

Yong-Tao Wang,^{a*} Gui-Mei Tang,^a Da-Wei Qin,^a Hong-Dong Duan^a and Seik Weng Ng^b^aDepartment of Chemical Engineering, Shandong Institute of Light Industry, Jinan, Shandong 250100, People's Republic of China, and ^bDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: ceswyt@yahoo.com.cn

Key indicators

Single-crystal X-ray study
 $T = 295\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
Disorder in solvent or counterion
 R factor = 0.059
 wR factor = 0.165
Data-to-parameter ratio = 11.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

2,5-Di-4-pyridinio-1,3,4-oxadiazole bis(perchlorate) monohydrate

In the title structure, $\text{C}_{12}\text{H}_{10}\text{N}_4\text{O}^{2+} \cdot 2\text{ClO}_4^- \cdot \text{H}_2\text{O}$, the dication is not planar and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds link it to perchlorate anions and water molecules. In addition, $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds link these units into a three-dimensional network.

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Comment

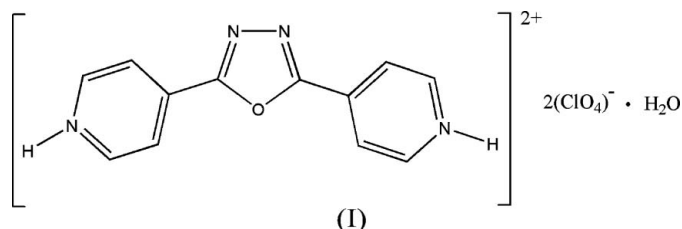
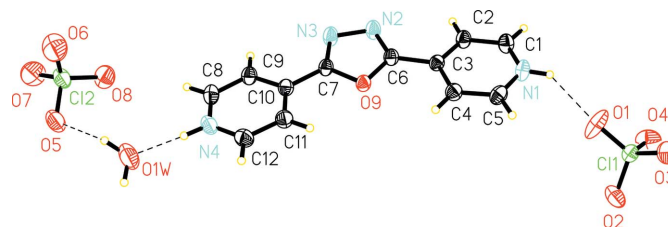
2,5-Di-4-pyridyl-1,3,4-oxadiazole (bpo) is a heterocycle having several N-donor sites and it has afforded a large number of metal complexes, as noted from the Cambridge Structural Database (Conquest Version 1.8, 2005; Allen, 2002). Its crystal structure has been reported (Cheng *et al.*, 2005; Stockhause *et al.*, 2001) but curiously, other organic derivatives have not been crystallographically examined. It yields a diperchlorate salt as a monohydrate, (I), when treated with perchloric acid.The asymmetric unit of (I) consists of one bpo dication, two perchlorate anions and one water molecule, linked by hydrogen bonds (Fig. 1). The bond lengths and angles are in normal ranges (Allen *et al.*, 1987).In the dication, rings *A* (N1/C1–C5), *B* (C6/N2/N3/C7/O9) and *C* (N4/C8–C12) are each planar. The dihedral angles between the rings are $A/B = 7.4$ (1), $A/C = 4.1$ (2) and $B/C = 9.5$ (2)°.In the crystal structure, a three-dimensional network is formed *via* $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds (Table 1 and Fig. 2).

Figure 1

The asymmetric unit of the title structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

Experimental

A mixture of 2,5-bis(4-pyridyl)-1,3,4-oxadiazole (22 mg, 0.1 mmol) (Dong *et al.*, 2002) and perchloric acid (1 ml) was recrystallized from methanol and water (1:1) (yield 31 mg, 70%; m.p. 483–484 K), from which a colourless plate-shaped crystal suitable for X-ray diffraction was selected. Analysis, found: C 32.1, H 2.72, N 12.57%; calculated: C 32.52, H 2.73, N 12.64%.

Crystal data

$C_{12}H_{10}N_4O^{2+} \cdot 2ClO_4^- \cdot H_2O$	$Z = 4$
$M_r = 443.16$	$D_x = 1.698 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 5.4662$ (5) Å	$\mu = 0.44 \text{ mm}^{-1}$
$b = 19.448$ (2) Å	$T = 295$ (2) K
$c = 16.383$ (1) Å	Plate, colourless
$\beta = 95.609$ (1)°	$0.47 \times 0.35 \times 0.04 \text{ mm}$
$V = 1733.2$ (3) Å ³	

Data collection

Bruker APEX area-detector diffractometer	3882 independent reflections
φ and ω scans	2879 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.032$
10264 measured reflections	$\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0819P)^2 + 0.8074P]$
$R[F^2 > 2\sigma(F^2)] = 0.059$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.165$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
3882 reflections	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$
332 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1N \cdots O1$	0.84 (1)	2.14 (3)	2.84 (1)	140 (4)
$N1-H1N \cdots O1'$	0.84 (1)	2.34 (4)	3.01 (2)	136 (4)
$N1-H1N \cdots O2^i$	0.84 (1)	2.39 (4)	2.94 (1)	124 (4)
$N1-H1N \cdots O2^{ii}$	0.84 (1)	2.40 (4)	3.00 (2)	128 (4)
$N4-H4N \cdots O1W$	0.85 (1)	1.77 (2)	2.62 (1)	172 (5)
$O1W-H1W1 \cdots O3^{ii}$	0.86	2.39	2.91 (1)	120
$O1W-H1W1 \cdots O3^{iii}$	0.86	2.34	3.11 (2)	148
$O1W-H1W2 \cdots O5$	0.89	2.19	2.79 (1)	124

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

Atoms H1N and H4N were located in a difference synthesis and only their positions were refined [$N-H = 0.84$ (1)– 0.85 (1) Å]. The remaining H atoms were positioned geometrically, with $O-H = 0.86$ and 0.89 Å (for water H) and $C-H = 0.93$ Å for aromatic H, and were constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N,O})$. In the perchlorate anions, the O atoms were refined

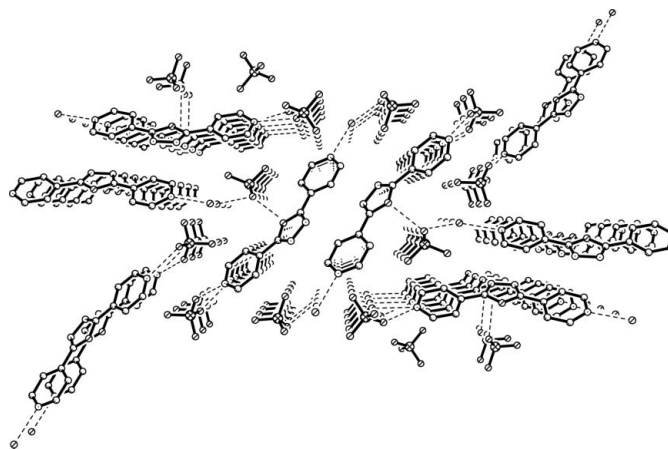


Figure 2

A packing diagram for (I). Hydrogen bonds are shown by dashed lines. H atoms have been omitted.

with statistical disorder over two positions, with partial site-occupancy factors of 0.73 (2) (for O1, O2, O3 and O4), 0.27 (2) (for O1', O2', O3' and O4') and 0.50 (for O5, O6, O7, O8, O5', O6', O7' and O8'). The occupancy could not be refined, unlike that of the other perchlorate, and was arbitrarily fixed as 50:50.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINTE* (Bruker, 2003); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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