Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,1′-(2,5-Dimethyl-1,4-phenylenemethylene)-di-1*H*-imidazole dihydrate

Wen-Hua Wang,[a] Wei Dou,[a] Zhong-Lu You,[a] Wei-Sheng Liu[a]* and Da-Qi Wang[b]

[a]College of Chemistry and Chemical Engineering and State Key Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou 730000, People's Republic of China, and [b]Department of Chemistry, Liaocheng University, Liaocheng, 252000, People's Republic of China

Correspondence e-mail: liuws@lzu.edu.cn

Received 22 March 2006
Accepted 30 March 2006

In the title compound, $C_{16}H_{18}N_4\cdot 2H_2O$, there is one half-molecule (organic) and one water molecule in the asymmetric unit, the organic molecule being centrosymmetric. The separation of the two terminal N atoms is 11.296 (8) Å and the dihedral angle between the benzene and imidazole rings is 85.27 (18)°; the molecule adopts a *Z*-shaped conformation. A one-dimensional water chain links the organic molecules, forming a two-dimensional ladder-shaped network along the *b*-axis direction through intermolecular O—H···O and N—H···O hydrogen bonds.

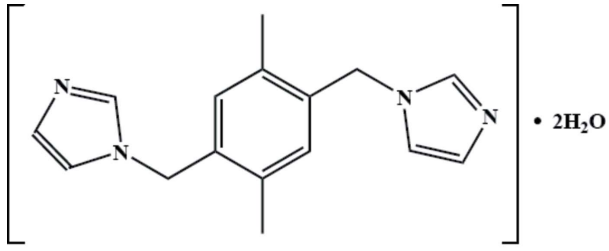
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.060
wR factor = 0.199
Data-to-parameter ratio = 14.3

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

Comment

By precisely selecting the modular building unit, chemists have now successfully synthesized a great many one-, two- and three-dimensional supramolecular architectures (Zheng *et al.*, 2005). Different sized and shaped organic spacer molecules have been chosen such that their nature and lengths vary systematically, whilst maintaining two terminal N-donor sites (Felloni *et al.*, 2002). In the present work, the crystal structure of a two terminal N-donor ligand, (I), a new spacer for metal-organic frameworks (MOFs), is reported.



(I)

In the crystal structure of (I), there is one half-molecule (organic) and one water molecule in the asymmetric unit and all the bond lengths are within normal ranges (Kubicki, 2004). The separation of the two terminal N atoms is 11.296 (8) Å and the dihedral angle between the benzene and imidazole rings is 85.27 (18)°; the molecule adopts a *Z*-shaped conformation (Fig. 1).

A one-dimensional water chain links the organic molecules, forming a two-dimensional ladder-shaped network along the *b*-axis direction (Fig. 2) through intermolecular O—H···O and N—H···O hydrogen bonds (Table 1). No obvious π–π stacking interactions were found.

Experimental

A mixture of KOH (0.22 g, 4.0 mmol) and 10 ml DMSO was stirred at room temperature for 5 min and then imidazole (0.54 g, 8.0 mmol)

was added. Stirring was continued for 45 min and then 1,4-bis-(bromomethyl)-2,5-dimethylbenzene (1.16 g, 4.0 mmol) was added. After stirring for an additional 45 min, the mixture was diluted with 20 ml water. The mixture was extracted with three 10 ml portions of diethyl ether, and each ether layer was washed with three 5 ml portions of water. The combined ether layers were dried over $CaCl_2$, and the solvent was removed at slightly reduced pressure. Recrystallization from ethanol gave the title compound (yield 1.36 g, 80%) as a colourless solid.

Crystal data

$C_{16}H_{18}N_4{\cdot}2H_2O$
M_r = 302.38
Monoclinic, $P2_1/n$
a = 8.692 (9) Å
b = 5.083 (2) Å
c = 19.068 (3) Å
β = 98.4080 (18)°
V = 833.3 (10) Å^3
Z = 2
D_x = 1.205 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
Block, colourless
0.49 × 0.17 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.961, T_{max} = 0.991
3999 measured reflections
1442 independent reflections
735 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.199
S = 1.01
1442 reflections
101 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1003P)^2 + 0.0713P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H8···N2	0.85	2.41	2.849 (5)	113
O1—H9···O1	0.85	2.13	2.818 (5)	138

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with fixed C—H distances of 0.93 (CH) [U_{iso}(H) = 1.2U_{eq}(C)] and 0.97 Å (CH_3) [U_{iso}(H) = 1.5U_{eq}(C)]. Water H atoms are refined freely [O—H = 0.85 Å and U_{iso}(H) = 1.2U_{eq}(O)].

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *PLATON* (Spek, 2005).

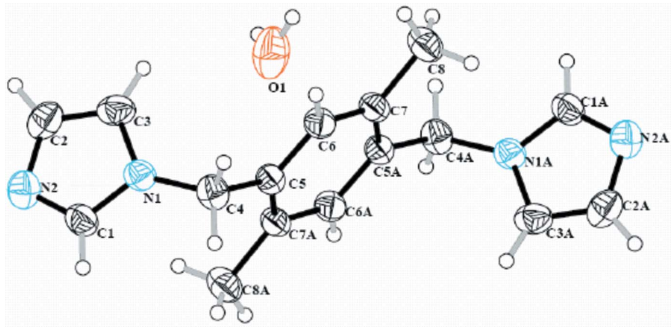


Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are drawn as spheres of arbitrary radii. [Symmetry code: (*A*) 1 − *x*, 2 − *y*, −*z*.]

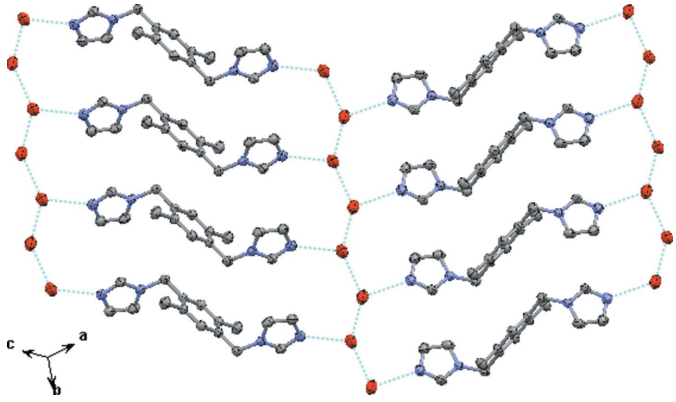


Figure 2
The crystal structure of compound (I) showing the two-dimensional ladder-type network along the *b*-axis direction. H atoms have been omitted

We acknowledge financial support from the NSFC (grant Nos. 20371022, 20431010 and 20021001), the Specialized Research Fund for the Doctoral Program of Higher Education, and the Key Project of the Ministry of Education of China (grant No. 01170).

References

Brandenburg, K. (2005). *DIAMOND*. Release 3.1a. Crystal Impact GbR, Bonn, Germany.
Bruker (2000). *SMART* (Version 5.0), *SAINT-Plus* (Version 6), *SHELXTL* (Version 6.1) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Felloni, M., Blake, A. J., Hubberstey, P., Wilson, C. & Schröder, M. (2002). *CrystEngComm*, **4**, 483–495.
Kubicki, M. (2004). *Acta Cryst.* B**60**, 333–342.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Spek, A. L. (2005). *PLATON*. Utrecht University, The Netherlands.
Zheng, P.-Q., Ren, Y.-P., Long, L.-S., Huang, R.-B. & Zheng, L.-S. (2005). *Inorg. Chem.* **44**, 1190–1192.