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Tris[2-(2-oxidophenyl)-5-phenyl-1,3,4-oxadiazole-κ^2O^2,N^3]aluminium 0.167-hydrate

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Key indicators

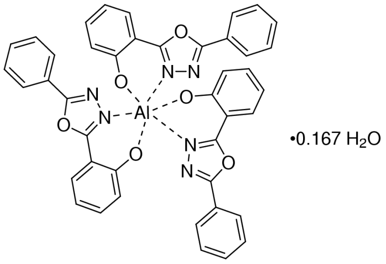
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.010 Å
Disorder in main residue
R factor = 0.074
wR factor = 0.235
Data-to-parameter ratio = 10.2

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

The title compound, $[\mathrm{Al(C_{14}H_9N_2O_2)_3}]\cdot0.167\mathrm{H_2O}$, crystallizes with three crystallographically independent molecules in the asymmetric unit. The Al atom is coordinated by one O atom and one N atom from each ligand. One half molecule of water is incorporated into the structure per three $\mathrm{Al(C_{14}H_9N_2O_2)_3}$ units.

Comment

Oxadiazole derivatives are among the most widely employed electron-conducting and hole-blocking materials in organic light-emitting devices (OLEDs) (Adachi *et al.*, 1989; Wang *et al.*, 1999; Tanaka, Tokito *et al.*, 1998; Tokito *et al.*, 2000; Tokito & Taga, 2000; Tanaka, Okada *et al.*, 1998; Jabbour *et al.*, 2002; Wang *et al.*, 2001). We (Wang *et al.*, 1999) and others (Tanaka, Tokito *et al.*, 1998) prepared the title oxadiazole metal complex, (I), which proved to be a good electron transporter with excellent shelf-life stability when incorporated into OLEDs (Wang *et al.*, 1999; Tanaka, Tokito *et al.*, 1998; Tokito *et al.*, 2000; Tokito & Taga, 2000).



(I)

The asymmetric unit of (I) contains three crystallographically independent $\mathrm{Al(C_{14}H_9N_2O_2)_3}$ complex molecules and one half-occupancy water molecule. These molecules are related by a translation of approximately one-third along the c axis of the unit cell and, indeed, initial indexing suggested a c unit-cell length of 12.192 Å; however, examination of the diffraction pattern confirms that the larger unit cell is correct. The three molecules (Fig. 1) have similar conformations with small but significant differences in the relative orientations of the oxadiazole ligands: the angle between the mean planes of these ligands (taken through atoms O1 to C10 for each ligand) ranges from 65.54 (9) (ligands D and E in molecule 2) to 88.33 (10)° (ligands A and C, molecule 1). The torsion angle (measured through atoms O9, C10, C13 and C18 for each ligand) of the 5-phenyl group varies from 7.7 (19) (ligand B) to 27.5 (9)° (ligand H).

The water molecule is located within hydrogen-bonding distance of only one of the three molecules. There are two

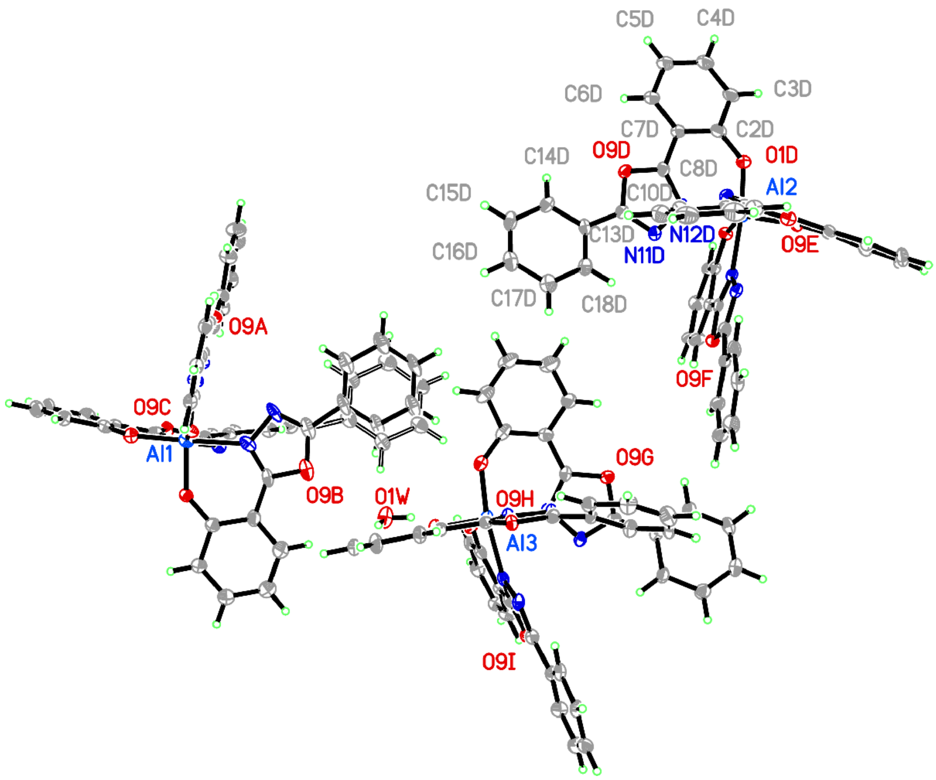


Figure 1

The asymmetric unit of (I), with displacement ellipsoids drawn at the 30% probability level. The full labelling scheme is shown for one ligand; the remaining ligands are numbered in a similar fashion with only one O atom per ligand labelled in the figure. The atoms in each ligand are distinguished by the final letter in the label. One ligand (*B*, molecule 1) has one disordered phenyl ring.

possible hydrogen-bonding contacts to phenolate O atoms in two separate ligands (Table 1). The formation of hydrogen bonds does not appear to significantly alter the geometry of the complex.

The crystal structure confirms our earlier supposition regarding the bonding in (I), which was based on comparisons of IR spectra of the complex and free ligand (Wang *et al.*, 1999) and is consistent with other published crystal structures involving this ligand (Hu *et al.*, 1999).

Experimental

Compound (I) was prepared according to the method of Wang *et al.* (1999) and purified by sublimation under high vacuum (133 322 × 10^{-3} Pa). Most of the compound deposited as a solvent-free amorphous film in a zone close to the heat source. However, a few crystals grew in a lower temperature zone located further away from the heat source. One of these crystals was selected for X-ray analysis. The crystals were found to be stable to solvent loss over a period of six years.

Crystal data

$[Al(C_{14}H_9N_2O_2)_3]\cdot 0.167H_2O$
$M_r = 741.68$
Monoclinic, $P2_1/n$
$a = 15.3472\ (11)$ Å
$b = 19.6520\ (14)$ Å
$c = 36.444\ (3)$ Å
$\beta = 91.8281\ (17)^\circ$
$V = 10986.0\ (13)$ Å^3
$Z = 12$
$D_x = 1.345$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1889 reflections
$\theta = 2.2$–17.7°
$\mu = 0.11$ mm^{-1}
$T = 100\ (2)$ K
Block, colourless
0.21 × 0.16 × 0.16 mm

Data collection

Bruker SMART1000 CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{min} = 0.814$, $T_{max} = 0.980$
93 111 measured reflections
15 811 independent reflections
6575 reflections with $I > 2\sigma(I)$
$R_{int} = 0.258$
$\theta_{max} = 23.3^\circ$
$h = -17 \rightarrow 17$
$k = -21 \rightarrow 21$
$l = -40 \rightarrow 40$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.074$
$wR(F^2) = 0.235$
$S = 1.01$
15 811 reflections
1556 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0935P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}
Extinction correction: *SHELXH*97
Extinction coefficient: 0.00096 (11)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1*W*—H1*A*$\cdots$O1H	0.8400 (11)	2.49 (9)	2.946 (10)	115 (7)
O1*W*—H1*B*$\cdots$O1I	0.8400 (12)	2.033 (12)	2.866 (10)	171 (7)

Aromatic H atoms were positioned geometrically (C—H = 0.95 Å) and refined using a riding model with U_{iso} values set at $1.2U_{eq}$(C). Water H atoms could not be located in a difference map and were placed in a position consistent with the shorter of the hydrogen-bonding contacts. The positions of these H atoms were refined; hard

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restraints were used to avoid clashes with other atoms. The O—H distances were restrained to 0.840 (1) Å and U_{iso}(H) values were set at 1.2U_{eq}(O). No attempt was made to model the likely disorder of these H atoms over the two hydrogen-bonding contacts. The occupancy of the water molecule [0.483 (7)] was refined (using a fixed isotropic displacement parameter for the O atom) and converged to 50% . The occupancy was then fixed and anisotropic displacement parameters used for oxygen.

The crystal diffracted rather poorly, especially at higher angle. For this reason, a resolution cut-off of 0.9 Å was applied during integration; the poor diffraction is also reflected in the poor merging statistics. The very high value of R_{int} could indicate non-merohedral twinning; this was excluded using *ROTAX* (Cooper *et al.*, 2002). The poor R_{int} is a symptom of the weak diffraction; deletion of reflections with $F^2/\sigma(F^2) < 1$ from the data set gave an R_{int} value of 0.11.

One phenyl group in molecule 1 is disordered over two positions. The relative occupancies were refined and converged to 0.586 (18):0.414 (18); these occupancies were fixed in the final refinement. An apparent short contact [2.76 (2) Å] is formed between the water molecule and a C atom in the minor component of this disordered group (C25*B*). This could be evidence of a weak hydrogen-bonding interaction (if the groups are occupied at the same time) or the two sites may have mutually exclusive occupancies.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXM* (Schneider & Sheldrick, 2002); program(s) used to refine structure: *SHELXH*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXH*97.

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