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Diaquabis[1,4-bis(1-imidazolyl)benzene]trinitratolanthanum(III) 1,4-bis(1-imidazolyl)benzene hemisolvate monohydrate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
H-atom completeness 94%
R factor = 0.026
wR factor = 0.075
Data-to-parameter ratio = 14.2

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

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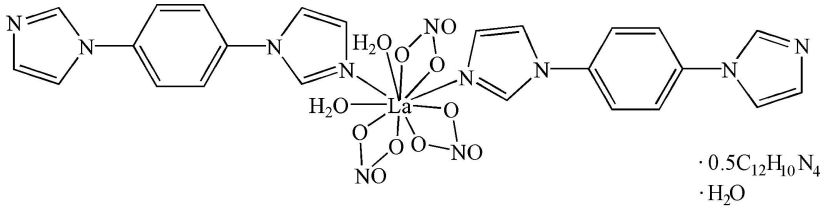
In the title complex, $[La(NO_3)_2(bib)_2(H_2O)_2]\cdot 0.5bib\cdot H_2O$ [bib is 1,4-bis(1-imidazolyl)benzene, $C_{12}H_{10}N_4$], the La^{III} atom is coordinated by two N atoms from two bib ligands and eight O atoms from three nitrate and two water ligands. This mononuclear complex is further extended into a three-dimensional structure *via* hydrogen bonds with free bib and water molecules of crystallization, the former lying on inversion centers.

Comment

In recent years, there has been great interest in the synthesis of metal organic frameworks (MOFs) with organic ligands and rare earth metals because of their novel structures, fascinating properties and potential applications (Tsukube & Shinoda, 2002; Zhang *et al.*, 2005). As a part of our systematic studies on MOFs with imidazole-containing ligands, the title complex, (I), was prepared, and its structure is described here.



(I)

The structural analysis revealed that the asymmetric unit of (I) consists of a mononuclear $[La(bib)_2(H_2O)_2(NO_3)_2]$ complex [bib is 1,4-bis(1-imidazolyl)benzene], a half-molecule of bib and one water molecule of crystallization, the free bib molecule lying on an inversion center. The central metal atom is coordinated by two N atoms from two bib ligands and eight O atoms from three nitrate and two water molecules. Each nitrate anion is coordinated to the La^{III} atom using two of its three O atoms. It is noteworthy that each bib ligand coordinates to one metal atom using one of the two imidazole groups, while the other imidazole group is free of coordination, as illustrated in Fig. 1.

The uncoordinated imidazole N atoms form O—H···N hydrogen bonds with coordinated water molecules [H···N = 1.789 (3) and 1.921 (3) Å]. In addition, there are O—H···O hydrogen bonds between the uncoordinated water molecule and coordinated nitrate anion [H···O = 2.164 (4) and 2.260 (4) Å] and various C—H···O hydrogen bonds (H···O = 2.24–2.59 Å). Such O—H···N, O—H···O and C—H···O hydrogen bonds link the mononuclear complex and solvent molecules into a three-dimensional structure, as shown in Fig. 2.

Experimental

A methanol solution (2 ml) of bib (49.4 mg, 0.24 mmol) was added to a solution of lanthanum(III) nitrate hexahydrate (20.8 mg, 0.05 mmol) in methanol (2 ml), and the mixture was stirred for 20 min at room temperature and filtered. The filtrate was allowed to stand at ambient temperature for one week and colorless crystals of the title complex were obtained in 56% yield based on the metal salt.

Crystal data

$[La(NO_3)_2(C_{12}H_{10}N_4)_2(H_2O)_2]\cdot 0.5C_{12}H_{10}N_4\cdot H_2O$	$Z = 2$
$M_r = 904.56$	$D_x = 1.668$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 12.334$ (9) Å	Cell parameters from 6488 reflections
$b = 12.980$ (11) Å	$\theta = 3.0$–$27.5°$
$c = 13.688$ (10) Å	$\mu = 1.26$ mm^{-1}
$\alpha = 112.510$ (11)°	$T = 173.1$ K
$\beta = 90.343$ (2)°	Block, colorless
$\gamma = 114.825$ (10)°	0.20 × 0.20 × 0.12 mm
$V = 1801$ (2) Å^3	

Data collection

Rigaku Saturn diffractometer	7085 reflections with $F^2 > 2\sigma(F^2)$
ω scans	$R_{int} = 0.032$
Absorption correction: multi-scan (Jacobson, 1998)	$\theta_{max} = 27.5°$
$T_{min} = 0.715$, $T_{max} = 0.859$	$h = -13 \rightarrow 15$
27576 measured reflections	$k = -16 \rightarrow 16$
7594 independent reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)] = 0.026$	$w = 1/[0.0008F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$wR(F^2) = 0.075$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.00$	$\Delta\rho_{max} = 1.38$ e Å^{-3}
7594 reflections	$\Delta\rho_{min} = -0.75$ e Å^{-3}
534 parameters	

Table 1
Selected geometric parameters (Å, °).

La1—O1	2.654 (2)	La1—O7	2.669 (2)
La1—O2	2.632 (2)	La1—O8	2.608 (3)
La1—O4	2.643 (2)	La1—O10	2.468 (2)
La1—O5	2.635 (2)	La1—O11	2.507 (3)

The H atoms of water molecules were located in a difference Fourier map and refined freely. Only one H atom was found in each case for two coordinated water molecules. C-bound H atoms were treated as riding [C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.]

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *SHELXTL* (Sheldrick, 1997); software used to prepare material for publication: *CrystalStructure*.

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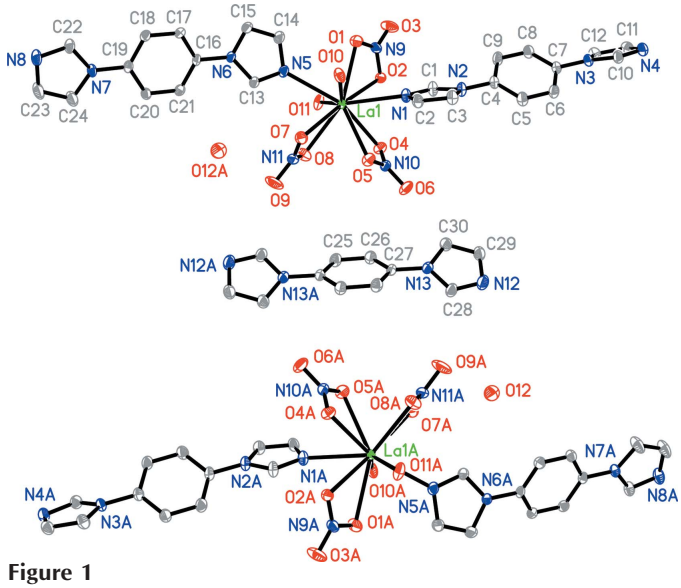

Figure 1
Twice the asymmetric unit of the title complex, with the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted. [Symmetry code: (i) $-x + 1, -y, -z + 1$.]

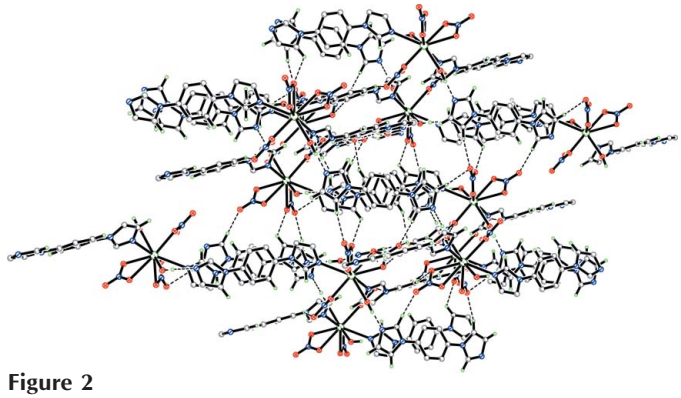

Figure 2
The three-dimensional packing of the title complex, viewed along the *b* axis. Dashed lines indicate the hydrogen-bonding interactions.

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