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† Dedicated on the occasion of the third anniversary of his death.

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Key indicators

Single-crystal X-ray study
 T = 293 K
 Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
 R factor = 0.041
 wR factor = 0.086
 Data-to-parameter ratio = 12.6

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

Octahedral arrangement of *trans*-bis(diphenylpropanedionato)bis(*o*-phenylenediamino)zinc(II)

The title compound, $[\text{Zn}(\text{C}_{15}\text{H}_{11}\text{O}_2)_2(\text{C}_6\text{H}_8\text{N}_2)_2]$, consists of a square-planar $\text{Zn}^{\text{II}}\text{O}_4$ array, with two molecules of axially coordinated 1,2-phenylenediamine. The Zn atom is located on a centre of symmetry. The monodentate *trans* arrangement of the diamine molecules results in the overall octahedral geometry. The phenyl groups adopt a butterfly-like conformation in relation to the ketoenolate frame. There are intramolecular $\text{N}-\text{H}\cdots\text{O}$ and intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds; the $\text{N}\cdots\text{O}$ and $\text{N}\cdots\text{N}$ distances are 3.130 (4) and 3.323 (5) Å, respectively.

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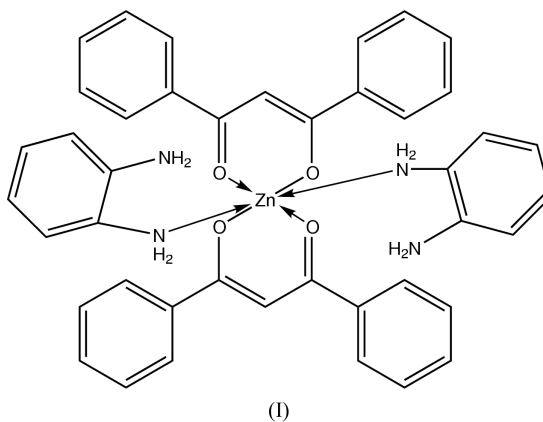
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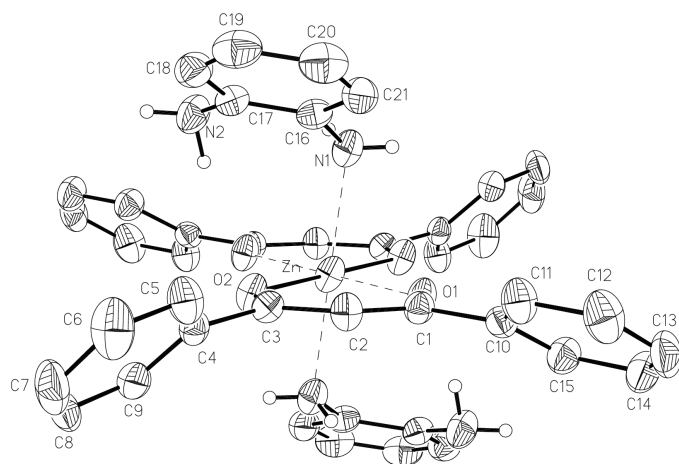
Comment

Dibenzoylmethane and its analogues have been reported as forming strong intra- and intermolecular hydrogen bonds (Bertolasi *et al.*, 1991; Kaitner & Mestrovic, 1993); while dibenzoylmethane is almost planar, this is not the case for the substituted analogues (Bertolasi *et al.*, 1991). Several transition-metal complexes have been synthesized using the dibenzoylmethane salt (DPhPO₂, 1,3-diphenyl-1,3-propanedionate-*O,O*), e.g. bis(diethyl ether)bis(DPhPO₂)Fe^{II} (Muller *et al.*, 1997), (II), bis(DPhPO₂)(1,2-ethanediamine)cobalt(II) (Levitin *et al.*, 1998), (III), and tris(DPhPO₂)cobalt(III) (Kaitner & Mestrovic, 1994), (IV), in which the planarity in the ketoenolate moiety is retained. Analogous 1,3-diketonate-metal complexes with 1,2-diaminobenzene as neutral ligands have been already described (Dickman, 2000), and the isostructural nickel(II) compound, (V), have already been described (Hotz *et al.*, 1987). In order to explore the structural changes in the coordination complex of zinc with 1,3-diketonates, the X-ray analysis of the title compound, (I), was undertaken.



(I)

In (I), the Zn atom is located on a centre of symmetry. The bidentate DPhPO₂ moieties are in a square-planar arrange-


Figure 1

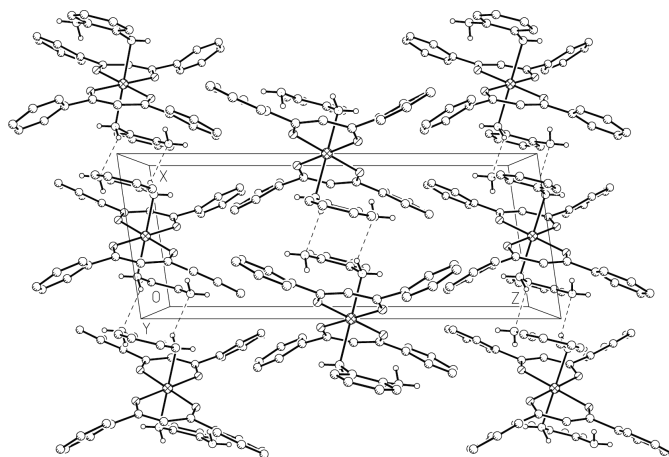
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are shown at the 40% probability level.

ment, and two 1,2-phenyldiamine moieties are in *trans*-axial coordination sites (Fig. 1). The structure contains several planar groups: the zinc–ketoenolate moiety *A* [(O1–C1–C2–C3–O2)Zn], with a mean deviation of 0.0407 Å and a maximum deviation of 0.0585 (18) Å; two aryl rings *B* (C4–C9, mean deviation 0.0087 Å) and *C* (C10–C15, 0.0065 Å), and the 1,2-diaminobenzene moiety *D* (N1/N2/C16–C21, 0.0355 Å). The aryl rings (*B* and *C*) are rotated around the C3–C4 and C1–C10 bonds, respectively; the dihedral angles are 46.21 (7), 31.52 (11) and 53.56 (9)° for the *A/B*, *A/C* and *B/C* planes, respectively; the dibenzoylmethane adopts a butterfly-like conformation. The values of these dihedral angles are similar to those in (V) (43.8, 31.6 and 52.9°; Hotz *et al.*, 1987) and larger than in (II) (2.6, 11.9 and 14.5°; Muller *et al.*, 1997). Moiety *D* displays dihedral angles of 26.47 (8), 44.20 (7) and 9.6 (2)° with *A*, *B* and *C*, respectively. Similar values were observed for compound (V) (28.2, 42.5 and 10.5°).

The O–C and C–C bond distances of the ketoenolate moiety of the DPhPO2 ligand (Table 1) indicate delocalization of the double bonds, as observed in other metal complexes. There is an intramolecular hydrogen bond, N2–H2A···O1ⁱⁱ (Table 2). The Zn complexes are stacked along the *a* axis, and are linked by an N1–H1A···N2ⁱ intermolecular hydrogen bond, forming a network which extends through the *ac* plane [symmetry code: (i) 2–*x*, 1–*y*, 2–*z*]. In addition, an intermolecular close contact is observed for C8–H8···C10 [C···C 3.55 (2) Å, H···C 2.78 Å and C–H···C 140.9°].

Experimental

The title compound, (I), was prepared by refluxing stoichiometric amounts of 1,2-diaminobenzene, dibenzylmethane and zinc(II) acetate tetrahydrate in toluene with a Dean–Stark trap for 2 h. After work-up and purification, light-yellow crystals were obtained and used for X-ray analysis. IR spectral data for (I) (cm^{−1}), KBr: 3413, 3356, 3300, 3232, 1592, 1549, 1520, 1412. Elemental analysis: C 68%, H 5.45%, N 7.87%, Zn 9.16%.


Figure 2

The crystal packing of (I). Broken lines indicate hydrogen bonds.

Crystal data

[Zn(C₁₅H₁₁O₂)₂(C₆H₈N₂)₂]
 $M_r = 728.13$
 Monoclinic, $P2_1/n$
 $a = 7.553$ (1) Å
 $b = 12.328$ (2) Å
 $c = 19.038$ (2) Å
 $\beta = 98.317$ (8)°
 $V = 1754.0$ (4) Å³
 $Z = 2$

$D_x = 1.379$ Mg m^{−3}
 Mo $K\alpha$ radiation
 Cell parameters from 49 reflections
 $\theta = 2.0$ – 12.1°
 $\mu = 0.75$ mm^{−1}
 $T = 293$ (2) K
 Prism, yellow
 0.40 × 0.16 × 0.10 mm

Data collection

Siemens P4/PC diffractometer
 ω scans
 Absorption correction: none
 3334 measured reflections
 3085 independent reflections
 1902 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.032$

$\theta_{max} = 25.0^\circ$
 $h = 0 \rightarrow 8$
 $k = 0 \rightarrow 14$
 $l = -22 \rightarrow 22$
 3 standard reflections every 97 reflections
 intensity decay: <2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.086$
 $S = 0.93$
 3085 reflections
 245 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.26$ e Å^{−3}
 $\Delta\rho_{min} = -0.34$ e Å^{−3}
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0108 (6)

Table 1

Selected geometric parameters (Å, °).

Zn–O2	2.041 (2)	N1–C16	1.434 (5)
Zn–O1	2.063 (2)	N2–C17	1.415 (5)
Zn–N1	2.285 (4)	C1–C2	1.401 (4)
O1–C1	1.271 (4)	C2–C3	1.391 (4)
O2–C3	1.270 (4)		
O2–Zn–O1	90.38 (9)	C16–N1–Zn	117.5 (2)
O2–Zn–N1	94.70 (10)	O1–C1–C2	125.4 (3)
O1–Zn–N1	87.21 (11)	C3–C2–C1	126.6 (3)
C1–O1–Zn	125.2 (2)	O2–C3–C2	126.7 (3)
C3–O2–Zn	125.0 (2)		

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1A \cdots N2 ⁱ	0.86 (4)	2.61 (4)	3.323 (5)	142 (3)
N1—H1B \cdots O1	0.78 (4)	2.77 (4)	3.003 (4)	99 (3)
N2—H2A \cdots O1 ⁱⁱ	0.94 (4)	2.21 (4)	3.130 (4)	166 (3)

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

H2B, attached to N2, was not found to be involved in hydrogen bonding. The positional parameters of the amino H atoms were refined, and those of the other H atoms were positioned geometrically. All H atoms were assigned $U_{iso} = 1.2U_{eq}$ of the parent atom.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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