction of Na[Co(edda)Co₃] (Halloran, Gillie & Legg, 1978) with picolinic acid in dilute hydrochloric acid (stoichiometric quantity) at 322 K. The solution was stirred for 2 h, filtered and concentrated to give the product as a pink-red microcrystalline powder. Suitable crystals were grown from a saturated aqueous solution at 328 K.

Intensities were measured at room temperature on an Enraf-Nonius CAD-4 diffractometer (graphite-monochromated Mo $K\alpha$ radiation); crystal dimen-

sions $0.25 \times 0.15 \times 0.15$ mm. The cell dimensions were determined from least-squares refinement of 23 accurately measured reflections ($16 \leq \theta \leq 18^\circ$). 2717 reflections were measured up to $\theta = 25^\circ$ ($0 \leq h \leq 10$, $0 \leq k \leq 28$, $0 \leq l \leq 14$) in the $\omega:2\theta$ scan mode [scan width $(0.6 + 0.35 \tan \theta)^\circ$, scan speed range 0.9 to $5.5^\circ \text{ min}^{-1}$]. Three standard reflections (379, 066, 0,0,10) showed a 10.3% variation over the data collection, and were linearly corrected. The data were also corrected for Lorentz and polarization effects. The structure was solved by Patterson methods and refined (Sheldrick, 1976) by full-matrix least-squares refinement of 200 parameters using 1481 observed reflections with $|F| > 3\sigma(F)$. All non-H atoms were located from difference maps and refined anisotropically. H atoms were placed in calculated positions and refined with a common isotropic temperature factor. At convergence $R = 0.052$, $wR = 0.044$ [$w = 2.07/\sigma^2(F)$], $(\Delta/\sigma)_{\text{max}} = 0.023$, $\Delta\rho_{\text{max}} = 0.56$, $\Delta\rho_{\text{min}} = -0.56 \text{ e \AA}^{-3}$. Scattering factors used were those for C, H, N, O atoms given in SHELX76 (Sheldrick, 1976) and those for Co⁰ corrected for f' and f'' (Hamilton & Ibers, 1974). Atomic parameters are given in Table 1,* bond distances and angles around the Co atom in Table 2 and the atom-numbering scheme is shown in Fig. 1.

Related literature. The bond lengths and bond angles in the coordination sphere of cobalt do not differ significantly from those of the other edda Co^{III} complexes where the edda has *uns-cis* coordination, namely in [Co(edda)(R-1,2-diaminopropane)]Cl.H₂O (Halloran, Caputo, Willett & Legg, 1975) and [Co(edda)(CO₃)]guanidinium (Egan, 1988) or in [Co(edda-acetate)(ethylenediamine)]Cl.3H₂O (Chuklanova, Polynova, Poznyak, Bel'skii & Porai-Koshits 1983) and [Co(edda)(ethylenediamine)]ClO₄ (Chuklanova, Polynova, Sobolev & Porai-Koshits

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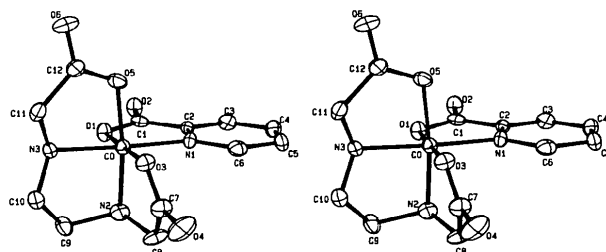


Fig. 1. Stereoview of the molecule with the atomic numbering scheme.

1984) where the edda coordination to the cobalt is *trans*. The three chelate rings of edda have a $\delta\lambda\delta$ conformation and the nitrogen configurations are *R* and *S*, the same as in [Co(edda)(*R*-1,2-diaminopropane)]⁺ (Halloran *et al.*, 1975), but differing from the $\delta\lambda\lambda$ conformation and *SS* or *RR* nitrogen configuration in the complex [Co(edda)(CO₃)] (Egan, 1988). The picolinate ligand binds the Co atom with bond lengths and angles similar to those in [Co(pipecolinato)(en)₂]²⁺ (Brown, Majeste, Chung & Trefonos, 1977), where pipecolinate is the unsaturated analogue of picolinate. One other cobalt complex of picolinate has been reported, [Co(pic)₃].H₂O (Pellizzi & Pellizzi, 1981).

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Structure of 1,1'-Bis(diphenylphosphinetetramethyl)ferrocene

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Abstract. [Fe(C₂₁H₂₂P)₂], *M_r* = 666.61, triclinic, *P* $\bar{1}$, *a* = 7.698 (3), *b* = 8.663 (2), *c* = 13.724 (4) Å, α = 80.25 (2), β = 83.58 (3), γ = 75.69 (3)°, *V* = 871.6 Å³, *Z* = 1, *D_x* = 1.270 g cm⁻³, λ (Mo *K*α) = 0.71073 Å, μ = 5.490 cm⁻¹, *F*(000) = 352, *T* = 296 (1) K, *R* = 0.047 for 2712 reflections with *I* > 3.5σ(*I*). The title complex, a precursor for bi- or polymetallic compounds, bears a new bifunctional permethylated ligand which is more electron rich than the known diphenylphosphinecyclopentadienyl moiety. The geometry of the complex is affected little by the substitution of methyl groups for H atoms in the rings. The molecule is centrosymmetric with the Fe atom at the crystallographic inversion centre.

Experimental. The compound was synthesized by reaction of FeCl₂ with C₅Me₄PPh₂Li in THF. Recrystallization from CHCl₃/pentane afforded yellow single crystals. A specimen (0.08 × 0.08 × 0.15 mm) was mounted on an Enraf-Nonius CAD-4 diffractometer. The unit cell was determined and refined from angle data of 25 randomly selected reflections in the range 11 < θ < 19° (CAD-4 rou-

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tines). Intensities measured with filtered Mo *K*α radiation, ω–2θ scan, 2 < θ < 28°. Data (4506 reflections) collected in the range *h* 0 → 10, *k* –11 → 11, *l* –18 → 18 corresponding to 2712 unique reflections with *I* > 3.5σ(*I*) used in structure determination and refinement. Intensities of three standard reflections (108, 136 and 008) measured at 60 min intervals showed a positive (0.6%) linear decay which was corrected. Lorentz–polarization correction. Empirical absorption corrections [ψ scan; transmission factors: min. 54.00, max. 99.94%, and *DIFABS* (Walker & Stuart, 1983) absorption: min. 0.7017, max. 1.2214]. Structure solved by heavy-atom, least-squares and difference Fourier techniques and refined (on *F*) by full-matrix least squares with unit weights and anisotropic thermal parameters for non-H atoms. All H atoms of the methyl groups located from difference Fourier map and refined with *B*_{iso} fixed at 5.0 Å², those of the phenyl groups placed in calculated positions, riding on the C atoms bearing them and included in the final refinements with *B*_{iso} = 5.0 Å². Secondary-extinction coefficient refined to $g = 2.7636 \times 10^{-7} \{F_c = F_c/[1 + g(F_c)^2 Lp]\}$. $\Delta/\sigma_{\max} = 0.02$ for non-H atoms, $\Delta\rho$ max. and min. +0.5 and –0.6 e Å⁻³. Final *R* factor:

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