Acta Cryst. (1997). C53, 1007–1010

[2,2,2-Crypt-K]$_2$[Cr$_2$(CO)$_{10}$]: the First Dinuclear Metal Decacarbonyl Dianion Structure with an Eclipsed Geometry

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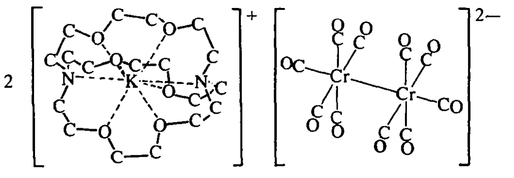
(*Received 9 January 1997; accepted 18 March 1997*)

Abstract

Bis[(1,10-diaza-4,7,13,16,21,24-hexaoxabicyclo[8.8.8]hexacosane)potassium(+)] eclipsed-decacarbonyldichromate(*Cr*—*Cr*)(2−), [K(C$_{18}$H$_{36}$N$_2$O$_6$)]$_2$[Cr$_2$(CO)$_{10}$], was obtained from the reaction between the trigonal bipyramidal Pb$_2$Ch$_3^{2-}$ anions (Ch = Se, Te) and Cr(CO)$_6$ in ethylenediamine (en) solvent and in the presence of the macrobicyclic ligand, 2,2,2-crypt. The anion geometry is eclipsed (approximate D_{4h} point symmetry) and contrasts with the staggered geometry (approximate D_{4d} point symmetry) observed for the anion in the [2,2,2-crypt-Na]$^+$, DME-solvated K$^+$ (DME is 1,2-dimethoxyethane) and PPN$^+$ {PPN$^+$ is bis[bis(triphenylphosphine)iminium]} salts.

Comment

The reactivities between homopolyatomic Zintl anions and transition metal compounds have only been explored recently (Eichhorn, Haushalter & Pennington, 1988; Eichhorn & Haushalter, 1990; Ansari & Ibers, 1990; Roof & Kolis, 1993). Many of the resulting compounds reported to date are structurally related to their hydrocarbon analogs and can be obtained from simple metathesis reactions, *e.g.* M_7^{3-} + LM'(CO)$_3$ → (CO)$_3M'M_7^{3-}$ + L, where M = P, Sb or As, M' = Cr, Mo or W, and L is mesitylene or cycloheptatriene (Charles, Eichhorn, Rheingold & Bott, 1994), and P$_7^{3-}$ + [Ni(CO)$_2$(PPh$_3$)$_2$] → η^4-[P$_7$Ni(CO)]$^{3-}$ + CO + 2PPh$_3$ (Charles, Fettinger, Bott & Eichhorn, 1996). Other compounds, however, have been obtained from less obvious synthetic pathways and possess novel structures, *e.g.* *nido*-[(CO)$_3$Ni$_3$Sb$_7$]$^{3-}$, which was obtained from the reaction between Sb$_7^{3-}$ and [Ni(CO)$_2$(PPh$_3$)$_2$] in a 1:1 molar ratio (Charles, Eichhorn & Bott, 1993). We recently explored the reactions between Zintl anions and transition metal compounds to include classically bonded heteropolyatomic Zintl anions and structurally characterized the compound [2,2,2-crypt-K]$_2$[Te{Mo(CO)$_5$}$_3$] by X-ray crystallography (DiCiommo, Mercier, Pirani & Schrobilgen, 1997), which was obtained from the reaction between the trigonal bipyramidal Pb$_2$Te$_3^{2-}$ anion (Björgvinsson, Sawyer & Schrobilgen, 1991) and Mo(CO)$_6$. In the present study, we investigated the reactions between the Pb$_2$Ch$_3^{2-}$ (Ch = Se, Te) anions (Björgvinsson, Sawyer & Schrobilgen, 1987) and Cr(CO)$_6$, and obtained the eclipsed conformer, (I), of the Cr$_2$(CO)$_{10}^{2-}$ anion, which represents the first reported molecular structure of an eclipsed conformer of an [M_2(CO)$_{10}$]$^{2-}$ (M = Cr, Mo, W) dianion.



(I)

The experimental process involved the syntheses of the ternary KPb$_x$Ch (Ch = Se, Te; x = 0.49 for Se and 0.65 for Te) alloys by fusion of the elements followed by extraction in ethylenediamine (en) in the presence of a stoichiometric excess of 2,2,2-crypt with respect to K$^+$. The resulting orange (Se) and deep-red (Te) solutions, which had been previously shown by ^{207}Pb, ^{77}Se and ^{125}Te NMR spectroscopy to contain only the trigonal-bipyramidal Pb$_2$Se$_3^{2-}$ and Pb$_2$Te$_3^{2-}$ anions (Björgvinsson, Sawyer & Schrobilgen, 1987), respectively, were allowed to react with Cr(CO)$_6$, giving rise to olive-green solutions which slowly evolved CO gas and deposited amorphous grey metallic precipitates and metallic mirrors on the glass reactor surfaces. No ^{207}Pb, ^{77}Se and ^{125}Te NMR resonances were observed for the olive-green solutions at 273 K, which suggested either the absence of Pb- and Ch-containing species, the presence of paramagnetic species or intermediate chemical exchange on the NMR time scale. Crystals of [2,2,2-crypt-K]$_2$[Cr$_2$(CO)$_{10}$], (I), were obtained from the olive-green solutions by the slow vapor-phase diffusion of tetrahydrofuran (THF) in the en solutions. The proposed reaction leading to the formation of (I) is given in equation (1):

$$\begin{aligned}&[2,2,2\text{-crypt-K}]_2[\mathrm{Pb_2Ch_3}] + 2\mathrm{Cr(CO)_6}\\ &\quad\rightarrow [2,2,2\text{-crypt-K}]_2[\mathrm{Cr_2(CO)_{10}}] + 2\mathrm{CO}\\ &\qquad\qquad + 2\mathrm{Pb} + 3\mathrm{Ch} \qquad (1),\end{aligned}$$

and contrasts with the reaction between [2,2,2-crypt-K]$_2$[Pb$_2$Te$_3$] and Mo(CO)$_6$ which, under similar experimental conditions, gave rise to the transition metal main-group compound, [2,2,2-crypt-K]$_2$[Te{Mo(CO)$_5$}$_3$] (DiCiommo, Mercier, Pirani & Schrobilgen, 1997).

Compound (I) consists of two crystallographically independent [2,2,2-crypt-K]$^+$ cations and two centrosymmetrically eclipsed [Cr$_2$(CO)$_{10}$]$^{2-}$ units, and crystallizes

Acta Crystallographica Section C
ISSN 0108-2701 © 1997

in the space group $P\bar{1}$. The structure of the [2,2,2-crypt-K]$^+$ cation (Fig. 1) is similar to that determined in [2,2,2-crypt-K]$_4$[Sn$_4$Se$_{10}$], which also crystallizes in $P\bar{1}$ (Campbell, DiCiommo, Mercier, Pirani, Schrobilgen & Willuhn, 1995), and therefore will not be discussed. The $[\mathrm{Cr_2(CO)_{10}}]^{2-}$ anion in (I) (Fig. 2) possesses an eclipsed geometry (approximate D_{4h} point symmetry) and contrasts with the staggered geometry (approximate D_{4d} point symmetry) observed for the anion in [2,2,2-crypt-Na]$_2$[Cr$_2$(CO)$_{10}$] (Borrmann, Pirani & Schrobilgen, 1997), [K]$_2$[Cr$_2$(CO)$_{10}$].DME (Hey-Hawkins & von Schnering, 1991) and [PPN]$_2$[Cr$_2$(CO)$_{10}$] (Lee, Geib & Cooper, 1996), and for the structurally related dianion $[\mathrm{Mo_2(CO)_{10}}]^{2-}$ (Bachman & Whitmire, 1993). The dihedral angles between the equatorial carbonyl groups observed for the anion in (I) are in the range 0.8–2.8°, whereas those observed in the [2,2,2-crypt-Na]$^+$, K$^+$ and PPN$^+$ salts, and in $[\mathrm{Mo_2(CO)_{10}}]^{2-}$, are approximately 45°. The coordination around the Cr atoms is distorted from octahedral geometry with the C$_{eq}$—Cr—C$_{eq}$ (eq = equatorial) bond angles in the range 86.62 (7)–92.36 (7)° or 173.55 (6)–175.56 (6)°, the C$_{eq}$—Cr—C$_{ax}$ (ax = axial) bond angles in the range 90.72 (6)–95.02 (7)°, and C$_{ax}$—Cr—Cr bond angles of 174.3 (6)–176.5 (6)°. The O—C—Cr bond angles are nearly linear and are in the range 174.14 (12)–178.80 (12)°. The Cr—Cr bonds in (I) have lengths of 3.085 (1) and 3.094 (1) Å and are significantly longer ($>3\sigma$) than those observed in the [2,2,2-crypt-Na]$^+$ [2.9761 (8) Å], K$^+$ [2.999 (2) Å] and PPN$^+$ [2.995 (1) Å] salts. This is consistent with a longer bond anticipated for the eclipsed conformer owing to greater steric interactions between the carbonyl groups. The average axial Cr—C bond length in (I) [1.813 (2) Å] is intermediate between those observed in the [2,2,2-crypt-Na]$^+$ [1.828 (2) Å] and in the K$^+$ [1.802 (6) Å] and PPN$^+$ [1.792 (4) Å] salts. The average equatorial Cr—C [1.881 (6) Å] and the average axial [1.168 (3) Å] and equatorial [1.157 (6) Å] C—O bond distances observed in (I) are similar at the 3σ level to the corresponding bond distances observed in the [2,2,2-crypt-Na]$^+$ [equatorial Cr—C 1.876 (4) Å; C—O axial 1.160 (2), equatorial 1.154 (4) Å], K$^+$ [equatorial Cr—C 1.861 (5) Å; C—O axial 1.180 (8), equatorial 1.157 (5) Å] and PPN$^+$ [equatorial Cr—C 1.879 (5) Å; C—O axial 1.177 (4), equatorial 1.154 (5) Å] salts.

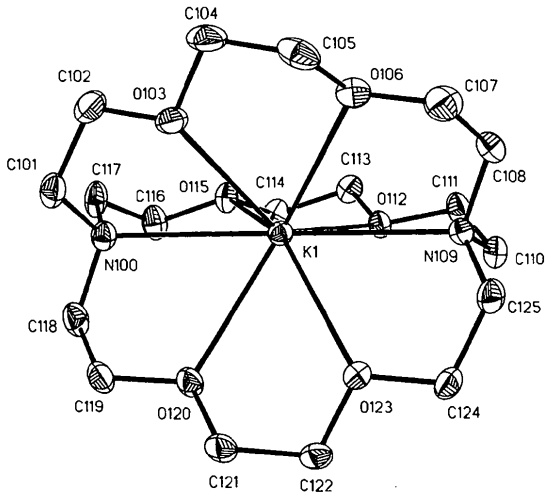


Fig. 1. View of one of the crystallographically independent [2,2,2-crypt-K]$^+$ cations in (I) with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

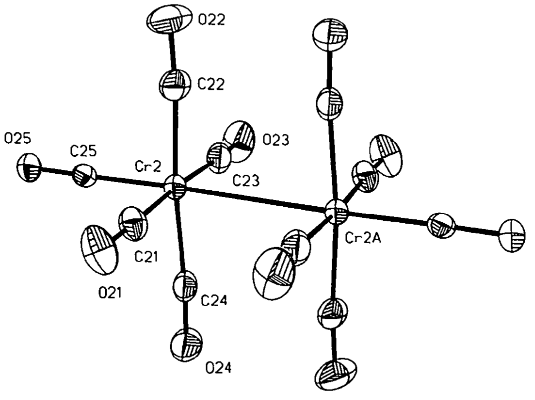


Fig. 2. View of one of the crystallographically independent $[\mathrm{Cr_2(CO)_{10}}]^{2-}$ anions in (I) with displacement ellipsoids drawn at the 50% probability level.

In all structures, the axial Cr—C bonds are shorter than the equatorial Cr—C bonds, which can be attributed to greater electron back-donation from the metal centers into the π^* orbital of the axial CO ligands. The axial C—O bond distances are consequently longer than the equatorial ones.

Experimental

The extreme air- and moisture-sensitive natures of the compounds used required all manipulations to be carried out under rigorously anhydrous conditions, in a nitrogen atmosphere drybox (two-station, Vacuum Atmospheres Model DLS, with moisture and oxygen levels < 0.1 p.p.m.; for general solid and crystal handling), on a general-purpose grease-free glass vacuum line equipped with Pyrex glass/Teflon stopcocks (J. Young Scientific Glassware) and in a glovebag (for solution handling) which had been purged with dry nitrogen for at least 12 h prior to use. Solutions of the $\mathrm{Pb_2Ch_3}^{2-}$ (Ch = Se, Te) anions were prepared as previously described (Björgvinsson, Sawyer & Schrobilgen, 1987) by loading the following quantities of reagents into one arm of a two-arm reaction vessel. $\mathrm{Pb_2Se_3}^{2-}$: KPb$_{0.49}$Se, 0.0783 g, 0.267 mmol; 2,2,2-crypt, 0.1094 g, 0.290 mmol. $\mathrm{Pb_2Te_3}^{2-}$: KPb$_{0.65}$Te, 0.0922 g, 0.420 mmol; 2,2,2-crypt, 0.1600 g, 0.425 mmol. The resulting orange (Se) and deep-red (Te) solutions were allowed to react with Cr(CO)$_6$ (Se 0.0392 g, 0.178 mmol; Te 0.0525 g, 0.238 mmol) giving rise to olive-green solutions which slowly evolved CO gas and deposited amorphous grey metallic precipitates and metallic mirrors on the glass reactor surfaces. The CO gas was periodically pumped off under dynamic vacuum. After one week, the green en solutions were decanted off the unreacted alloy residues into the second arm of the Pyrex vessels. An excess of THF (1:2 *v*/*v*) was then condensed under vacuum onto the unreacted alloy residues at 273 K while the en solutions were maintained at room temperature. Crystals of (I) were obtained by vapor-phase diffusion of the THF into the en solutions under static vacuum and at room temperature. Crystal data were collected on a single crystal (sealed under dry nitrogen in a 0.5 mm Lindemann glass capillary) obtained from the reaction between $[Pb_2Se_3]^{2-}$ and $Cr(CO)_6$. The reaction between $[Pb_2Te_3]^{2-}$ and $Cr(CO)_6$ yielded crystals with identical habits [unit-cell dimensions (Ag $K\alpha$ radiation) obtained on a single crystal: a = 11.096 (4), b = 12.308 (5), c = 20.674 (9) Å, α = 87.34 (4), β = 85.58 (3), γ = 88.23 (3)°, V = 2807.3(20) Å^3] and were therefore not studied further. A Stoe Imaging Plate Diffractometer System (Stoe & Cie, 1995) equipped with a one-circle goniometer and a graphite monochromator was used for data collection. Data were collected in two stages: (*a*) 500 exposures (4.5 min per exposure) were obtained at 48 mm with $0 \leq \varphi \leq 200°$ and with the crystal oscillated through 0.4° in φ; (*b*) 200 exposures (2 min per exposure) were obtained at 125 mm with $0 \leq \varphi \leq 200°$ and with the crystal oscillated through 1° in φ. The two data sets were merged and the final data set contained 92% of the calculated number of reflections.

Crystal data

$[K(C_{18}H_{36}N_2O_6)]_2$-$[Cr_2(CO)_{10}]$
M_r = 1215.28
Triclinic
$P\overline{1}$
a = 11.140 (2) Å
b = 12.358 (2) Å
c = 20.727 (4) Å
α = 87.34 (3)°
β = 85.66 (3)°
γ = 88.21 (3)°
V = 2841.1 (9) Å^3
Z = 2
D_x = 1.421 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 1727 reflections
θ = 3–58°
μ = 0.607 mm^{-1}
T = 150 (2) K
Irregular
0.51 × 0.43 × 0.37 mm
Yellow

Data collection

Stoe Imaging Plate Diffractometer System (IPDS)
Oscillation scans
Absorption correction: none
34 650 measured reflections
13 846 independent reflections

$R_{\rm int}$ = 0.0358
$\theta_{\rm max}$ = 29°
$h = -13 \rightarrow 13$
$k = -17 \rightarrow 17$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0295
$wR(F^2)$ = 0.0833
S = 0.989
13 720 reflections
973 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 0.703P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max}$ = 0.002
$\Delta\rho_{\rm max}$ = 0.479 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.411 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Cr1—Cr1$^{\rm i}$	3.094 (1)	Cr2—Cr2$^{\rm ii}$	3.085 (1)
Cr1—C11	1.888 (2)	Cr2—C21	1.881 (2)
Cr1—C12	1.880 (2)	Cr2—C22	1.881 (2)
Cr1—C13	1.8782 (15)	Cr2—C23	1.881 (2)
Cr1—C14	1.882 (2)	Cr2—C24	1.879 (2)
Cr1—C15	1.8148 (14)	Cr2—C25	1.8112 (14)
C11—O11	1.154 (2)	C21—O21	1.160 (2)
C12—O12	1.156 (2)	C22—O22	1.155 (2)
C13—O13	1.157 (2)	C23—O23	1.159 (2)
C14—O14	1.157 (2)	C24—O24	1.154 (2)
C15—O15	1.168 (2)	C25—O25	1.167 (2)
C11—Cr1—C12	89.21 (7)	C21—Cr2—C22	90.90 (8)
C11—Cr1—C13	173.55 (6)	C21—Cr2—C23	175.56 (6)
C11—Cr1—C14	92.36 (7)	C21—Cr2—C24	88.19 (7)
C11—Cr1—C15	92.71 (6)	C21—Cr2—C25	93.46 (6)
C12—Cr1—C13	86.62 (7)	C22—Cr2—C23	90.43 (8)
C12—Cr1—C14	173.94 (6)	C22—Cr2—C24	173.62 (6)
C12—Cr1—C15	95.02 (7)	C22—Cr2—C25	92.03 (7)
C13—Cr1—C14	91.29 (7)	C23—Cr2—C24	90.02 (8)
C13—Cr1—C15	92.56 (6)	C23—Cr2—C25	90.72 (6)
C14—Cr1—C15	90.75 (7)	C24—Cr2—C25	94.32 (7)
C15—Cr1—Cr1$^{\rm i}$	174.3 (6)	C25—Cr2—Cr2$^{\rm ii}$	176.5 (6)
O11—C11—Cr1	174.72 (14)	O21—C21—Cr2	175.22 (12)
O12—C12—Cr1	175.86 (13)	O22—C22—Cr2	175.06 (13)
O13—C13—Cr1	175.08 (12)	O23—C23—Cr2	174.86 (13)
O14—C14—Cr1	174.14 (12)	O24—C24—Cr2	175.59 (12)
O15—C15—Cr1	178.55 (12)	O25—C25—Cr2	178.80 (12)

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

All computations were performed on a Silicon Graphics Inc., Model 4600 PC Workstation. During refinement, 126 reflections with $F_o^2 < -2\sigma(F_o^2)$ were suppressed.

Data collection: *IPDS* (Stoe & Cie, 1995). Cell refinement: *IPDS*. Data reduction: *IPDS*. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1995). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

The authors thank the Natural Sciences and Engineering Research Council of Canada for support in the form of a research grant and the Ontario Ministry of Education and Training for the award of a graduate scholarship to AMP. We are also grateful to Professor A. Simon, Stuttgart, for making the X-ray equipment available.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1178). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1010–1011

A (3,5-Dimethylpyridine-*N*)(*N*-salicylideneglycinato-*O*,*N*,*O*′:*O*″)copper(II) Polymer

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(*Received 20 January 1997; accepted 18 March 1997*)

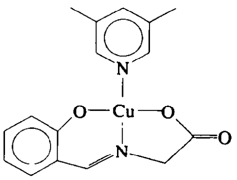
Abstract

The title compound, $[Cu(C_9H_7NO_3)(C_7H_9N)]$, adopts a square-pyramidal Cu^{II} coordination with the tridentate *N*-salicylideneglycinato Schiff base dianion and the 3,5-dimethylpyridine ligand bound in the basal plane. The apex of the pyramid is occupied by a carboxylic O atom from the neighbouring chelate at an apical distance of 2.386 (2) Å leading to infinite one-dimensional chains along the crystallographic *a* axis.

Comment

Copper(II) complexes with tridentate Schiff base dianions of the *N*-salicylideneaminoalkanoato type (TSB^{2-}) represent a relatively simple model for studies of cooperative bonding effects, which can be investigated by EPR (electron paramagnetic resonance) spectroscopy. In these complexes, which are of the general type $[Cu(TSB)(L)]_n$ (Warda, 1994), three donor atoms (O, N, O) of the Schiff base and a fourth donor atom from the neutral ligand *L* (N, O or S) normally define the base of a square pyramid. For isolated (monomeric) structures, the copper coordination can be square planar or square pyramidal when a neutral donor ligand is located in the apical site ($n = 1$; Ueki, Ashida, Sasada & Kakudo, 1969; Warda, Friebel, Sivý, Plesch & Švajlenová, 1996); $n = 4$ is found in the case of (4-ethylpyridine)(*N*-salicylideneglycinato)copper(II) (Warda, 1997). Polymeric structures ($n = \infty$) are achieved when the apical position is occupied by a carboxylic O atom from an adjacent molecule to form infinite zigzag chains (Warda, Friebel, Sivý, Plesch & Bláhová, 1997).

The title molecule, (I), is characterized by a square-pyramidal Cu^{II} coordination with the tridentate Schiff base *N*-salicylideneglycinato dianion and a monodentate ligand (3,5-dimethylpyridine) in the basal plane. The apical Cu—O3*A* bond length [2.386 (2) Å; symmetry code: $x, \frac{1}{2}-y, \frac{1}{2}+z$], originating from a carboxylic O atom of a neighbouring chelate, is comparable with the parent compound of this structure type, namely aqua(*N*-salicylideneglycinato)copper(II) hemihydrate [2.334 (6) Å]. In a new refinement of this crystal structure (Warda, 1994), the apical distance is 2.331 (2) Å. In the title compound, an infinite one-dimensional chain is formed by the Cu···O3···Cu bonds along the crystallographic *a* axis (Fig. 2). EPR patterns display a coupled *g* tensor indicating a distorted antiferrodistortive ordering with $2\gamma = 85°$ (EPR result), where the distance of the differently oriented paramagnetic centres is shorter than the critical distance of 8.3 Å (Warda, 1994). From X-ray data, the intrachain Cu···Cu spacing is 6.0742 (7) Å ($x, \frac{1}{2}-y, \frac{1}{2}+z$) and the tilting angle is 84.60 (11)°, in good agreement with the results of the EPR measurement.



(I)

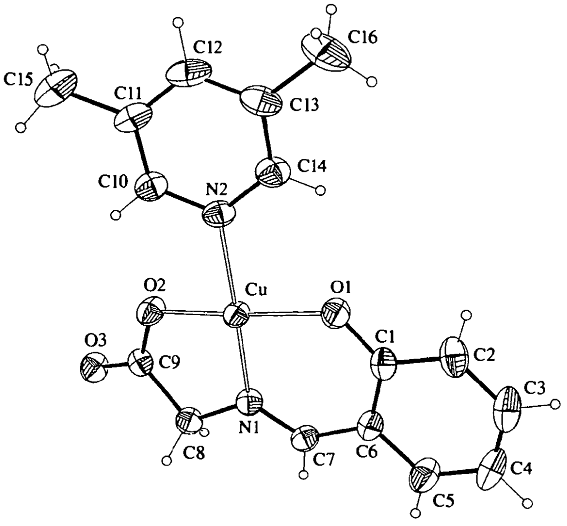


Fig. 1. The asymmetric unit of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Acta Crystallographica Section C
ISSN 0108-2701 © 1997