metal-organic papers

Tris(quinolin-8-olato)indium(III)

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.010 Å
R factor = 0.050
wR factor = 0.150
Data-to-parameter ratio = 15.1

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

The single-crystal structure of the title compound, [In(C$_9$H$_6$NO)$_3$], is described. Quinolinolates of the elements of the group IIIB (denoted by MQ_3), Al, Ga, and In, have been of continuous interest to organometallic and physical chemists, in particular, for the last 50 years. Organic light-emitting diodes (OLEDs) utilizing GaQ_3 and InQ_3, the gallium and indium analogs, respectively, of the most widely used OLED material AlQ_3, were first explored in the early 1980s and continue to be the subject of current research. To the best of our knowledge, the structure reported here is the first-ever facial MQ_3-type structure, confirmed by single-crystal X-ray crystallography.

Comment

In 1987, efficient electroluminescence from an organic light-emitting diode (OLED) device using low molecular weight organic materials was first reported (Tang & Van Slyke, 1987). This device was constructed of two active layers and used the metal–quinolinolate tris(8-hydroxyquinoline)aluminium. This discovery generated renewed interest in metal quinolinolates. The tris-chelate 8-hydroxyquinoline metal complexes, MQ_3, can occur in two different geometrical forms, *viz.* facial or meridional. To date, all published AlQ_3 single-crystal structures (including different polymorphs and solvates) and GaQ_3 structures were found to be meridional isomers (Brinkmann *et al.*, 2000; Ul-Haque *et al.*, 1991; Fujii *et al.*, 1996; Wang *et al.*, 1999). Single-crystal structures of facial isomers of MQ_3-type complexes have been elusive so far. We report here the crystal structure of the title compound, InQ_3, (I).

(I)
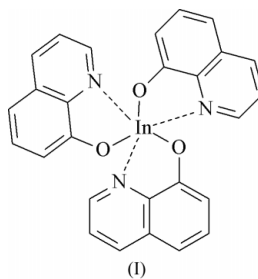
The molecular structure of InQ_3 (Fig. 1) contains a six-coordinated indium atom. The angles around the In atom indicate approximate octahedral geometry. The average In—O and In—N distances are 2.108 and 2.261 Å, respectively. There is intermolecular π–π stacking of the ligands (8-hydroxyquinolines) in a multidirectional fashion. Such intermolecular stacking was also observed in the GaQ_3 structure (Wang *et al.*, 1999). The ligands in InQ_3 are stacked in the unit cell, with interplanar distances in the range 3.406–3.428 Å, comparable to 3.35–3.41 Å for GaQ_3 (Fig. 2).
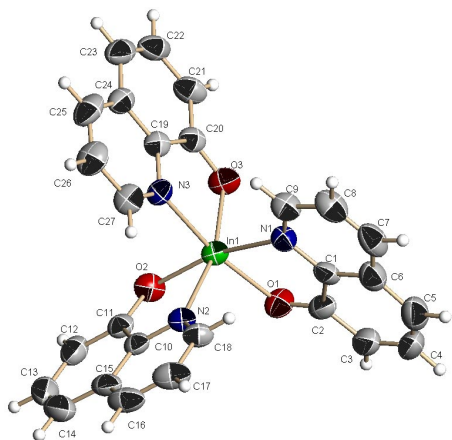
Figure 1
A view of the molecule of the title compound, (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.
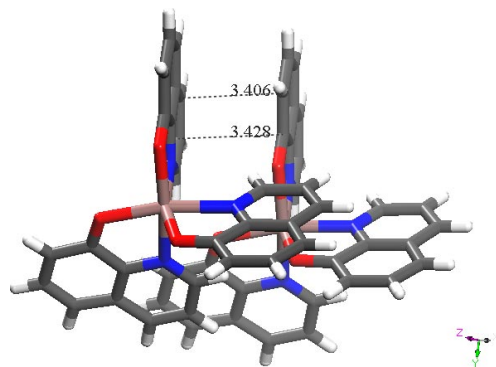

Figure 2
Part of the crystal structure of InQ_3, illustrating the stacking of 8-hydroxyquinoline ligands. Stacking distances between various planes are indicated.

Experimental

InQ_3 was synthesized according to established methods (Lytle *et al.*, 1973). InQ_3 was chemically purified by washing and recrystallization and subjected to vacuum temperature-gradient sublimation three consecutive times until a purity of 99.9% was achieved. The purity was determined by NMR in d_6-DMSO. We note that, although the remaining impurities could not be positively characterized, they could consist of different forms, *e.g.* isomers or polymorphs, of the target compound. InQ_3 was sublimed at 0.6 Torr and the temperature was gradually increased from 533 to 573 K over a period of 1–3 d. It was also characterized by HPLC, LC–MS, and ESIMS, showing results consistent with the structure and purity determined by NMR.

Crystal data

[In(C$_9$H$_6$NO)$_3$]	$Z = 2$
$M_r = 547.26$	$D_x = 1.641$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 6.1860\,(12)$ Å	Cell parameters from 10 088
$b = 13.436\,(3)$ Å	reflections
$c = 14.725\,(3)$ Å	$\theta = 1.0$–$26.7°$
$\alpha = 65.63\,(3)°$	$\mu = 1.10$ mm^{-1}
$\beta = 88.15\,(3)°$	$T = 293\,(2)$ K
$\gamma = 83.55\,(3)°$	Needle, yellow
$V = 1107.7\,(4)$ Å^3	$0.60 \times 0.07 \times 0.06$ mm

Data collection

Nonius KappaCCD diffractometer	2896 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.112$
Absorption correction: multi-scan	$\theta_{max} = 26.8°$
(*SORTAV*; Blessing, 1995)	$h = -7 \rightarrow 7$
$T_{min} = 0.517$, $T_{max} = 0.991$	$k = -16 \rightarrow 15$
11 360 measured reflections	$l = -18 \rightarrow 14$
4647 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.1127P)^2]$
$wR(F^2) = 0.150$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.74$	$(\Delta/\sigma)_{max} < 0.001$
4647 reflections	$\Delta\rho_{max} = 0.59$ e Å^{-3}
307 parameters	$\Delta\rho_{min} = -1.05$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

In1—O3	2.101 (4)	In1—N3	2.250 (5)
In1—O2	2.110 (4)	In1—N1	2.264 (5)
In1—O1	2.112 (4)	In1—N2	2.269 (4)
O3—In1—O2	96.96 (17)	O1—In1—N1	76.89 (17)
O3—In1—O1	101.69 (17)	N3—In1—N1	92.01 (17)
O2—In1—O1	98.33 (17)	O3—In1—N2	163.81 (17)
O3—In1—N3	76.88 (16)	O2—In1—N2	76.89 (16)
O2—In1—N3	93.12 (18)	O1—In1—N2	94.06 (17)
O1—In1—N3	168.54 (17)	N3—In1—N2	88.41 (17)
O3—In1—N1	98.64 (17)	N1—In1—N2	88.45 (17)
O2—In1—N1	164.31 (16)		

The quality of the InQ_3 crystals was not very good, as indicated by a rather high R_{int} value (0.112). There was a minor twin component in the crystals, which was left untreated. During subsequent refinement, positional parameters of H atoms were calculated geometrically and allowed to ride on the C atoms to which they were bonded. In the final difference Fourier map, deepest hole is 0.93 Å from In1.Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001) and *Materials Studio* (Accelrys, 2001); software used to prepare material for publication: *SHELXTL*.

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