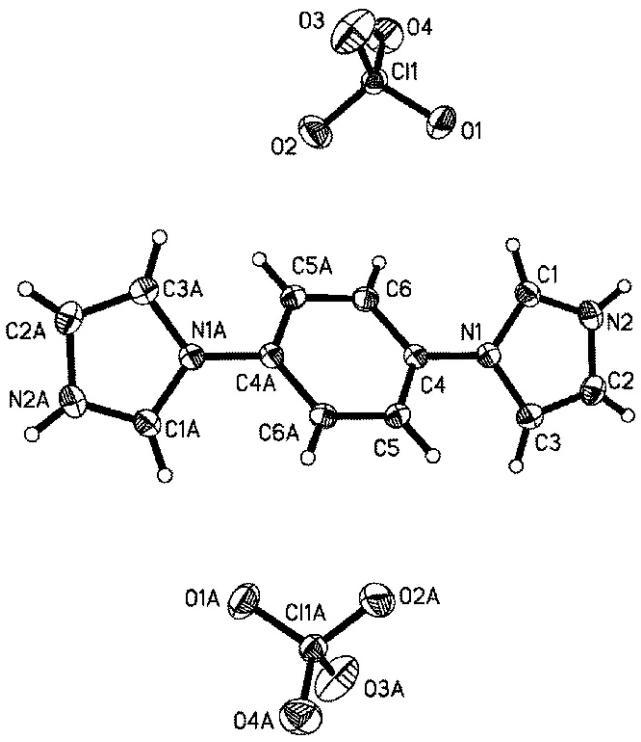
Crystal structure of (1,4-di(hydrogen-imidazolyl)-benzene)bisperchlorate, $(C_{12}H_{12}N_4)(ClO_4)_2$

Yi-Fang Deng* and Xue Nie

Department of Chemistry and Materials Science, Hengyang Normal University, Hengyang 421008, Hunan, P. R. China

Received March 13, 2011, accepted and available on-line July 15, 2011; CCDC no. 1267/3444



Abstract

$C_{12}H_{12}Cl_2N_4O_8$, monoclinic, $P12_1/c1$ (no. 14), a = 5.147(1) Å, b = 10.757(3) Å, c = 15.141(4) Å, β = 105.725(3)°, V = 806.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.042, $wR_{ref}(F^2)$ = 0.109, T = 273 K.

Source of material

The title compound was prepared from of $Mn(ClO_4)_2 \cdot 6H_2O$ (0.031 g, 0.1 mmol), 1,4-di(1-imidazolyl)benzene (0.021 g, 0.1 mmol), CH_3CH_2OH (4 ml) and H_2O (6 ml) sealed in a 15 ml Teflon-lined stainless steel reactor, which was heated at 393 K for 72 h and then it was cooled to room temperature. Block-shaped colorless crystals of the title compound were collected.

Experimental details

All the hydrogen atoms were generated geometrically and refined isotropically using the riding model with U_{iso}(H) = 1.2 U_{eq}(C/N).

Discussion

Imidazole derivatives have been found to possess widespread biological activities, many of them have been developed as medical and functional materials [1,2].

The title crystal structure contains a protonated 1,4-di(1-imidazolyl)benzene cation and perchlorate anions. The two imidazolyl rings (C1/N2/C2/C3/N1) and (C1A/N2A/C2A/C3A/N1A) are coplanar, but the benzene ring (C4/C5/C6/C4A/C5A/C6A) and imidazolyl rings are twisted. The dihedral angle between the benzene and imidazolyl rings is 34.88°. The bond lengths and angles are within normal ranges. The uncoordinated nitrogen atoms of imidazole group and perchlorate oxygen atoms are the hydrogen-bonding donor and acceptor, respectively, resulting in the complex one-dimensional architecture through supramolecular interactions such as N–H···O hydrogen bonding (d(N2—O3) = 2.959(4) Å and ∠N2–H2A–O3 = 151°). Moreover, these chains are further interconnected by weak intermolecular C–H···O hydrogen bonds (d (C1—O1) = 3.457(4) Å and ∠C1–H1–O1 = 167°) and weak C–H···π interactions to generate a three-dimensional supramolecular structure.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.08 × 0.10 × 0.14 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.56 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6036, 1588
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1487
$N(param)_{refined}$:	119
Programs:	SHELXS-97, SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	1.2536	0.5996	0.0334	0.051
H(1)	4*e*	0.7361	0.7232	0.1582	0.044
H(2A)	4*e*	1.0484	0.5644	0.1542	0.050
H(3)	4*e*	1.0669	0.7872	–0.0461	0.049
H(5)	4*e*	0.6193	0.8843	–0.1129	0.045
H(6)	4*e*	0.6715	0.9467	0.1532	0.045

* Correspondence author (e-mail: yifang7124@163.com)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.8429(4)	0.7941(2)	0.0489(1)	0.0345(9)	0.0307(9)	0.0328(9)	0.0002(7)	0.0095(7)	0.0008(7)
C(2)	4*e*	1.1336(5)	0.6458(2)	0.0470(2)	0.036(1)	0.041(1)	0.051(1)	0.005(1)	0.011(1)	–0.005(1)
C(1)	4*e*	0.8413(5)	0.7157(2)	0.1173(2)	0.040(1)	0.035(1)	0.034(1)	–0.0001(9)	0.0087(9)	0.0040(9)
N(2)	4*e*	1.0147(4)	0.6261(2)	0.1166(1)	0.044(1)	0.034(1)	0.044(1)	0.0036(8)	0.0050(9)	0.0057(8)
C(3)	4*e*	1.0260(5)	0.7507(2)	0.0042(2)	0.040(1)	0.043(1)	0.042(1)	0.002(1)	0.016(1)	0.000(1)
C(4)	4*e*	0.6711(4)	0.9006(2)	0.0240(2)	0.035(1)	0.028(1)	0.032(1)	–0.0005(8)	0.0101(8)	0.0019(8)
C(5)	4*e*	0.5711(5)	0.9308(2)	–0.0680(2)	0.051(1)	0.036(1)	0.030(1)	0.005(1)	0.0154(9)	–0.0008(9)
C(6)	4*e*	0.6021(5)	0.9685(2)	0.0919(2)	0.048(1)	0.039(1)	0.026(1)	0.005(1)	0.0098(9)	0.0034(8)
Cl(1)	4*e*	0.3556(1)	0.89212(5)	0.30809(4)	0.0404(4)	0.0379(4)	0.0374(3)	–0.0009(2)	0.0116(2)	–0.0039(2)
O(1)	4*e*	0.4368(5)	0.7953(2)	0.2574(2)	0.079(2)	0.048(1)	0.078(1)	–0.000(1)	0.040(1)	–0.016(1)
O(2)	4*e*	0.4369(6)	1.0099(2)	0.2806(2)	0.143(2)	0.044(1)	0.071(2)	–0.017(1)	0.053(2)	–0.004(1)
O(3)	4*e*	0.0712(4)	0.8907(3)	0.2941(2)	0.043(1)	0.110(2)	0.092(2)	–0.002(1)	0.009(1)	–0.043(2)
O(4)	4*e*	0.4788(5)	0.8799(2)	0.4040(2)	0.076(2)	0.089(2)	0.045(1)	0.008(1)	0.005(1)	0.005(1)

Acknowledgment. This work was supported by the Hengyang Bureau of Science & Technology (grant no. 2009KJ28).

References

1. Bromberg, L.; Chen, L.; Chang, E. P.; Wang, S.; Hatton A. T.: Reactive silver and cobalt nanoparticles modified with fatty acid ligands functionalized by imidazole derivatives. Chem. Mater. **22** (2010) 5383-5391.
2. Huang, X. C.; Zhang, J. P.; Lin, Y. Y.; Yu, X. L.; Chen, X. M.: Two mixed-valence copper(I,II) imidazolate coordination polymers: metal-valence tuning approach for new topological structures. Chem. Commum. (2004) 1100-1101.
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.