Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Bis(acetylacetonato)dimethanolzinc(II)

Hans-Wolfram Lerner and Michael Bolte*

Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail:
bolte@chemie.uni-frankfurt.de

Received 6 September 2004
Accepted 9 September 2004
Online 25 September 2004

Key indicators

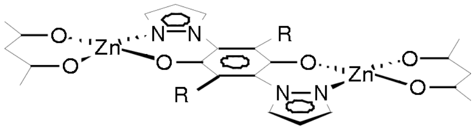
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.025
wR factor = 0.069
Data-to-parameter ratio = 21.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Zn(C_5H_7O_2)_2(CH_4O)_2]$, crystallizes with the octahedrally coordinated Zn atom on a centre of inversion and one bidentate acetylacetonate anion and one methanol molecule in the asymmetric unit.

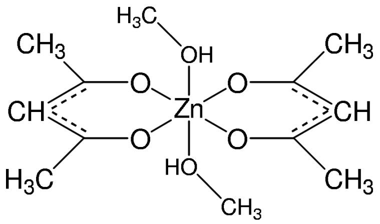
Comment

Transition metal complexes with redox-active ligands are of great current interest. Owing to their electrochemical reversibility, quinone derivatives are candidates as redox-active π-conjugated ligands (Dinnebier *et al.*, 2002). To that end, we are investigating the physical and chemical properties of the dinuclear transition metal complex (1). As part of these studies, we have prepared diacetylacetonatozinc, $Zn(C_5H_7O_2)_2$ and we have reported the X-ray crystal structure analysis of diacetylacetonatozinc, $[Zn(C_5H_7O_2)_2(H_2O)_2]$ (Harbach *et al.*, 2003).



(1)

Now we have prepared the acetylacetonatozinc methanol adduct, $[Zn(C_5H_7O_2)_2(CH_3OH)_2]$, (I). The methanol complex has been obtained from $Zn(C_5H_7O_2)_2$, which has been synthesized by a literature procedure (Rudolph & Henry, 1967) from zinc sulfate and acetylacetone (2,4-pentadione) in the presence of NaOH.



(I)

In (I), the Zn atom is located on a centre of inversion and the asymmetric unit contains one acetylacetonate anion and one methanol molecule. The bond lengths and angles in (I) (Table 1) are normal (Allen, 2002). The acetylacetonate species is almost planar (for the non-H atoms, root-mean-square deviation from the least-squares plane = 0.033 Å). The Zn atom is displaced from this plane by 0.214 (1) Å. The bond

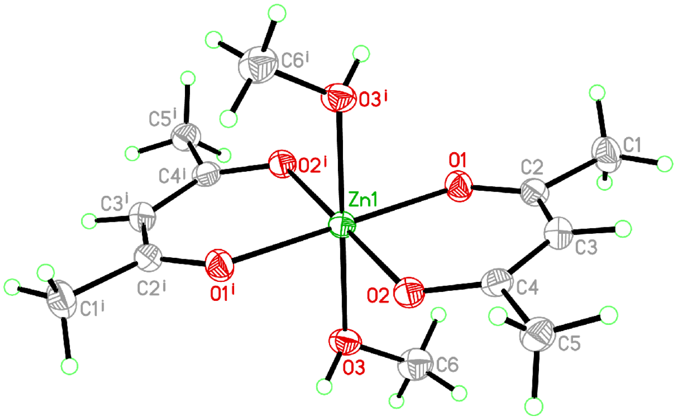


Figure 1
Perspective view of (I), with the atom numbering; displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres. Symmetry code as in Table 1.

from Zn to the methanol O atom is significantly longer than the bonds to the acetylacetonate O atoms. The crystal packing is stabilized by hydrogen bonds from the methanol molecule to an acetylacetonate O atom of an adjacent complex (Table 2).

Experimental

A slurry of $ZnSO_4$ (14.4 g), 2,4-pentadione (10 g), NaOH (4 g), and water (50 ml) was stirred at room temperature for 1 h, resulting in the quantitative precipitation of $[Zn(C_5H_7O_2)_2(H_2O)_2]$, which was filtrated off from the solution. After removing all volatile compounds *in vacuo*, the remaining residue was dissolved in 45 ml of methanol and (I) was crystallized from this solution.

Crystal data

$[Zn(C_5H_7O_2)_2(CH_4O)_2]$
$M_r = 327.67$
Triclinic, $P\bar{1}$
$a = 5.2986$ (7) Å
$b = 7.4809$ (10) Å
$c = 10.4575$ (15) Å
$\alpha = 69.184$ (10)°
$\beta = 81.385$ (11)°
$\gamma = 75.485$ (11)°
$V = 374.25$ (9) Å^3
$Z = 1$
$D_x = 1.454$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 23971 reflections
$\theta = 3.9$–28.6°
$\mu = 1.66$ mm^{-1}
$T = 100$ (2) K
Block, colourless
0.43 × 0.23 × 0.22 mm

Data collection

Stoe IPDSII two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{min} = 0.525$, $T_{max} = 0.698$
8019 measured reflections
2077 independent reflections
2017 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 29.5°$
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.069$
$S = 1.09$
2077 reflections
96 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 0.0996P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.73$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.028 (6)

Table 1
Selected geometric parameters (Å, °).

Zn1—O1	2.0355 (10)	Zn1—O3	2.1621 (10)
Zn1—O2	2.0678 (10)		
O1^i—Zn1—O2	89.96 (4)	O1^i—Zn1—O3	87.83 (4)
O1—Zn1—O2	90.04 (4)	O1—Zn1—O3	92.17 (4)
O2—Zn1—O3^i	90.47 (4)	O2—Zn1—O3	89.53 (4)

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

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{ii}	0.826 (10)	1.918 (10)	2.7401 (14)	173 (2)

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

The H atom bonded to O was located in a difference map and refined isotropically, with the O—H distance restrained to 0.84 (1) Å. H atoms bonded to C were placed in idealized positions [C—H = 0.95 for Csp^2 and 0.98 Å, for C_{methyl}] and refined as riding on their carrier atoms with the constraints $U_{iso}(H) = 1.2 U_{eq}(C)$ or $U_{iso}(H) = 1.5 U_{eq}(C_{methyl})$. In addition, the methyl group was allowed to rotate but not to tip.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Dinnebier, R., Lerner, H.-W., Ding, L., Shankland, K., David, W. I. F., Stephens, P. W. & Wagner, M. (2002). *Z. Anorg. Allg. Chem.* **628**, 310–314.
Harbach, P., Lerner, H.-W. & Bolte, M. (2003). *Acta Cryst.* E**59**, m724–m725.
Rudolph, G., & Henry, M. C. (1967). *Inorg. Synth.* **10**, 74–77.
Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.