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cis-Bis(3,5-di-*tert*-butyl-2-hydroxybenzoato-κ^2O,O')bis(ethanol)zinc(II)

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The title compound, $[Zn(C_{15}H_{21}O_3)_2(C_2H_6O)_2]$, is a powerful charge-control agent used widely in electrophotography. The present Zn^{II} complex has C_2 symmetry and exhibits a deformed octahedral coordination. Two O atoms of the ethanol molecules are coordinated in a *cis* fashion to the Zn atom.

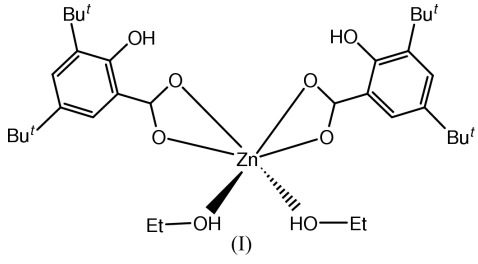
Key indicators

Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.004 Å
H-atom completeness 97%
Disorder in main residue
R factor = 0.042
wR factor = 0.126
Data-to-parameter ratio = 15.8

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

Comment

The title compound, (I), is a powerful charge-control agent used widely in electrophotography (Suganami *et al.*, 2002). Compound (I) is a 1:2 complex of the zinc salt of 3,5-bis(1,1-dimethylethyl)-2-hydroxybenzoic acid (TBS) with ethanol. The structure of TBS has recently been published (Mizuguchi, 2003). TBS itself exhibits an excellent charge-control effect. The effect is, however, greatly enhanced by the formation of metal complexes with a variety of metals such as Zn, Cr, Al, Fe *etc*. The present structure analysis has, therefore, been carried out in order to elucidate the charge-control mechanism on the basis of structural information.



(I)

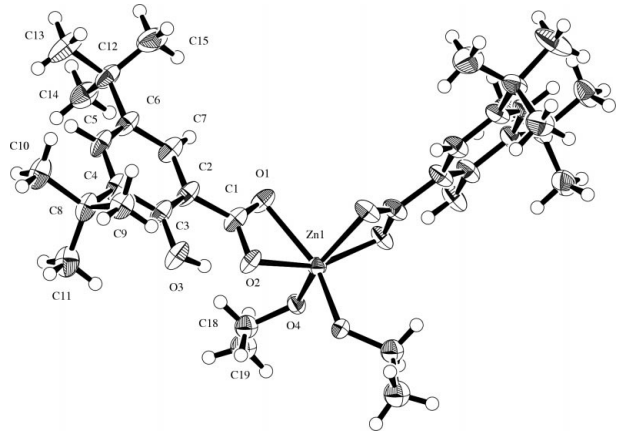


Figure 1
A view of the molecular structure of (I), showing 50% probability displacement ellipsoids. The occupation factors of the ethanol atoms C18 and C19 are 50%, and the other possible orientations of the ethanol molecules have been omitted. The H atom bonded to atom O4 was not located.

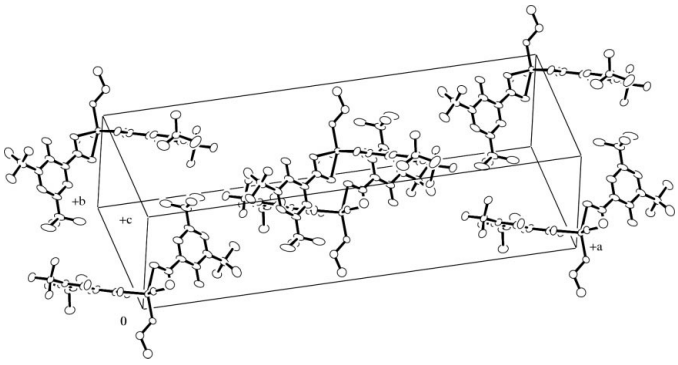


Figure 2
The crystal structure of (I). One of two possible orientations of the disordered ethanol molecules has been omitted for clarity.

Fig. 1 shows a molecular view of complex (I), which has C_2 symmetry. Two O atoms of the ethanol molecules are coordinated in a *cis* fashion to the Zn atom to form a deformed octahedral complex (Table 1). The chelate rings formed by the TBS ligands are almost perpendicular to each other. There is an intramolecular hydrogen bond between the OH group and the carbonyl O atom in the TBS ligand (Table 2). The molecules pack as shown in Fig. 2. The short O1···O4 ($1-x$, $2-y$, $1-z$) distance of 2.625 (3) Å suggests an intermolecular hydrogen bond.

Experimental

The zinc salt of TBS was obtained from the API Corporation. Single crystals of (I) were grown from an ethanol solution. Since the solvent molecules are rapidly lost at room temperature, data collection was carried out at 93 K.

Crystal data

$[Zn(C_{15}H_{21}O_3)_2(C_2H_6O)_2]$
M_r = 656.18
Monoclinic, $C2/c$
a = 33.891 (4) Å
b = 11.191 (1) Å
c = 9.491 (1) Å
β = 93.75 (1)°
V = 3592.1 (7) Å^3
Z = 4
D_x = 1.213 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 15 255 reflections
θ = 4.2–68.1°
μ = 1.32 mm^{-1}
T = 93.2 K
Needle, colourless
0.45 × 0.10 × 0.10 mm

Data collection

Rigaku *R-AXIS RAPID* imaging-plate diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.548, T_{max} = 0.877
16 078 measured reflections
3054 independent reflections
2621 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.040
θ_{max} = 68.2°
$h = -40 \rightarrow 40$
$k = -13 \rightarrow 13$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.126
S = 1.65
3054 reflections
193 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + \{0.05[\max(F_o^2,0) + 2F_c^2]/3\}^2]$
$(\Delta/\sigma)_{max}$ = 0.009
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O1	2.237 (2)	O3—C3	1.348 (4)
Zn1—O2	2.070 (2)	O4—C16	1.469 (6)
Zn1—O4	2.034 (2)	O4—C18	1.458 (6)
O1—C1	1.264 (3)	C16—C17	1.503 (8)
O2—C1	1.280 (3)	C18—C19	1.507 (9)
O1—Zn1—O1^{i}	87.9 (1)	O4—Zn1—O4^{i}	97.7 (1)
O1—Zn1—O2	60.86 (7)	Zn1—O1—C1	86.7 (2)
O1—Zn1—O2^{i}	100.27 (8)	Zn1—O2—C1	93.9 (2)
O1—Zn1—O4	94.01 (7)	Zn1—O4—C16	119.2 (3)
O1—Zn1—O4^{i}	151.28 (7)	Zn1—O4—C18	126.7 (3)
O2—Zn1—O2^{i}	155.2 (1)	O4—C16—C17	110.0 (5)
O2—Zn1—O4	105.68 (7)	O4—C18—C19	111.9 (5)
O2—Zn1—O4^{i}	90.67 (7)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1···O2	0.91	1.72	2.573 (3)	156

The ethanol molecule was disordered; the minimum R value was obtained with an occupancy of 0.50 (1) for atoms C16, C17, C18 and C19. The well modelled pairs are O4/C16/C17 and O4/C18/C19. The H atom of the hydroxyl group of the TBS ligand was found in a difference density map but not refined. The hydroxyl H atom of the ethanol molecule could not be located in the difference density map and therefore this H atom remained undetermined. All other H atoms were positioned by calculation and allowed for as riding.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *TEXSAN*.

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