

N(2)—C(6)	1.47 (4)	N(2)—C(7)	1.57 (4)
N(2)—C(8)	1.48 (4)	C(1)—C(2)	1.52 (4)
C(5)—C(6)	1.47 (4)		
Cl(1)—Hg(1)—Cl(2)	135.4 (3)	Cl(1)—Hg(1)—O(1)	93.5 (6)
Cl(2)—Hg(1)—O(1)	114.6 (7)	Cl(1)—Hg(1)—N(2a)	118.1 (6)
Cl(2)—Hg(1)—N(2a)	98.0 (6)	O(1)—Hg(1)—N(2a)	87.6 (8)
Cl(1)—Hg(2)—Cl(3)	98.2 (3)	Cl(1)—Hg(2)—Cl(4)	100.2 (3)
Cl(3)—Hg(2)—Cl(4)	160.4 (3)	Cl(1)—Hg(2)—O(2)	79.4 (4)
Cl(3)—Hg(2)—O(2)	99.0 (5)	Cl(4)—Hg(2)—O(2)	91.1 (5)
Hg(1)—Cl(1)—Hg(2)	101.8 (3)	Hg(1)—O(1)—C(1)	106 (2)
Hg(2)—O(2)—C(1)	118 (2)	C(2)—N(1)—C(3)	106 (2)
C(2)—N(1)—C(4)	105 (2)	C(3)—N(1)—C(4)	109 (2)
C(2)—N(1)—C(5)	114 (2)	C(3)—N(1)—C(5)	114 (2)
C(4)—N(1)—C(5)	109 (2)	C(6)—N(2)—C(7)	105 (2)
C(6)—N(2)—C(8)	109 (2)	C(7)—N(2)—C(8)	107 (2)
C(6)—N(2)—Hg(1a)	119 (2)	C(7)—N(2)—Hg(1a)	109 (2)
C(8)—N(2)—Hg(1a)	108 (2)	O(1)—C(1)—O(2)	124 (3)
O(1)—C(1)—C(2)	117 (2)	O(2)—C(1)—C(2)	118 (2)
N(1)—C(2)—C(1)	121 (2)	N(1)—C(5)—C(6)	112 (2)
N(2)—C(6)—C(5)	110 (2)		

Symmetry code: (a) $1 - x, 1 - y, 1 - z$.

Table 3. Contact distances (Å)

Hg(1)···Hg(2)	4.144	Hg(1)···Cl(3b)	3.54 (1)
Hg(2)···Cl(2c)	3.43 (1)	Hg(2)···Cl(4d)	3.63 (1)

Symmetry codes: (b) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$; (c) $x - 1, y, z$; (d) $-x, 1 - y, -z$.

The structure was solved by direct and Fourier methods and refined by full-matrix least squares using the *SHELXTL/PC* program (Sheldrick, 1990). H atoms were placed in their idealized locations and included in structure-factor calculations.

Data collection: Siemens *P3* software. Cell reduction: Siemens *P3* software. Data reduction: *SHELXTL-Plus XDISK* (Sheldrick, 1987). Structure solution: *SHELXTL/PC XS*. Structure refinement: *SHELXTL/PC XLS*. Molecular graphics: *SHELXTL/PC XP*. Preparation of material for publication *SHELXTL/PC XPBUL*.

This work is supported by a Hong Kong Research Grants Council Earmarked Grant (CUHK 22/91E).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KH1002). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Allmann, R. (1973). *Z. Kristallogr.* **138**, 366–377.
 Chen, X.-M. & Mak, T. C. W. (1992). *J. Chem. Soc. Dalton Trans.* pp. 1585–1590.
 Kopfmann, G. & Huber, R. (1968). *Acta Cryst.* **A24**, 348–351.
 Lloyd, A. W., Baker, J. A., Smith, G., Olliff, C. J. & Rutt, K. J. (1992). *J. Pharm. Pharmacol.* **44**, 507–511.
 Mak, T. C. W., Yip, W. H., Kennard, C. H. L. & Smith, G. (1990). *Aust. J. Chem.* **43**, 1431–1437.
 Roberts, P. J., Ferguson, G., Goel, R. G., Ogini, W. O. & Restivo, R. J. (1978). *J. Chem. Soc. Dalton Trans.* pp. 253–256.
 Sheldrick, G. M. (1987). *SHELXTL-Plus*. PC version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1990). *SHELXTL/PC User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Wu, D.-D. & Mak, T. C. W. (1994a). *Polyhedron*, **13**, 3333–3339.
 Wu, D.-D. & Mak, T. C. W. (1994b). *J. Mol. Struct.* **326**, 193–202.
 Wu, D.-D. & Mak, T. C. W. (1994c). *J. Chem. Soc. Dalton Trans.* in the press.

Acta Cryst. (1995). **C51**, 859–861

cis-Dipyridine Complex of Bis(ω -nitroacetophenonato)zinc(II)

LARRY R. FALVELLO

Departamento de Química Inorgánica, Universidad de Zaragoza-CSIC, Facultad de Ciencias, Plaza San Francisco s/n, 50009 Zaragoza, Spain

SCOTT HICKE AND TIMOTHY E. MUELLER

Saint Joseph's University, Department of Chemistry, 5600 City Avenue, Philadelphia, PA 19131, USA

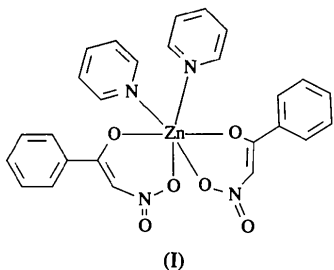
(Received 20 October 1994; accepted 9 November 1994)

Abstract

The title compound, bis(ω -nitroacetophenonato-*O,O'*)-bis(pyridine)zinc(II), $[\text{Zn}(\text{C}_5\text{H}_5\text{N})_2(\text{C}_8\text{H}_6\text{NO}_3)_2]$, was synthesized by the reaction of zinc acetate dihydrate with ω -nitroacetophenone (ω -nap) in ethanol, followed by warming of the resulting product in pyridine. The ω -nap ligands chelate the Zn center forming six-membered rings with a bite angle of 83.23 (8)°. The distance from Zn to the nitro O atom [2.152 (2) Å] is longer than that from Zn to the carbonyl O atom [2.039 (2) Å], indicating that the latter is more strongly bound and suggesting a keto-enol mechanism preceding the binding of ω -nap to the Zn atom.

Comment

The pyridine complex of bis(ω -nitroacetophenonato)-zinc(II) was synthesized as part of a wider study of complexes of first-row *d*-block elements with polyfunctional ligands and of their packing arrangements in the solid state in the presence of hydrogen bonding. The ω -nitroacetophenone (ω -nap) ligand is capable of acting as an acceptor, but not as a donor, in hydrogen-bonding interactions and thus a secondary ligand or unligated moiety capable of hydrogen-bond donation is necessary for studies of the topological response of compounds of ω -nap to the presence of hydrogen-bonded networks. In addition, as was pointed out by Bonamico, Dessy, Fares & Scaramuzza (1972), stabilization of complexes of ω -nap is aided by the presence of an additional ligand and because of the weakly ligating nature of ω -nap. The present complex, (I), synthesized as a molecular prototype, cannot alone form hydrogen bonds in a crystalline environment. It is expected that the molecular structure of this complex is determined by intramolecular energetics in the absence of strong interactions with the crystalline surroundings.



(I)

The title compound crystallizes in space group *I2/a* with four molecules per unit cell. The two ligated molecules of pyridine (py) are *cis* with respect to each other and the Zn center is six-coordinate overall. The molecule sits on a crystallographic twofold axis so that the symmetry element bisects the N(py)—Zn—N(py) bond angle. The ω -nap ligand chelates the Zn center forming a six-membered ring with a bite angle of 83.23 (8)°. The Zn-to-ligand bond distances are all in the expected range of values for this type of complex. The distance from Zn to the ligated atom of the nitro group [Zn1—O3 2.152 (2) Å], however, is longer than the distance from Zn to the O atom of the carbonyl group [Zn1—O1 2.039 (2) Å]. This indicates that the carbonyl O atom of ω -nap is more strongly bound to the Zn center and suggests a prebinding mechanism involving a keto-enol transformation followed by loss of hydrogen, leaving more negative charge at the original carbonyl end of the ligand.

The packing interactions in the crystal structure are relatively weak. There are no significant ring-stacking arrangements and the stability of the crystal is due to van der Waals forces only. The absence of strong intermolecular interactions may explain the fact that the crystals undergo slow decomposition after removal from the mother liquid.

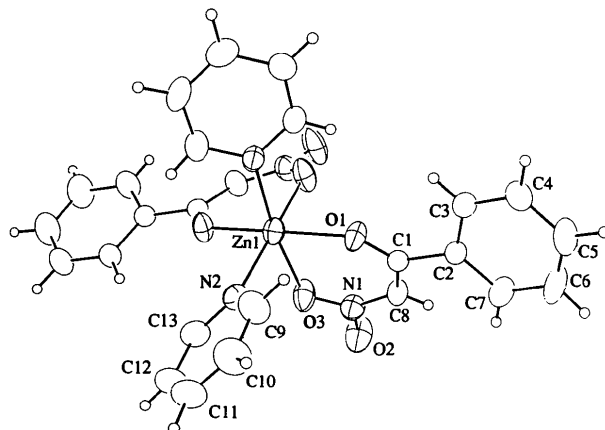


Fig 1. A perspective view of the title complex. Non-H atoms are represented by their 40% probability ellipsoids and H atoms by spheres of arbitrary radii. Unlabeled atoms are related to the labeled atoms by a crystallographic twofold axis.

Experimental

The title compound was prepared by a two-stage synthetic procedure. In the first step, [Zn(OAc)₂].2H₂O (OAc = acetate) was dissolved in 150 ml of deoxygenated 95% ethanol. This was added to a warm suspension of ω -nap. The colorless solution was gently heated until a pale yellow color developed, at which time the solution was allowed to cool, yielding yellow-white crystals of [Zn(ω -nap)₂].2EtOH. These were heated with 20 ml of pyridine and cooled giving the title complex, crystals of which were obtained by the slow evaporation of a dilute acetone solution.

Crystal data

[Zn(C₅H₅N)₂(C₈H₆NO₃)₂]
M_r = 551.85
 Monoclinic
I2/a
a = 13.899 (2) Å
b = 12.156 (2) Å
c = 15.781 (3) Å
 β = 105.84 (2)°
V = 2565.1 (7) Å³
Z = 4
D_x = 1.429 Mg m⁻³

Mo *K* α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 11.4–15.8°
 μ = 1.005 mm⁻¹
T = 294 (2) K
 Well formed plate
 0.44 × 0.35 × 0.18 mm
 Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
 ω -2 θ scans
 Absorption correction: empirical *via* ψ scans
 T_{\min} = 0.834, T_{\max} = 0.933
 2354 measured reflections
 2251 independent reflections

1639 observed reflections
 $[I > 2\sigma(I)]$
 R_{int} = 0.0324
 θ_{max} = 24.97°
 h = 0 → 16
 k = 0 → 14
 l = -18 → 18
 3 standard reflections
 frequency: 180 min
 intensity decay: 1.5%

Refinement

Refinement on F^2
 $R(F)$ = 0.0386
 $wR(F^2)$ = 0.0818
 S = 1.065
 2251 reflections
 212 parameters
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0245P)^2 + 2.3460P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.22 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.24 \text{ e } \text{Å}^{-3}$
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Zn1	1/4	0.04057 (4)	0	0.0424 (2)
O1	0.36371 (14)	0.0257 (2)	0.11264 (12)	0.0477 (5)
O2	0.1795 (2)	-0.2395 (2)	0.1283 (2)	0.0800 (9)
O3	0.1841 (2)	-0.0892 (2)	0.0582 (2)	0.0593 (7)
N1	0.2266 (2)	-0.1561 (2)	0.1179 (2)	0.0471 (7)

C1	0.3805 (2)	-0.0484 (2)	0.1701 (2)	0.0351 (6)
C2	0.4758 (2)	-0.0388 (2)	0.2422 (2)	0.0367 (6)
C3	0.5523 (2)	0.0257 (3)	0.2287 (2)	0.0451 (8)
C4	0.6393 (2)	0.0425 (3)	0.2944 (2)	0.0588 (9)
C5	0.6513 (3)	-0.0054 (4)	0.3749 (3)	0.0721 (12)
C6	0.5766 (3)	-0.0701 (4)	0.3897 (3)	0.0769 (13)
C7	0.4896 (3)	-0.0873 (3)	0.3239 (2)	0.0577 (10)
C8	0.3199 (2)	-0.1381 (3)	0.1726 (2)	0.0455 (8)
N2	0.1668 (2)	0.1566 (2)	0.0506 (2)	0.0422 (6)
C9	0.2129 (3)	0.2297 (3)	0.1110 (2)	0.0613 (10)
C10	0.1627 (3)	0.3008 (3)	0.1506 (3)	0.0725 (12)
C11	0.0605 (3)	0.2988 (3)	0.1268 (3)	0.0660 (11)
C12	0.0116 (3)	0.2262 (3)	0.0642 (2)	0.0573 (9)
C13	0.0671 (2)	0.1575 (3)	0.0287 (2)	0.0483 (8)

Table 2. Selected geometric parameters (Å, °)

Zn1—O1	2.039 (2)	O3—N1	1.262 (3)
Zn1—N2	2.114 (2)	N1—C8	1.364 (4)
Zn1—O3	2.152 (2)	C1—C8	1.385 (4)
O1—C1	1.254 (3)	C1—C2	1.496 (4)
O2—N1	1.242 (3)		
O1 ⁱ —Zn1—O1	169.86 (11)	C1—O1—Zn1	129.9 (2)
O1—Zn1—N2 ⁱ	90.85 (8)	N1—O3—Zn1	128.6 (2)
O1—Zn1—N2	95.92 (9)	O2—N1—O3	117.9 (3)
N2 ⁱ —Zn1—N2	96.30 (13)	O2—N1—C8	119.4 (3)
O1 ⁱ —Zn1—O3	89.33 (8)	O3—N1—C8	122.7 (3)
O1—Zn1—O3	83.23 (8)	O1—C1—C8	126.2 (3)
N2 ⁱ —Zn1—O3	172.26 (9)	O1—C1—C2	116.4 (3)
N2—Zn1—O3	89.28 (9)	C8—C1—C2	117.3 (3)
O3—Zn1—O3 ⁱ	85.68 (13)	N1—C8—C1	126.1 (3)

Symmetry code: (i) $\frac{1}{2} - x, y, -z$.

Upon being harvested from the mother liquid, crystals of the title compound were colorless with well defined faces. After several days in a sealed vial, they began to display a flat white crust, indicating that they had undergone slow decomposition, perhaps by loss of the coordinated pyridine. For X-ray data collection, a small colorless crystal was cut from a sample with evidence of decomposition at the surface. The crystal was covered with a thin layer of epoxy and did not show signs of significant change during the course of the data collection. Three intensity standards changed by less than 1.5% in the 28 h span during which data were gathered.

The empirical absorption correction was based on nine complete ψ scans. The crystal was indexed using a body-centered monoclinic lattice in which the a and c axes were the shortest repeats in the ac plane. The principal lattice repeats were verified by oscillation photographs about a , b , c and the body diagonal [111].

The development and refinement of the structure proceeded routinely. A direct-methods calculation (Sheldrick, 1990) located all of the non-H atoms. All H atoms were located in a difference Fourier map and were refined independently. Anisotropic displacement parameters were used for all non-H atoms.

Data collection: *CAD-4-PC* (Enraf-Nonius, 1993). Cell refinement: *CAD-4-PC*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

We acknowledge support from the Comisión Interministerial de Ciencia y Tecnología, Spain (grant PB92-0360).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1155). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Bonamico, M., Dessy, G., Fares, V. & Scaramuzza, L. (1972). *J. Chem. Soc. Dalton Trans.* pp. 2477–2483.
 Enraf-Nonius (1993). *CAD-4-PC*. Version 1.2. Enraf-Nonius, Delft, The Netherlands.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Acta Cryst. (1995). **C51**, 861–863

Dichloro[(Z)-2-chloro-2-phenylvinyl]-(4-methoxyphenyl)tellurium(IV)

JULIO ZUKERMAN-SCHPECTOR

*Universidade Federal de São Carlos,
 Departamento de Química, Caixa Postal 676,
 13565-905 São Carlos SP, Brazil*

JOÃO VALDIR COMASSETO AND HELIO ANDRÉ STEFANI

*Instituto de Química, Universidade de São Paulo,
 01498 São Paulo SP, Brazil*

(Received 18 January 1994; accepted 21 April 1994)

Abstract

In the title compound, $[\text{TeCl}_2(\text{C}_8\text{H}_6\text{Cl})(\text{C}_7\text{H}_7\text{O})]$, the Te^{IV} atom is in a trigonal bipyramidal configuration with the lone pair of electrons occupying one of the equatorial positions. Distances and angles are: Te—Cl 2.521 (2) and 2.485 (2), Te—C 2.073 (7) and 2.110 (7) Å (aryl); Cl—Te—Cl 117.65 (8), Cl—Te—C 87.1 (2), 90.6 (2), 90.8 (2) and 90.6 (2), C—Te—C 95.0 (3)°.

Comment

Vinyllic tellurides are intermediates in the synthesis of vinyl lithium compounds (Barros, Comasseto & Berriel, 1989), which are, in turn, important intermediates in organic synthesis, either as precursors of the widely used vinyl cuprate compounds or as nucleophiles leading to chain elongation products by reaction with many electrophiles (Comasseto & Berriel, 1990; Lipshutz, 1989). The crystal structure determination of compound (1) was