

## Experimental

The title compound was prepared as described by Couper *et al.* (1998). Suitable crystals were obtained directly from the synthesis; they were relatively large and could not be cut without causing damage which resulted in a deterioration of the diffraction pattern.

### Crystal data

[Li<sub>2</sub>(C<sub>7</sub>H<sub>14</sub>N)<sub>2</sub>(C<sub>6</sub>H<sub>16</sub>N<sub>2</sub>)]

$M_r = 354.47$

Triclinic

$P\bar{1}$

$a = 7.904$  (5) Å

$b = 8.899$  (6) Å

$c = 9.335$  (7) Å

$\alpha = 108.26$  (4)°

$\beta = 112.63$  (3)°

$\gamma = 91.41$  (3)°

$V = 567.6$  (7) Å<sup>3</sup>

$Z = 1$

$D_x = 1.037$  Mg m<sup>-3</sup>

$D_m$  not measured

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 30

reflections

$\theta = 8.5$ – $12.0$ °

$\mu = 0.060$  mm<sup>-1</sup>

$T = 160$  (2) K

Block

$1.15 \times 0.85 \times 0.65$  mm

Colourless

### Data collection

Stoe–Siemens diffractometer  
with Cryostream cooler  
(Cosier & Glazer, 1986)

$\omega/\theta$  scans

Absorption correction: none

4012 measured reflections

2004 independent reflections

1631 reflections with

$I > 2\sigma(I)$

$R_{int} = 0.026$

$\theta_{max} = 24.99$ °

$h = -9 \rightarrow 9$

$k = -10 \rightarrow 10$

$l = -11 \rightarrow 11$

5 standard reflections

frequency: 60 min

intensity decay: 2.9%

### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.109$

$S = 1.027$

2004 reflections

122 parameters

H atoms: see below

$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.1264P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.199$  e Å<sup>-3</sup>

$\Delta\rho_{min} = -0.181$  e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Li—N1	2.038 (3)	Li—N2	2.161 (2)
Li—N1 <sup>i</sup>	2.050 (3)		
N1—Li—N1 <sup>i</sup>	107.89 (11)	Li—N1—Li <sup>i</sup>	72.11 (11)
N1—Li—N2	133.67 (11)	C8—N2—Li	122.92 (10)
N1 <sup>i</sup> —Li—N2	118.15 (11)	N2—C8—C8 <sup>ii</sup>	113.35 (13)
C1—N1—C5	106.17 (11)		

Symmetry codes: (i)  $1 - x, 1 - y, 1 - z$ ; (ii)  $1 - x, 2 - y, 1 - z$ .

H atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds), and with  $U_{iso}$  constrained to be 1.2 (1.5 for methyl groups) times  $U_{eq}$  of the carrier atom.

Data collection: *DIF4* (Stoe & Cie, 1988). Cell refinement: *DIF4*. Data reduction: local programs. Program(s) used to

solve structure: *SHELXTL* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and local programs.

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Supplementary data for this paper are available from the IUCR electronic archives (Reference: LN1078). Services for accessing these data are described at the back of the journal.

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## Dichlorobis(2-hydroxymethyl-1-methylimidazole-*N*<sup>3</sup>)zinc(II)

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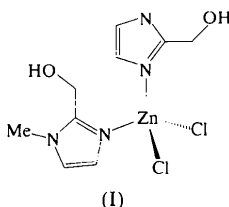
## Abstract

The crystal structure of the title complex, [ZnCl<sub>2</sub>(C<sub>5</sub>H<sub>8</sub>N<sub>2</sub>O)<sub>2</sub>], is composed of discrete molecules. The Zn<sup>II</sup> atom is coordinated by two chloride ligands and two N atoms from the imidazole ligands in a distorted tetrahedral coordination environment, with Zn—N distances of

1.998 (5) and 2.005 (5) Å, Zn—Cl distances of 2.230 (2) and 2.247 (2) Å, and N—Zn—N and Cl—Zn—Cl angles of 111.15 (19) and 114.59 (8)°, respectively.

### Comment

Metal complexes of imidazole and its derivatives are of current interest in the investigation of model complexes relevant to metalloenzymes, since imidazole, a residue of histidine, is frequently found to ligate metal ions in the active centres of metalloenzymes. Only a limited number of zinc(II) imidazole complexes have been structurally characterized (Chen *et al.*, 1994, 1996; Kimura *et al.*, 1991). In the course of our systematic study of metal complexes of imidazole-like ligands, we have isolated the title complex, [ZnCl<sub>2</sub>(MeImOH)<sub>2</sub>] (MeImOH is 2-hydroxy-methyl-1-methylimidazole; Palleé & Henning, 1966), (I), and report herein its preparation and structure.



The crystal structure of (I) consists of discrete [ZnCl<sub>2</sub>(MeImOH)<sub>2</sub>] molecules. The Zn<sup>II</sup> atom is coordinated by two chloride ligands and an N atom from each of two MeImOH ligands in a distorted tetrahedral coordination environment (Fig. 1). The Zn—N bond lengths [1.998 (5) and 2.005 (5) Å] are comparable to the Zn—N<sub>Him</sub> (Him = imidazole) bond lengths found in tetrahedrally coordinated Zn<sup>II</sup> complexes, such as [Zn(Him)<sub>2</sub>(MeCO<sub>2</sub>)<sub>2</sub>] [1.996 (2) and 2.003 (3) Å; Chen *et al.*, 1994] and [Zn(Him)<sub>2</sub>(ClO<sub>4</sub>)<sub>2</sub>] (1.999 Å; Bear *et al.*, 1975). The Zn—Cl bond lengths [2.230 (2) and 2.247 (2) Å] are also comparable to those found

in aquadichloro(pyridine betaine)zinc(II) [2.219 (1) and 2.231 (1) Å] and dichlorobis(pyridine betaine)zinc(II) [2.238 (2) and 2.262 (1) Å] (Chen & Mak, 1991). Bond angles at the Zn atom deviate from the ideal value of 109.5°.

The structure is consolidated by extensive intermolecular hydrogen bonds between chloride ligands and the hydroxyl group of the organic ligands. Adjacent discrete molecules are bridged by hydrogen bonds between atoms Cl1 and O1, and by bifurcated hydrogen bonds between three hydroxyl groups from the organic ligands (Table 2). This hydrogen bonding produces a three-dimensional network in the solid.

### Experimental

An aqueous solution (5 ml) of ZnCl<sub>2</sub> (0.14 g, 1.0 mmol) was added to an aqueous ethanolic solution (10 ml) of MeImOH (0.22 g, 2 mmol). The mixture was stirred for 20 min. The resulting solution was allowed to stand in air, and colourless crystals of the title complex were formed after one month.

#### Crystal data

[ZnCl<sub>2</sub>(C<sub>5</sub>H<sub>8</sub>N<sub>2</sub>O)<sub>2</sub>]

*M<sub>r</sub>* = 360.54

Monoclinic

*P*2<sub>1</sub>/*n*

*a* = 7.281 (2) Å

*b* = 16.084 (7) Å

*c* = 12.849 (6) Å

β = 100.51 (2)°

*V* = 1479.5 (10) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.619 Mg m<sup>-3</sup>

*D<sub>m</sub>* not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 7.5–15.0°

μ = 2.024 mm<sup>-1</sup>

*T* = 293 (2) K

Block

0.60 × 0.50 × 0.25 mm

Colourless

#### Data collection

Siemens *R3m* diffractometer

ω scans

Absorption correction:

empirical via ψ scans

(North *et al.*, 1968)

*T<sub>min</sub>* = 0.458, *T<sub>max</sub>* = 0.603

3127 measured reflections

2894 independent reflections

1924 reflections with

*I* > 2σ(*I*)

*R<sub>int</sub>* = 0.025

θ<sub>max</sub> = 26°

*h* = 0 → 8

*k* = 0 → 19

*l* = -15 → 15

2 standard reflections

every 118 reflections

intensity decay: 1.1%

#### Refinement

Refinement on *F*<sup>2</sup>

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.060

*wR*(*F*<sup>2</sup>) = 0.166

*S* = 1.033

2894 reflections

172 parameters

H atoms treated by a

mixture of independent

and constrained refinement

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0740*P*)<sup>2</sup> + 2.7045*P*]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = 0.001

Δρ<sub>max</sub> = 0.865 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.570 e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from

*International Tables for Crystallography* (Vol. C)

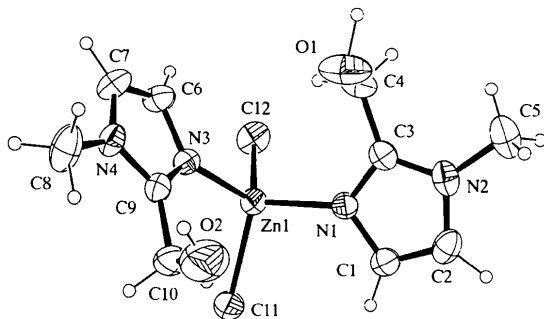


Fig. 1. ORTEP-3 (Farrugia, 1997) plot showing the molecular structure of (I), with 35% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radii.

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Zn1—N1	1.998 (5)	Zn1—Cl2	2.230 (2)
Zn1—N3	2.005 (5)	Zn1—Cl1	2.247 (2)
N1—Zn1—N3	111.15 (19)	Cl2—Zn1—Cl1	114.59 (8)
N1—Zn1—Cl2	109.90 (14)	C3—N1—Zn1	128.6 (4)
N3—Zn1—Cl2	106.42 (15)	C1—N1—Zn1	125.4 (4)
N1—Zn1—Cl1	104.56 (15)	C9—N3—Zn1	129.8 (4)
N3—Zn1—Cl1	110.31 (15)	C6—N3—Zn1	123.5 (4)

Table 2. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...Cl1 <sup>i</sup>	1.08	1.99	3.071 (7)	176.0
O2—H2...O1 <sup>ii</sup>	1.05	2.35	2.767 (10)	102.1
O2—H2...O2 <sup>ii</sup>	1.05	2.41	2.877 (14)	106.1

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

Hydroxy H atoms were fixed at positions found in a difference map. Other H atoms were refined using a riding model, with  $U(\text{H}) = 0.08 \text{\AA}$ .

Data collection: *R3m Software* (Siemens, 1990). Cell refinement: *R3m Software*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997). Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1299). Services for accessing these data are described at the back of the journal.

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## Disodium 4,4'-difluoro-3,3'-carbonyl-dibenzenesulfonate hydrofluoride tetra-hydrate

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## Abstract

The structure determination of the title compound,  $2\text{Na}^+ \cdot \text{C}_{13}\text{H}_6\text{F}_2\text{O}_7\text{S}_2^- \cdot \text{HF} \cdot 4\text{H}_2\text{O}$ , indicates that intermolecular physical crosslinking through  $\text{Na}^+$  ions exists in the crystal so that ionic aggregates are formed.

## Comment

Sulfones have received considerable attention owing to their high performance in the membrane separation process (Ueda *et al.*, 1993; Wang *et al.*, 1998). This method of synthesizing sulfonated polymers *via* nucleophilic polycondensation of disodium 4,4'-difluoro-3,3'-carbonyldibenzenesulfonate hydrofluoride tetrahydrate, (I), and bis-phenols is more advantageous than traditional routes of polymer sulfonation, and polymers thus obtained are suitable for gas dehumidification and ion-exchange membranes.

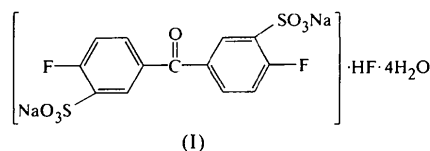


Fig. 1 shows the molecule with the atom-numbering scheme and Fig. 2 illustrates the intermolecular linking through  $\text{Na}^+$  ions. The phenyl rings are not coplanar; the torsion angle  $\text{C9—C8—C3—C4}$  is  $57.2(3)^\circ$ , which is larger than the corresponding value of  $47.1(1)^\circ$  in 4,4'-difluorobenzophenone (Maginn & Davey, 1994), owing to the introduction of bulky sodium sulfonate groups. It can be seen from Fig. 2 that physical crosslinking through  $\text{Na}^+$  ions is present in the compound. The  $\text{Na}^+$  ions coordinate with water molecules, O atoms and the hydrofluoride  $\text{F}^-$  ion. The ionic aggregate is constructed through coordination of the different molecules with  $\text{Na}^+$  ions. The water molecules and  $\text{F}^-$  ion, as main ligands, strengthen the intermolecular forces.