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Key indicators

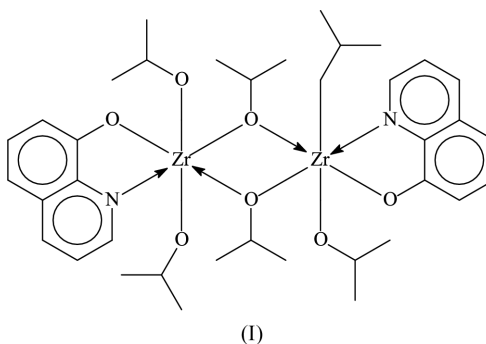
Single-crystal X-ray study
 $T = 163$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.032
 wR factor = 0.082
Data-to-parameter ratio = 19.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Di- μ -isopropoxy-bis[(8-hydroxyquinolinato)-
diisopropoxyzirconium(IV)]Each Zr atom in the dimeric title compound, $[\text{Zr}_2(\text{C}_9\text{H}_6\text{NO})_2(\text{C}_3\text{H}_7\text{O})_6]$, is chelated by the oxinate group; the two Zr atoms are linked through two isopropoxy bridges. The molecule lies on an inversion center and the Zr atoms exist in octahedral environments.

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Comment

The reaction of titanium tetraalkoxides with 8-hydroxyquinoline affords $(\text{C}_9\text{H}_6\text{NO})_n(\text{OR})_{4-n}\text{Ti}$, with $n = 1$ for the ethoxide (Amini *et al.*, 2004) and $n = 2$ for the isopropoxide (Zeng *et al.*, 2002). As the ethoxide exists as a dimeric compound whereas the isopropoxide is only monomeric, steric factors probably account for the difference in the manner in which the oxinate group replaces the alkoxide group. On the basis of steric factors, the analogous reaction with zirconium tetraisopropoxide should yield $(\text{C}_9\text{H}_6\text{NO})_2(\text{OR})_2\text{Zr}$, but the reaction yielded instead dimeric $[(\text{C}_9\text{H}_6\text{NO})(\text{OC}_3\text{H}_7)_3\text{Zr}]_2$, (I) (Fig. 1). Evidently, the molecule is able to accommodate the isopropyl unit at the bridging atom [$\text{Zr}-\text{O} = 2.154$ (2) and 2.213 (2) Å] owing to the longer Zr–O bonds, as the Zr atom is a much larger atom than a Ti atom. The molecule lies on an inversion center and the Zr atoms exist in octahedral environments.

Experimental

Manipulations were performed under nitrogen using standard Schlenk techniques. Zirconium(IV) tetraisopropoxide was prepared from zirconium tetrachloride (Fluka) according to a reported procedure (Bradley & Wardlaw, 1950) and purified by vacuum distillation. 8-Hydroxyquinoline (0.61 g, 4.26 mmol) was reacted with this reagent (1.4 g, 4.26 mmol) in toluene (12 ml). The mixture was stirred for a day and the solvent then removed under reduced pressure to furnish a yellow solid. The solid was crystallized from dichloromethane–hexane (5:1) to give orange crystals (m.p. 456–457 K). UV in CH_2Cl_2 : 254 ($\pi \rightarrow \pi^*$), 375 ($n \rightarrow \pi^*$) nm. IR: 1572 (C=C, C=N), 1272 (C–O) cm^{-1} . ^1H NMR in CDCl_3 : 1.2 (CH_3 , doublet), 4.0 (CH, multiplet), 6.5–8.5 (aromatic H atoms) p.p.m. MS: 678 $[(M - \text{C}_9\text{H}_6\text{NO})]^+$.

Crystal data

[Zr₂(C₉H₆NO)₂(C₃H₇O)₆]
M_r = 825.25
 Monoclinic, *C2/c*
a = 19.851 (1) Å
b = 12.890 (1) Å
c = 16.301 (1) Å
 β = 105.518 (1)°
V = 4018.8 (4) Å³
Z = 4

D_x = 1.364 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 6641 reflections
 θ = 2.6–26.4°
 μ = 0.57 mm⁻¹
T = 163 (2) K
 Block, orange
 0.50 × 0.25 × 0.10 mm

Data collection

Siemens *P4*/CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.725, *T_{max}* = 0.946
 24817 measured reflections

4116 independent reflections
 3355 reflections with *I* > 2σ(*I*)
R_{int} = 0.042
 θ_{max} = 26.5°
h = -24 → 24
k = -16 → 8
l = -20 → 20

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.032
wR(*F*²) = 0.082
S = 1.02
 4116 reflections
 217 parameters
 H-atom parameters constrained

w = 1/[σ²(*F_c*²) + (0.0425*P*)² + 4.6465*P*]
 where *P* = (*F_c*² + 2*F_c*)/3
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.59 e Å⁻³
 Δρ_{min} = -0.50 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Zr1—O1	2.087 (2)	Zr1—O3	1.938 (2)
Zr1—O2	2.213 (2)	Zr1—O4	1.935 (2)
Zr1—O2 ⁱ	2.154 (2)	Zr1—N1	2.440 (2)
O1—Zr1—O2	91.25 (7)	O2—Zr1—N1	85.98 (6)
O1—Zr1—O2 ⁱ	150.82 (6)	O2 ⁱ —Zr1—O3	105.97 (7)
O1—Zr1—O3	98.70 (7)	O2 ⁱ —Zr1—O4	93.20 (7)
O1—Zr1—O4	98.18 (8)	O2 ⁱ —Zr1—N1	84.02 (6)
O1—Zr1—N1	70.84 (6)	O3—Zr1—O4	98.96 (8)
O2—Zr1—O2 ⁱ	71.92 (6)	O3—Zr1—N1	169.54 (7)
O2—Zr1—O3	94.10 (7)	O4—Zr1—N1	83.28 (7)
O2—Zr1—O4	162.50 (7)	Zr1—O2—Zr1 ⁱ	108.08 (6)

Symmetry code: (i) $\frac{3}{2} - x, \frac{1}{2} - y, 1 - z$.

H atoms were placed in calculated positions [*C*—*H* = 0.95 Å for the aromatic H atoms and *U*_{iso}(H) = 1.2*U*_{eq}(C); 1.00 Å for the methine H atoms and *U*_{iso}(H) = 1.2*U*_{eq}(C); 0.98 Å for the methyl H atoms and *U*_{iso}(H) = 1.5*U*_{eq}(C)] and were included in the refinement in the riding-model approximation.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine

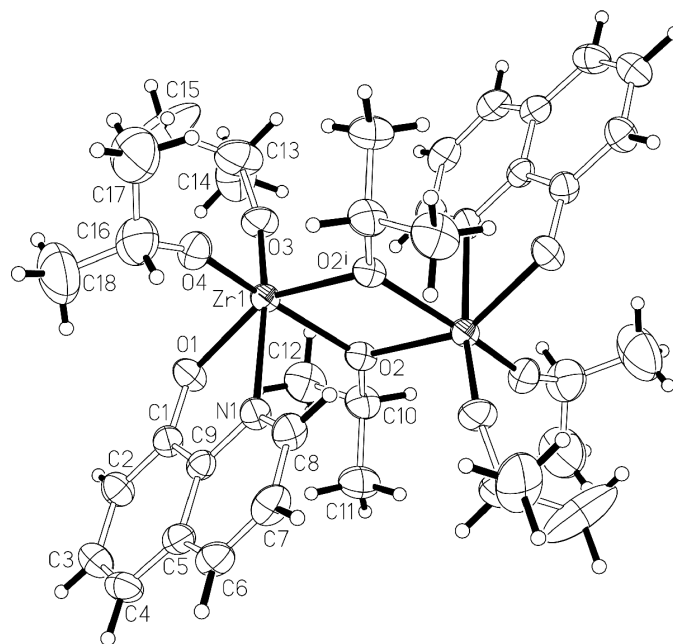


Figure 1

ORTEP (Johnson, 1976) plot of (I); displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. Symmetry code (i) as in Table 1.

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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