metal-organic compounds

catena-Poly[[[diaqualithium(I)]-μ-bis-(1H-imidazol-1-yl)methane-κ^2N^3:$N^{3'}$] chloride]

In-Chul Hwang* and Jeong Ah Chang

Department of Chemistry and Center for Basic Sciences, Pohang University of Science and Technology, Pohang 790-784, Republic of Korea
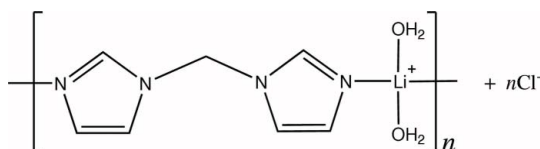Correspondence e-mail: spfe@postech.ac.kr

The title compound, $\{[Li(C_7H_8N_4)(H_2O)_2]Cl\}_n$, has a polymeric structure with Li^+ tetrahedrally coordinated by two N atoms of bis(imidazol-1-yl)methane and two water molecules, connected by O—H···Cl hydrogen bonds. The compound was prepared by a one-step reaction of LiCl and bis(imidazol-1-yl)methane in acetonitrile.

Related literature

For related literature, see: Diez-Barra *et al.* (1992); Cui *et al.* (2005); Duncan *et al.* (1996); Kitagawa *et al.* (2004).

Experimental

Crystal data

[Li(C$_7$H$_8$N$_4$)(H$_2$O)$_2$]Cl	V = 2249.7 (3) Å^3
M_r = 226.60	Z = 8
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 15.3208 (13) Å	μ = 0.32 mm^{-1}
b = 10.6729 (9) Å	T = 295 (2) K
c = 14.8266 (12) Å	0.35 × 0.25 × 0.25 mm
β = 111.887 (2)°	

Data collection

Bruker SMART CCD area-detector diffractometer	6383 measured reflections
	2310 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	2005 reflections with $I > 2\sigma(I)$
T_{min} = 0.837, T_{max} = 0.922	R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.126	
S = 1.07	
2310 reflections	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
153 parameters	$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.312 (2)	Li1—N4^i	2.070 (3)
C4—N4	1.305 (2)	Li1—N1	2.114 (3)
Li1—O2	1.926 (3)	N4—Li1^{ii}	2.070 (3)
Li1—O1	1.929 (3)		
O2—Li1—O1	115.43 (15)	O1—Li1—N1	103.73 (13)
O2—Li1—N4^i	109.63 (14)	N4^i—Li1—N1	104.66 (13)
O1—Li1—N4^i	115.59 (14)	C4—N4—Li1^{ii}	137.92 (14)
O2—Li1—N1	106.61 (13)	C6—N4—Li1^{ii}	115.97 (13)

Symmetry codes: (i) $x + \frac{1}{2}, y - \frac{1}{2}, z$; (ii) $x - \frac{1}{2}, y + \frac{1}{2}, z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1W···Cl1^{iii}	0.72 (3)	2.54 (3)	3.2394 (17)	165 (3)
O1—H2W···Cl1	0.87 (3)	2.31 (3)	3.1769 (17)	173 (2)
O2—H3W···Cl1^{iv}	0.93 (3)	2.25 (3)	3.1766 (16)	174 (3)
O2—H4W···Cl1^v	0.85 (3)	2.31 (3)	3.1631 (17)	174 (3)
C1—H1···Cl1^{iv}	0.93	2.74	3.6726 (17)	177

Symmetry codes: (iii) $-x + \frac{1}{2}, -y + \frac{3}{2}, -z$; (iv) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (v) $x + \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2121).

References

Bruker (2000). *SADABS* (Version 2.03), *SAINT* (Version 6.22), *SHELXTL* (Version 6.10) and *SMART* (Version 5.622). Bruker AXS Inc., Madison, Wisconsin, USA.

Cui, G. H., Li, J. R., Tian, J. L., Bu, X. H. & Batten, S. R. (2005). *Cryst. Growth Des.* **5**, 1775–1780.

Diez-Barra, E., de la Hoz, A., Sanchez-Migallon, A. & Tejeda, J. (1992). *Heterocycles*, **34**, 1365–1372.

Duncan, P. C. M., Goodgame, D. M. L., Menzer, S. & Williams, D. J. W. (1996). *Chem. Commun.* pp. 2127–2128.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Kitagawa, S., Kitaura, R. & Noro, S. (2004). *Angew. Chem. Int. Ed.* **43**, 2334–2375.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

supplementary materials

catena-Poly[[[diaqualithium(I)]-μ-bis(1*H*-imidazol-1-yl)methane-$\kappa^2 N^3$:$N^{3'}$] chloride]

I.-C. Hwang and J. A. Chang

Comment

The design and synthesis of superfunctional coordination polymers have been increased due to their intriguing architectures and flexible bridging ligands in supramolecular chemistry (Kitagawa *et al.*, 2004). The metal coordination architectures with the various heterocyclic aromatic compounds containing S–, N–, and O–donors are of diverse structural types. Significant progress has been achieved by Duncan *et al.* (1996), Cui *et al.* (2005) and others in this area. The formation of lithium coordination frameworks constructed from flexible *N,N'*–(1,1'–methyl)bis(imidazole) ligands and the exploitation of new synthetic methods are still less investigated. The selected *N,N'*–(1,1'–methyl)bis(imidazole) organic ligand with the N–hetero aromatic ring system could be a metal atom linker forming polymeric structure. The title compound (I) (Fig. 1) reveals tetrahedrally coordinated Li $^+$ cations interlinked by (imidazol–1–yl)methane into polymer structure connected by O—H···Cl$^-$ hydrogen bonds (Fig. 2, Table 1).

Experimental

N,N'-(1,1-methyl)bis(imidazole) was synthesized using a reported procedure (Diez-Barra *et al.*, 1992) and sublimated for purification at 453 K under high vacuum. A mixture of LiCl (15 mg, 0.357 mmol) and *N,N'*–(1,1'–methyl)bis(imidazole) (50 mg, 0.338 mmol) was placed in a 10 ml glass flask in CH3CN/H2O solution. The reaction mixture was heated at 333 K and then cooled to room temperature at a rate of 3 K/h. Colourless cubic single crystals were obtained in excellent yield.

Refinement

H atoms on *N,N'*–(1,1'–methyl)bis(imidazole) were positioned geometrically, with C—H = 0.93 Å and 0.97 Å, and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms on water molecules were localized from Fourier difference maps, but their atomic coordinates were not refined.
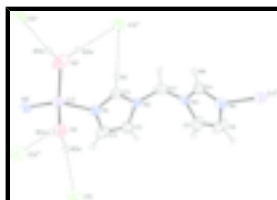
Figures

Fig. 1. The structure of (I) shows the atom–numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by circles of arbitrary size. [Symmetry code: a=-1/2 + *x*,3/2 − *y*,1/2 + *z*].

Fig. 2. View of the structure of (I) along the direction [010].

catena-Poly[[[diaqualithium(I)]-μ-bis(1*H*-imidazol-1-yl)methane- $\kappa^2 N^3 : N^{3'}$] chloride]

Crystal data

[Li(C$_7$H$_8$N$_4$)(H$_2$O)$_2$]Cl	$F_{000} = 944$
$M_r = 226.60$	$D_x = 1.338$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 3763 reflections
$a = 15.3208$ (13) Å	θ = 2.4–26.4º
$b = 10.6729$ (9) Å	μ = 0.32 mm^{-1}
$c = 14.8266$ (12) Å	$T = 295$ (2) K
β = 111.887 (2)º	Cubic, colourless
$V = 2249.7$ (3) Å^3	0.35 × 0.25 × 0.25 mm
$Z = 8$	

Data collection

Bruker SMART CCD area-detector diffractometer	2310 independent reflections
Radiation source: fine-focus sealed tube	2005 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.037$
$T = 295(2)$ K	$\theta_{max} = 26.4$º
φ and ω scans	$\theta_{min} = 2.4$º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -19 \rightarrow 17$
$T_{min} = 0.837$, $T_{max} = 0.922$	$k = -13 \rightarrow 13$
6383 measured reflections	$l = -18 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0746P)^2 + 0.5534P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.126$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.30$ e Å^{-3}
2310 reflections	$\Delta\rho_{min} = -0.28$ e Å^{-3}
153 parameters	Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0112 (12)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.22713 (11)	0.84155 (15)	0.38908 (11)	0.0445 (4)
H1	0.2621	0.9141	0.4118	0.053*
C2	0.17854 (13)	0.66703 (16)	0.31882 (14)	0.0564 (4)
H2	0.1741	0.5946	0.2825	0.068*
C3	0.11830 (12)	0.69897 (16)	0.36197 (13)	0.0557 (4)
H3	0.0662	0.6539	0.3615	0.067*
C4	0.02217 (10)	1.07972 (15)	0.38388 (11)	0.0460 (4)
H4	0.0734	1.1336	0.4074	0.055*
C5	−0.06186 (12)	0.90934 (17)	0.35524 (15)	0.0627 (5)
H5	−0.0813	0.8266	0.3540	0.075*
C6	−0.11482 (11)	1.00761 (17)	0.31130 (14)	0.0643 (5)
H6	−0.1787	1.0033	0.2736	0.077*
C7	0.10813 (13)	0.88548 (17)	0.46271 (11)	0.0570 (5)
H7A	0.0899	0.8301	0.5046	0.068*
H7B	0.1550	0.9432	0.5039	0.068*
Cl1	0.13680 (3)	0.63139 (4)	0.03017 (3)	0.0632 (2)
Li1	0.36216 (18)	0.7809 (3)	0.29113 (18)	0.0501 (6)
N1	0.24754 (9)	0.75656 (13)	0.33611 (9)	0.0487 (3)
N2	0.14968 (9)	0.81128 (12)	0.40654 (9)	0.0448 (3)
N3	0.02675 (8)	0.95582 (12)	0.40227 (8)	0.0420 (3)
N4	−0.06257 (10)	1.11534 (13)	0.32919 (10)	0.0507 (4)
O1	0.30302 (11)	0.81249 (16)	0.15348 (10)	0.0683 (4)
H1W	0.3229 (18)	0.834 (2)	0.119 (2)	0.080 (8)*
H2W	0.254 (2)	0.768 (2)	0.119 (2)	0.093 (8)*
O2	0.43632 (11)	0.91675 (16)	0.36717 (12)	0.0790 (5)
H3W	0.418 (2)	0.983 (3)	0.396 (2)	0.124 (10)*
H4W	0.490 (2)	0.899 (3)	0.409 (2)	0.097 (8)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0435 (8)	0.0459 (8)	0.0414 (8)	0.0082 (6)	0.0126 (6)	0.0043 (6)

C2	0.0577 (10)	0.0487 (9)	0.0617 (10)	0.0109 (8)	0.0210 (8)	−0.0091 (7)
C3	0.0520 (9)	0.0495 (9)	0.0683 (11)	0.0041 (7)	0.0255 (8)	0.0008 (8)
C4	0.0440 (8)	0.0460 (8)	0.0539 (8)	0.0018 (6)	0.0249 (7)	−0.0027 (7)
C5	0.0501 (9)	0.0487 (9)	0.0934 (13)	−0.0034 (8)	0.0316 (9)	−0.0213 (9)
C6	0.0417 (9)	0.0680 (11)	0.0723 (12)	0.0084 (8)	0.0086 (8)	−0.0321 (9)
C7	0.0646 (11)	0.0690 (11)	0.0401 (8)	0.0271 (9)	0.0227 (7)	0.0052 (7)
Cl1	0.0673 (3)	0.0568 (3)	0.0587 (3)	−0.00934 (19)	0.0158 (2)	0.00771 (18)
Li1	0.0513 (14)	0.0538 (15)	0.0450 (13)	0.0108 (12)	0.0177 (11)	0.0014 (11)
N1	0.0470 (7)	0.0540 (8)	0.0466 (7)	0.0138 (6)	0.0190 (6)	0.0028 (6)
N2	0.0473 (7)	0.0481 (7)	0.0406 (6)	0.0133 (5)	0.0181 (5)	0.0036 (5)
N3	0.0435 (7)	0.0448 (7)	0.0417 (6)	0.0063 (5)	0.0206 (5)	−0.0036 (5)
N4	0.0522 (8)	0.0560 (8)	0.0476 (7)	0.0148 (6)	0.0229 (6)	0.0000 (6)
O1	0.0636 (9)	0.0930 (11)	0.0448 (7)	−0.0140 (8)	0.0163 (7)	0.0036 (7)
O2	0.0591 (8)	0.0836 (10)	0.0792 (10)	0.0102 (7)	0.0084 (7)	−0.0280 (8)

Geometric parameters (Å, °)

C1—N1	1.312 (2)	C6—H6	0.9300
C1—N2	1.345 (2)	C7—N3	1.4450 (19)
C1—H1	0.9300	C7—N2	1.4564 (19)
C2—C3	1.348 (2)	C7—H7A	0.9700
C2—N1	1.376 (2)	C7—H7B	0.9700
C2—H2	0.9300	Li1—O2	1.926 (3)
C3—N2	1.367 (2)	Li1—O1	1.929 (3)
C3—H3	0.9300	Li1—N4[i]	2.070 (3)
C4—N4	1.305 (2)	Li1—N1	2.114 (3)
C4—N3	1.347 (2)	N4—Li1[ii]	2.070 (3)
C4—H4	0.9300	O1—H1W	0.72 (3)
C5—C6	1.337 (3)	O1—H2W	0.87 (3)
C5—N3	1.367 (2)	O2—H3W	0.93 (3)
C5—H5	0.9300	O2—H4W	0.85 (3)
C6—N4	1.369 (2)		
N1—C1—N2	111.94 (14)	O2—Li1—O1	115.43 (15)
N1—C1—H1	124.0	O2—Li1—N4[i]	109.63 (14)
N2—C1—H1	124.0	O1—Li1—N4[i]	115.59 (14)
C3—C2—N1	110.51 (15)	O2—Li1—N1	106.61 (13)
C3—C2—H2	124.7	O1—Li1—N1	103.73 (13)
N1—C2—H2	124.7	N4[i]—Li1—N1	104.66 (13)
C2—C3—N2	105.76 (15)	C1—N1—C2	104.69 (13)
C2—C3—H3	127.1	C1—N1—Li1	121.03 (14)
N2—C3—H3	127.1	C2—N1—Li1	134.20 (13)
N4—C4—N3	112.12 (14)	C1—N2—C3	107.11 (13)
N4—C4—H4	123.9	C1—N2—C7	125.78 (14)
N3—C4—H4	123.9	C3—N2—C7	127.11 (15)
C6—C5—N3	105.70 (15)	C4—N3—C5	106.63 (13)
C6—C5—H5	127.2	C4—N3—C7	127.12 (14)
N3—C5—H5	127.2	C5—N3—C7	126.22 (14)
C5—C6—N4	111.07 (14)	C4—N4—C6	104.48 (14)

C5—C6—H6	124.5	C4—N4—Li1ⁱⁱ	137.92 (14)
N4—C6—H6	124.5	C6—N4—Li1ⁱⁱ	115.97 (13)
N3—C7—N2	112.83 (12)	Li1—O1—H1W	130 (2)
N3—C7—H7A	109.0	Li1—O1—H2W	118.7 (17)
N2—C7—H7A	109.0	H1W—O1—H2W	105 (3)
N3—C7—H7B	109.0	Li1—O2—H3W	129.5 (18)
N2—C7—H7B	109.0	Li1—O2—H4W	117.5 (19)
H7A—C7—H7B	107.8	H3W—O2—H4W	102 (2)
N1—C2—C3—N2	0.5 (2)	C2—C3—N2—C1	−0.41 (18)
N3—C5—C6—N4	−0.2 (2)	C2—C3—N2—C7	179.65 (15)
N2—C1—N1—C2	0.13 (17)	N3—C7—N2—C1	101.44 (18)
N2—C1—N1—Li1	177.35 (12)	N3—C7—N2—C3	−78.6 (2)
C3—C2—N1—C1	−0.39 (19)	N4—C4—N3—C5	0.27 (17)
C3—C2—N1—Li1	−177.07 (15)	N4—C4—N3—C7	−177.63 (12)
O2—Li1—N1—C1	13.56 (19)	C6—C5—N3—C4	−0.06 (19)
O1—Li1—N1—C1	−108.73 (15)	C6—C5—N3—C7	177.86 (14)
N4ⁱ—Li1—N1—C1	129.70 (14)	N2—C7—N3—C4	−104.58 (18)
O2—Li1—N1—C2	−170.19 (16)	N2—C7—N3—C5	77.9 (2)
O1—Li1—N1—C2	67.5 (2)	N3—C4—N4—C6	−0.35 (17)
N4ⁱ—Li1—N1—C2	−54.1 (2)	N3—C4—N4—Li1ⁱⁱ	163.57 (15)
N1—C1—N2—C3	0.18 (17)	C5—C6—N4—C4	0.3 (2)
N1—C1—N2—C7	−179.88 (13)	C5—C6—N4—Li1ⁱⁱ	−167.78 (15)

Symmetry codes: (i) *x*+1/2, *y*−1/2, *z*; (ii) *x*−1/2, *y*+1/2, *z*.

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H1W⋯Cl1ⁱⁱⁱ	0.72 (3)	2.54 (3)	3.2394 (17)	165 (3)
O1—H2W⋯Cl1	0.87 (3)	2.31 (3)	3.1769 (17)	173 (2)
O2—H3W⋯Cl1ⁱᵛ	0.93 (3)	2.25 (3)	3.1766 (16)	174 (3)
O2—H4W⋯Cl1ᵛ	0.85 (3)	2.31 (3)	3.1631 (17)	174 (3)
C1—H1⋯Cl1ⁱᵛ	0.93	2.74	3.6726 (17)	177

Symmetry codes: (iii) −*x*+1/2, −*y*+3/2, −*z*; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2; (v) *x*+1/2, −*y*+3/2, *z*+1/2.

Fig. 1

Fig. 2