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

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The title compound (GaQ_3), [Ga(C_9H_6NO)$_3$], is the Ga analog of the most widely used organic light-emitting diode (OLED) material, AlQ_3. Its molecular structure is a six-coordinated gallium compound, with the angles around the Ga^{3+} ion indicating approximate octahedral geometry. There is intermolecular π–π stacking of the ligands (8-hydroxyquinolines) in a multidirectional fashion.

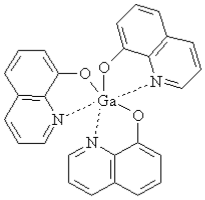
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.050
wR factor = 0.117
Data-to-parameter ratio = 14.7

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

Comment

Quinolinolates of the elements of Group III*B* (denoted 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 MQ_3 were first explored in the early 1980s (Tang & Van Slyke, 1985) and continue to be the subject of current research. In 1987, efficient electroluminescence from an OLED device using low molecular-weight organic materials was first reported (Tang & Van Slyke, 1987). The OLED device was constructed of two active layers and used a metal quinolinolate, tris(quinolin-8-olato)aluminum. This discovery generated renewed interest in metal quinolinolates. GaQ_3 and InQ_3 are the Ga and In analogs, respectively, of the most widely used OLED material, AlQ_3. In our continuing effort to understand how molecular configuration, packing, and polymorphism affect charge transport, electroluminescence evolution, operational stability, and device performance parameters for this series of metal quinolinolates, we report here the single-crystal structure of gallium tris(quinolin-8-olate), GaQ_3, (I).



(I)

Tris-chelate quinolin-8-olate metal complexes can occur in two different geometrical forms, facial or meridional. The crystal structure of (I) is a meridional form of GaQ_3. The molecular structure of GaQ_3 (Fig. 1) is a six-coordinated gallium compound. The angles around the Ga^{3+} ion indicate approximate octahedral geometry. The average Ga—O and Ga—N distances are 1.931 (3) and 2.091 (3) Å, respectively. These are comparable with those obtained for the solvated GaQ_3 structure (Wang *et al.*, 1999).

There is intermolecular π–π stacking of the ligands (8-hydroxyquinolines) in a multidirectional fashion. Such intermolecular stacking was also observed in the ethyl ether solvated GaQ_3 structure (Wang *et al.*, 1999; Brinkmann *et al.*, 2000). The ligands in GaQ_3 are stackedwith an interplanar distance of 3.385–3.460 Å, which is comparable with the values of 3.35–3.41 Å for ether-solvated GaQ_3 and 3.406–3.428 Å for the InQ_3 structure (Rajeswaran & Jarikov, 2003).

Experimental

GaQ_3, (I), was prepared according to established methods (Lytle *et al.*, 1973). The compound was chemically purified by repeated washings and recrystallizations and subjected to vacuum temperature-gradient sublimation, three consecutive times, until a purity of 99.9% was achieved. The purity of GaQ_3 was determined by NMR in d_7-dimethylformamide solutions because using CD_2Cl_2 and d_6-dimethylsulfoxide resulted in poor peak resolution, while the use of $CDCl_3$ produced decomposition (two types of new quinolinol structures were formed). GaQ_3 was sublimed at 0.6 Torr (1 Torr = 133.322 Pa) and the temperature was gradually increased from 533 to 573 K over a period of 1–3 d. HPLC, ESI LC–MS, and MS show results consistent with the structure. We note that, although the remaining 0.1% impurities could not be positively characterized, they may be different forms, *e.g.*, isomers, of the target compound.

Crystal data

$[Ga(C_9H_6NO)_3]$
$M_r = 502.16$
Triclinic, $P\bar{1}$
$a = 8.4250$ (3) Å
$b = 10.2900$ (3) Å
$c = 13.1390$ (5) Å
$\alpha = 71.4320$ (12)°
$\beta = 82.6670$ (12)°
$\gamma = 89.7690$ (14)°
$V = 1070.13$ (6) Å^3

$Z = 2$
$D_x = 1.558$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6837 reflections
$\theta = 1.0$–26.7°
$\mu = 1.32$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.32 × 0.25 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.557$, $T_{max} = 0.768$
16 177 measured reflections

4527 independent reflections
2412 reflections with $I > 2\sigma(I)$
$R_{int} = 0.144$
$\theta_{max} = 26.8°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.117$
$S = 0.89$
4527 reflections
307 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1357P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ e Å^{-3}

The positional parameters of the H atoms were calculated geometrically (C—H distances fixed at 0.96 Å) and refined using a riding model ($U_{iso} = 1.2U_{eq}$ of the parent atom). In the final difference Fourier map, the deepest hole is 0.97 Å from the Ga atom. The quality of the GaQ_3 crystals was not excellent, as indicated by a rather high R_{int} value of 0.144. There was a minor twin component in the crystals, which was left untreated.

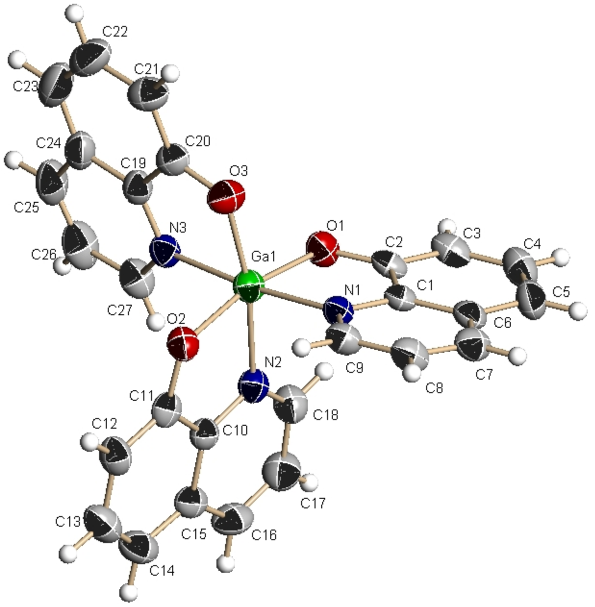


Figure 1
A view of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *COLLECT* (Nonius, 2000); 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); software used to prepare material for publication: *SHELXTL*.

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