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Key indicators

Single-crystal X-ray study
T = 294 K
Mean $\sigma(C-C)$ = 0.003 Å
R factor = 0.036
wR factor = 0.110
Data-to-parameter ratio = 17.3

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

1,1'-Dimethyl-2,2'-[p-phenylenebis(methylene-
thio)]di-1H-imidazole monohydrate

In the title compound, $C_{16}H_{18}N_4S_2 \cdot H_2O$, a crystallographic twofold axis runs perpendicular to the benzene ring; the solvent water oxygen atom also sits on a twofold axis. The terminal (1-methylimidazole-2-yl)-sulfanyl groups adopt a *trans* configuration with respect to the benzene ring and the dihedral angle between the two imidazole rings is $81.9(3)^\circ$.

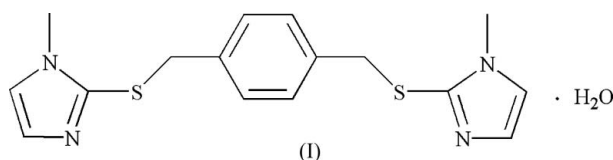
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Comment

As a type of ditopic ligand, dithioethers can be used as bridging ligands in the construction of coordination polymers with soft metal ions. *N*-heterocyclic groups have been synthesized and investigated (Sharma *et al.*, 1999; Constable *et al.*, 2002; Bu *et al.*, 2003; Hong *et al.*, 2000). Earlier studies have reported that several imidazole derivatives possess diverse coordination properties (Hester *et al.*, 1997, Yang *et al.*, 2000). In order to study the properties of these compounds, we have synthesized several new imidazole derivatives, and we present here the crystal structure of the title compound, (I).



In the molecular structure of (I), a crystallographic twofold axis runs perpendicular to the benzene ring; the solvent water oxygen atom also sits on a twofold axis. The terminal (1-methylimidazole-2-yl)-sulfanyl groups adopt a *trans* config-

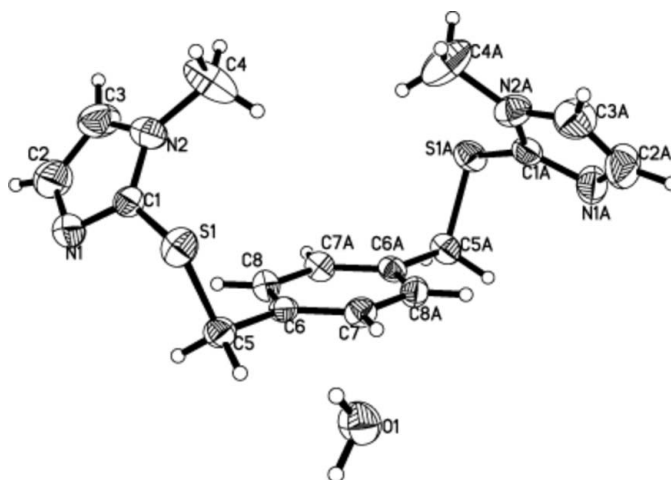


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. The suffix A corresponds to the symmetry position $\frac{3}{2} - x, y, -z$.

uration with respect to the benzene ring. The two imidazole rings are approximately perpendicular, with a dihedral angle of $81.9(3)^\circ$ between them. The dihedral angle between the imidazole ring and the benzene ring is $49.1(3)^\circ$. In the substituted imidazole ring, the atom C1 attached to the sulfanyl group has a distorted trigonal geometry, with the N2–C1–N1 [$110.6(2)^\circ$] and N1–C1–S1 [$125.24(18)^\circ$] angles deviating significantly from the ideal sp^2 hybridized values.

As a result of the p - π conjugation between atom S and the imidazole ring, the Csp^2 –S bond (S1–C1 = $1.743(2)$ Å) is significantly shorter than the Csp^3 –S bond (C5–S1 = $1.825(2)$ Å). These values compare well with the values of $1.726(2)$, $1.720(8)$ and $1.800(3)$, $1.811(2)$ Å reported in the literature (Wang *et al.*, 2004, 2005).

Experimental

A solution of 1,4-dibromomethylbenzene (1.32 g, 5 mmol) in ethanol was added dropwise to a mixture of 1-methyl-2-thio-imidazole (1.26 g, 11 mmol), KOH (0.615 g, 11 mmol) and ethanol (5 ml). The reaction mixture was then stirred for 24 h at room temperature. The precipitate was added gradually to 30 ml water and a new white precipitate appeared. This precipitate was filtered off, washed with water and recrystallized from ethanol and water (yield 65%, m.p. 341 – 342 K). Spectroscopic analysis: IR (KBr, vcm^{-1}): 3100, 2924, 1506, 1452, 1410, 1336, 1280, 1098, 840, 746, 686; ^1H NMR (CDCl_3 , δ , p.p.m.): 3.29 (3H, s), 4.14 (2H, s), 6.87 (1H, s), 7.04 (1H, s), 7.10 (1H, s). Anal. calcd for $\text{C}_{16}\text{H}_{18}\text{N}_4\text{S}_2\cdot\text{H}_2\text{O}$: C, 55.14; H, 5.78; N, 16.08%. Found: C, 55.22; H, 5.69; N, 16.11%. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in a mixture of ethanol and water (1:4 v/v).

Crystal data

$\text{C}_{16}\text{H}_{18}\text{N}_4\text{S}_2\cdot\text{H}_2\text{O}$	Mo $K\alpha$ radiation
$M_r = 348.48$	Cell parameters from 3069 reflections
Orthorhombic, <i>Ibca</i>	$\theta = 2.3$ – 25.9°
$a = 13.1962(19)$ Å	$\mu = 0.31$ mm^{-1}
$b = 15.049(2)$ Å	$T = 294(2)$ K
$c = 18.124(4)$ Å	Block, colourless
$V = 3599.4(10)$ Å ³	$0.22 \times 0.16 \times 0.10$ mm
$Z = 8$	
$D_x = 1.286$ Mg m^{-3}	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	1854 independent reflections
φ and ω scans	1288 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.040$
$T_{\text{min}} = 0.943$, $T_{\text{max}} = 0.970$	$\theta_{\text{max}} = 26.4^\circ$
9524 measured reflections	$h = -16 \rightarrow 16$
	$k = -7 \rightarrow 18$
	$l = -22 \rightarrow 21$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0286P)^2 + 3.8597P]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.110$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.22$ e \AA^{-3}
1854 reflections	$\Delta\rho_{\text{min}} = -0.23$ e \AA^{-3}
107 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.0045 (4)

Table 1

Selected geometric parameters (Å, °).

S1–C1	1.743 (2)	S1–C5	1.825 (2)
N1–C1–N2	110.6 (2)	N1–C1–S1	125.24 (18)
C2–N1–C1–S1	179.9 (2)	S1–C5–C6–C7	91.9 (2)

All C-bound H atoms were positioned geometrically and refined as riding (C–H 0.93–0.97 Å). For the CH and CH₂ groups, $U_{\text{iso}}(\text{H})$ values were set equal to $1.2U_{\text{eq}}(\text{carrier atom})$ and for the methyl groups they were set equal to $1.5U_{\text{eq}}(\text{carrier atom})$. The H atom attached to the water O atom was located in a difference map, then refined as riding with O–H = 0.88 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$.

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

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