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N,*N*′-Dimethyl-*N*,*N*′-bis[2-(2-pyridyl)-ethyl](*p*-phenylenedimethylene)-diammonium bis(perchlorate)

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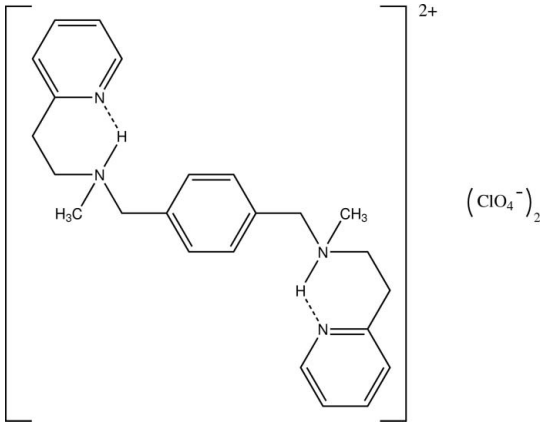
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.008 Å; disorder in solvent or counterion; R factor = 0.059; wR factor = 0.172; data-to-parameter ratio = 11.8.

In the title compound, $C_{24}H_{32}N_4^{2+}\cdot 2ClO_4^-$, the two N atoms of the amine are protonated. The H atoms of the protonated amine form hydrogen bonds with the pyridyl N atoms (intramolecular) and O atoms of adjacent perchlorate anions. In addition, there are weak C—H···O interactions which link the formula units into chains along [101]. Each perchlorate anion is disordered over three conformations, with occupancy factors of 0.482 (2), 0.331 (2) and 0.187 (4), and 0.532 (8), 0.324 (4) and 0.133 (7).

Related literature

For the synthesis, see: Ghosh & Mukherjee (1998). For the monooxygenase activity of the dicopper(I) complexes of the title compound, see: Cruse *et al.* (1988); Karlin *et al.* (1984).



Experimental

Crystal data

$C_{24}H_{32}N_4^{2+}\cdot 2ClO_4^-$
M_r = 575.44
Monoclinic, $P2_1/n$
a = 15.443 (3) Å
b = 11.484 (2) Å
c = 16.046 (3) Å
β = 104.284 (18)°
V = 2757.6 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 296 (2) K
0.45 × 0.34 × 0.12 mm

Data collection

Bruker $P4$ diffractometer
Absorption correction: ψ-scan (North *et al.*, 1968)
T_{min} = 0.794, T_{max} = 0.946
6558 measured reflections
6329 independent reflections
2137 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
3 standard reflections every 97 reflections
intensity decay: <2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.172
S = 0.95
6329 reflections
536 parameters
272 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1*A*—H1*NA*···N2*A*	0.90 (4)	1.95 (4)	2.721 (5)	143 (3)
N1*A*—H1*NA*···O14*A*	0.90 (4)	2.45 (4)	3.056 (11)	125 (3)
N1*A*—H1*NA*···O11*B*	0.90 (4)	2.72 (4)	3.180 (17)	113 (3)
N1*B*—H1*NB*···N2*B*	0.90 (4)	1.90 (4)	2.704 (5)	148 (3)
C5*A*—H5*AA*···O14	0.97	2.10	2.823 (12)	130
C6*A*—H6*AB*···O12^{i}	0.96	2.48	3.40 (2)	160
C6*A*—H6*AC*···O14	0.96	2.50	3.14 (2)	124
C6*A*—H6*AC*···O11*B*	0.96	2.46	3.085 (11)	122
C7*A*—H7*AB*···O13^{i}	0.97	2.42	3.27 (2)	145
C12*B*—H12*A*···O11*A*^{ii}	0.93	2.47	3.266 (8)	143
C4—H4*A*···O14*A*	0.93	2.56	3.406 (14)	151
C5*A*—H5*AB*···O11*B*^{i}	0.97	2.34	3.307 (15)	178
C6*B*—H6*BC*···O13*A*^{iii}	0.96	2.46	3.223 (18)	136
C8*B*—H8*BB*···O13*A*^{iii}	0.97	2.44	3.394 (7)	168
C5*B*—H5*BA*···O22*B*^{iv}	0.97	2.18	2.944 (19)	135
C5*B*—H5*BB*···O21	0.97	2.55	3.266 (15)	131
C8*B*—H8*BA*···O22^{v}	0.97	2.53	3.252 (16)	131
C8*B*—H8*BA*···O24*B*^{v}	0.97	2.56	3.51 (3)	167
C10*B*—H10*A*···O21*B*^{v}	0.93	2.37	3.25 (2)	158
C6*B*—H6*BA*···O23*B*	0.96	1.93	2.578 (15)	123
C6*B*—H6*BB*···O24*B*^{iv}	0.96	2.52	3.44 (3)	161
C8*A*—H8*AA*···O24^{vi}	0.97	2.29	3.146 (13)	147

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

Data collection: *XSCANS* (Bruker, 1997); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2378).

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supplementary materials

Acta Cryst. (2007). E**63**, o4891-o4892 [**doi:10.1107/S1600536807060576**]

N,*N*'-Dimethyl-*N*,*N*'-bis[2-(2-pyridyl)ethyl](*p*-phenylenedimethylene)diammonium bis(perchlorate)

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Comment

The stucture of the title compound, (I), is shown below. Dimensions are available in the archived CIF.

In the title compound $C_{24}H_{32}N_4^{2+}\cdot 2ClO_4^-$ (I), the two nitrogen atoms of the amine are protonated. The nitrogen atoms of the pyridyl ring are intramolecularly hydrogen bonded to the protonating protons of the amine as well as perchlorate oxygen atoms. In addition there are weak C—H···O interactions which link the formula units into chains in the 1 0 1 direction.

The *m*-xylyl-based dinucleating ligand, α,α'-bis[*N*-methyl-*N*-(2-pyridylethyl)amino]-*m*-xylene, had been synthesized and used to make dicopper(I) complexes in modeling the tyrosinase monooxygenase active site (Ghosh & Mukherjee, 1998). Several ligands consisting of a *m*-xylyl spacer bridging two tridentate donors have been used to synthesize dinuclear copper complexes and the monooxygenase activities of these complexes has been reported extensively (Karlin *et al.*, 1984; Cruse *et al.*, 1988).

Experimental

The neutral ligand α,α'-bis[*N*-methyl-*N*-(2-pyridylethyl)amino]-*m*-xylene, ligand was synthesized according to a literature method of Ghosh & Mukherjee (1998). The perchlorate salt of the protonated ligand (1) was formed as a side product of the reaction for the synthesis of a Ni(II) complex by the reaction of the neutral ligand with Ni(ClO4)2.6H2O in methanol. Slow evaporation of the methanol solution leads to the isolation of crystals of 1 suited for crystallographic structure determination.

Refinement

Each bridging perchlorates is disordered is disordered over three comformations with occupancy factors of 0.482 (2), 0.331 (2), 0.187 (4) and 0.532 (8), 0.324 (4) 133 (7), respectively. These were idealized to tetrahedral geometry. The N—H atoms were refined isotropically while C—H distances were idealized at 0.93 (aromatic C—H), 0.96 (CH_3), and 0.97 (CH_2) Å and $U_{iso}(H) = 1.2U_{eq}(C)$ ($1.5U_{eq}(C)$ for the CH_3 protons).

Figures

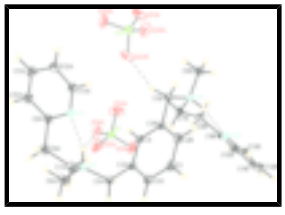


Fig. 1. The title compound with numbering scheme used. Hydrogen bonding interactions shown as dotted lines. Ellipsoids are drawn at the 20% probabilty level.

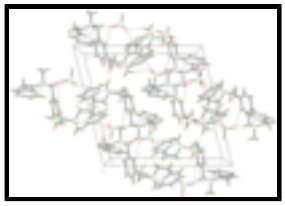


Fig. 2. The packing arrangement viewed down the *b* axis showing the intramolecular O—H···O and intermolecular N—H···O hydrogen bonding interactions (dashed bonds).

N,N'-Dimethyl-N,N'-bis[2-(2-pyridyl)ethyl](*p*-phenylenedimethylene)diammonium bis(perchlorate)

Crystal data

$C_{24}H_{32}N_4^{2+}\cdot 2ClO_4^-$	F_{000} = 1208
M_r = 575.44	D_x = 1.386 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 15.443 (3) Å	Cell parameters from 40 reflections
b = 11.484 (2) Å	θ = 2.2–12.5º
c = 16.046 (3) Å	μ = 0.29 mm^{-1}
β = 104.284 (18)º	T = 296 (2) K
V = 2757.6 (9) Å^3	Plate, colourless
Z = 4	0.45 × 0.34 × 0.12 mm

Data collection

Bruker P4 diffractometer	R_{int} = 0.026
Radiation source: fine-focus sealed tube	θ_{max} = 27.5º
Monochromator: graphite	θ_{min} = 2.1º
T = 296(2) K	h = 0→20
ω scans	k = 0→14
Absorption correction: empirical (using intensity measurements) ψ-scan (North *et al.*, 1968)	l = −20→20
T_{min} = 0.794, T_{max} = 0.946	3 standard reflections
6558 measured reflections	every 97 reflections
6329 independent reflections	intensity decay: <2%
2137 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.059	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.172	$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.95	$(\Delta/\sigma)_{max}$ = 0.009

6329 reflections	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
536 parameters	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
272 restraints	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0028 (5)

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
Cl1	0.71817 (9)	0.42115 (10)	0.40926 (8)	0.0785 (4)	
Cl2	0.11481 (9)	0.20277 (12)	0.24929 (9)	0.0873 (4)	
O11	0.6909 (17)	0.375 (3)	0.4785 (11)	0.173 (13)	0.187 (4)
O12	0.6568 (14)	0.5038 (18)	0.3686 (18)	0.142 (15)	0.187 (4)
O13	0.8008 (9)	0.473 (2)	0.4383 (16)	0.18 (2)	0.187 (4)
O14	0.7234 (19)	0.333 (2)	0.3520 (15)	0.192 (13)	0.187 (4)
O11A	0.6854 (9)	0.3173 (8)	0.4315 (8)	0.152 (9)	0.482 (19)
O12A	0.8093 (5)	0.4285 (16)	0.4438 (13)	0.162 (8)	0.482 (19)
O13A	0.6763 (15)	0.5138 (10)	0.4377 (13)	0.191 (8)	0.482 (19)
O14A	0.7017 (13)	0.4277 (12)	0.3199 (3)	0.120 (5)	0.482 (19)
O11B	0.7525 (15)	0.4199 (14)	0.3371 (10)	0.139 (9)	0.331 (19)
O12B	0.7804 (13)	0.4657 (14)	0.4784 (8)	0.148 (9)	0.331 (19)
O13B	0.6954 (11)	0.3085 (7)	0.4262 (9)	0.110 (9)	0.331 (19)
O14B	0.6422 (8)	0.4903 (14)	0.3925 (15)	0.117 (8)	0.331 (19)
O21	0.1794 (11)	0.2473 (18)	0.2104 (13)	0.183 (11)	0.334 (4)
O22	0.0568 (10)	0.1299 (12)	0.1899 (10)	0.138 (10)	0.334 (4)
O23	0.0659 (12)	0.2936 (12)	0.2724 (12)	0.188 (12)	0.334 (4)
O24	0.1558 (13)	0.1375 (13)	0.3213 (7)	0.170 (11)	0.334 (4)
O21A	0.1418 (6)	0.2731 (9)	0.1895 (5)	0.137 (5)	0.532 (8)
O22A	0.0880 (8)	0.2722 (8)	0.3099 (6)	0.162 (6)	0.532 (8)
O23A	0.0431 (6)	0.1323 (9)	0.2071 (6)	0.142 (7)	0.532 (8)
O24A	0.1859 (6)	0.1324 (8)	0.2915 (6)	0.123 (4)	0.532 (8)
O21B	0.0276 (8)	0.246 (2)	0.2331 (18)	0.098 (10)	0.133 (7)
O22B	0.1181 (19)	0.0929 (11)	0.2870 (16)	0.088 (10)	0.133 (7)
O23B	0.1721 (18)	0.278 (2)	0.3061 (16)	0.193 (17)	0.133 (7)
O24B	0.141 (2)	0.196 (3)	0.1725 (11)	0.159 (16)	0.133 (7)

N1A	0.6971 (2)	0.2649 (3)	0.1681 (2)	0.0652 (10)
H1NA	0.674 (2)	0.334 (3)	0.178 (2)	0.068 (13)*
N2A	0.6175 (2)	0.4745 (3)	0.1216 (2)	0.0732 (10)
N1B	0.3724 (2)	0.3260 (3)	0.3469 (2)	0.0671 (10)
H1NB	0.354 (2)	0.254 (3)	0.356 (2)	0.065 (13)*
N2B	0.3710 (2)	0.1169 (3)	0.4240 (2)	0.0677 (9)
C1	0.4331 (4)	0.0575 (5)	0.1654 (3)	0.0996 (16)
H1A	0.4017	−0.0086	0.1416	0.118 (18)*
C2A	0.5233 (4)	0.0640 (4)	0.1725 (3)	0.0856 (14)
H2AA	0.5528	0.0023	0.1540	0.088 (14)*
C3A	0.5704 (3)	0.1621 (4)	0.2071 (2)	0.0643 (11)
C4	0.5241 (3)	0.2525 (3)	0.2343 (2)	0.0598 (10)
H4A	0.5551	0.3191	0.2576	0.065 (11)*
C5A	0.6696 (3)	0.1685 (4)	0.2188 (3)	0.0707 (12)
H5AA	0.6979	0.1799	0.2793	0.086 (14)*
H5AB	0.6907	0.0951	0.2013	0.099 (15)*
C6A	0.7963 (3)	0.2768 (5)	0.1900 (3)	0.0903 (15)
H6AA	0.8124	0.3450	0.1626	0.14 (2)*
H6AB	0.8220	0.2093	0.1703	0.15 (2)*
H6AC	0.8183	0.2838	0.2512	0.113 (18)*
C7A	0.6590 (3)	0.2513 (4)	0.0731 (3)	0.0769 (13)
H7AA	0.5952	0.2373	0.0627	0.070 (12)*
H7AB	0.6856	0.1833	0.0536	0.096 (15)*
C8A	0.6737 (3)	0.3546 (4)	0.0208 (3)	0.0823 (13)
H8AA	0.6468	0.3385	−0.0394	0.091 (14)*
H8AB	0.7374	0.3641	0.0273	0.085 (14)*
C9A	0.6363 (3)	0.4672 (4)	0.0445 (3)	0.0695 (12)
C10A	0.6229 (3)	0.5617 (5)	−0.0115 (3)	0.0883 (15)
H10B	0.6352	0.5555	−0.0652	0.13 (2)*
C11A	0.5917 (3)	0.6633 (6)	0.0133 (4)	0.0985 (16)
H11B	0.5848	0.7279	−0.0226	0.104 (17)*
C12A	0.5704 (3)	0.6708 (5)	0.0913 (4)	0.0926 (15)
H12B	0.5474	0.7389	0.1088	0.105 (18)*
C13A	0.5844 (3)	0.5733 (5)	0.1425 (3)	0.0831 (13)
H13A	0.5697	0.5771	0.1953	0.070 (13)*
C2B	0.3888 (3)	0.1470 (5)	0.1929 (3)	0.0866 (14)
H2BA	0.3277	0.1407	0.1881	0.101 (16)*
C3B	0.4331 (3)	0.2459 (3)	0.2276 (2)	0.0619 (11)
C5B	0.3845 (3)	0.3443 (4)	0.2585 (3)	0.0726 (12)
H5BA	0.4176	0.4159	0.2576	0.071 (12)*
H5BB	0.3263	0.3535	0.2189	0.085 (14)*
C6B	0.3074 (4)	0.4120 (5)	0.3653 (4)	0.0983 (16)
H6BA	0.2530	0.4085	0.3208	0.15 (3)*
H6BB	0.3323	0.4888	0.3675	0.16 (3)*
H6BC	0.2950	0.3939	0.4197	0.084 (14)*
C7B	0.4594 (3)	0.3270 (4)	0.4134 (3)	0.0741 (12)
H7BA	0.4818	0.4062	0.4210	0.116 (18)*
H7BB	0.5025	0.2806	0.3930	0.091 (14)*
C8B	0.4516 (3)	0.2795 (4)	0.4998 (3)	0.0770 (13)

H8BA	0.5097	0.2848	0.5402	0.081 (13)*
H8BB	0.4109	0.3289	0.5212	0.104 (16)*
C9B	0.4194 (3)	0.1558 (4)	0.4983 (3)	0.0636 (11)
C10B	0.4389 (3)	0.0866 (5)	0.5711 (3)	0.0786 (13)
H10A	0.4731	0.1155	0.6230	0.108 (18)*
C11B	0.4070 (4)	−0.0249 (6)	0.5655 (4)	0.0921 (16)
H11A	0.4198	−0.0724	0.6140	0.113 (18)*
C12B	0.3565 (3)	−0.0673 (5)	0.4895 (4)	0.0907 (15)
H12A	0.3337	−0.1426	0.4845	0.14 (2)*
C13B	0.3414 (3)	0.0077 (4)	0.4209 (3)	0.0764 (12)
H13B	0.3079	−0.0197	0.3682	0.071 (13)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0866 (9)	0.0714 (9)	0.0784 (9)	−0.0058 (8)	0.0220 (7)	0.0007 (7)
Cl2	0.0897 (10)	0.0810 (9)	0.0872 (10)	0.0031 (8)	0.0141 (8)	0.0132 (8)
O11	0.29 (3)	0.13 (2)	0.14 (2)	−0.08 (2)	0.12 (2)	−0.01 (2)
O12	0.18 (3)	0.12 (2)	0.14 (3)	0.03 (2)	0.05 (2)	0.07 (2)
O13	0.16 (3)	0.13 (2)	0.17 (3)	−0.13 (2)	−0.09 (2)	0.12 (2)
O14	0.19 (2)	0.15 (2)	0.22 (3)	0.06 (2)	0.03 (2)	−0.12 (2)
O11A	0.121 (11)	0.117 (16)	0.22 (2)	−0.046 (11)	0.043 (12)	0.043 (14)
O12A	0.120 (10)	0.164 (13)	0.162 (14)	−0.027 (8)	−0.041 (9)	0.086 (11)
O13A	0.32 (2)	0.131 (9)	0.137 (12)	0.091 (11)	0.090 (13)	−0.060 (9)
O14A	0.177 (13)	0.140 (10)	0.043 (5)	−0.081 (9)	0.027 (5)	−0.022 (5)
O11B	0.205 (19)	0.096 (11)	0.171 (18)	−0.058 (13)	0.151 (16)	−0.044 (12)
O12B	0.158 (16)	0.173 (18)	0.097 (11)	−0.052 (15)	0.004 (11)	−0.074 (13)
O13B	0.19 (2)	0.070 (14)	0.072 (11)	−0.023 (13)	0.034 (12)	0.044 (10)
O14B	0.119 (11)	0.150 (16)	0.087 (13)	0.067 (10)	0.036 (10)	−0.005 (10)
O21	0.108 (14)	0.23 (2)	0.23 (2)	−0.044 (14)	0.074 (15)	−0.02 (2)
O22	0.19 (2)	0.093 (13)	0.078 (9)	−0.009 (12)	−0.063 (12)	−0.032 (9)
O23	0.207 (19)	0.103 (13)	0.22 (2)	0.053 (13)	−0.006 (17)	−0.080 (14)
O24	0.26 (2)	0.19 (2)	0.034 (7)	0.072 (18)	−0.002 (11)	0.004 (9)
O21A	0.090 (7)	0.189 (11)	0.120 (7)	−0.047 (7)	0.001 (6)	0.085 (7)
O22A	0.223 (14)	0.102 (8)	0.212 (12)	0.039 (8)	0.150 (11)	0.002 (8)
O23A	0.095 (8)	0.175 (15)	0.151 (11)	−0.045 (9)	0.021 (8)	0.042 (10)
O24A	0.126 (7)	0.132 (8)	0.084 (7)	0.060 (6)	−0.024 (6)	0.001 (6)
O21B	0.091 (18)	0.09 (2)	0.10 (2)	0.029 (17)	0.014 (15)	−0.019 (17)
O22B	0.12 (2)	0.032 (12)	0.11 (2)	0.007 (14)	0.03 (2)	0.009 (14)
O23B	0.19 (3)	0.12 (2)	0.24 (3)	−0.01 (3)	0.01 (3)	−0.09 (3)
O24B	0.19 (3)	0.15 (3)	0.17 (3)	−0.04 (3)	0.11 (3)	0.03 (3)
N1A	0.064 (2)	0.071 (3)	0.065 (2)	0.005 (2)	0.0233 (18)	0.003 (2)
N2A	0.083 (2)	0.075 (3)	0.065 (2)	−0.004 (2)	0.0262 (19)	0.001 (2)
N1B	0.068 (2)	0.063 (3)	0.077 (3)	0.0106 (19)	0.030 (2)	0.010 (2)
N2B	0.065 (2)	0.066 (2)	0.071 (2)	−0.0051 (18)	0.0161 (19)	0.0024 (19)
C1	0.111 (4)	0.077 (4)	0.121 (4)	−0.030 (3)	0.047 (3)	−0.022 (3)
C2A	0.106 (4)	0.058 (3)	0.107 (4)	−0.009 (3)	0.053 (3)	−0.014 (3)
C3A	0.080 (3)	0.055 (3)	0.066 (3)	0.001 (2)	0.032 (2)	0.005 (2)

C4	0.076 (3)	0.047 (2)	0.060 (2)	−0.007 (2)	0.024 (2)	0.000 (2)
C5A	0.083 (3)	0.064 (3)	0.072 (3)	0.009 (2)	0.033 (2)	0.010 (2)
C6A	0.062 (3)	0.120 (5)	0.089 (4)	0.003 (3)	0.020 (3)	0.006 (3)
C7A	0.078 (3)	0.090 (4)	0.067 (3)	−0.001 (3)	0.027 (2)	−0.003 (3)
C8A	0.084 (4)	0.106 (4)	0.063 (3)	0.006 (3)	0.028 (3)	0.009 (3)
C9A	0.054 (2)	0.084 (3)	0.069 (3)	−0.005 (2)	0.013 (2)	0.011 (3)
C10A	0.078 (3)	0.105 (4)	0.082 (4)	0.001 (3)	0.018 (3)	0.020 (3)
C11A	0.084 (4)	0.096 (5)	0.108 (5)	0.001 (3)	0.009 (3)	0.028 (4)
C12A	0.084 (3)	0.079 (4)	0.104 (4)	−0.001 (3)	0.004 (3)	−0.006 (4)
C13A	0.096 (4)	0.074 (3)	0.081 (3)	−0.002 (3)	0.026 (3)	−0.006 (3)
C2B	0.079 (4)	0.089 (4)	0.098 (4)	−0.018 (3)	0.034 (3)	−0.002 (3)
C3B	0.069 (3)	0.060 (3)	0.062 (3)	−0.002 (2)	0.027 (2)	0.007 (2)
C5B	0.077 (3)	0.072 (3)	0.077 (3)	0.014 (3)	0.034 (3)	0.020 (2)
C6B	0.106 (4)	0.100 (5)	0.103 (4)	0.038 (3)	0.054 (4)	0.007 (3)
C7B	0.070 (3)	0.074 (3)	0.081 (3)	−0.006 (3)	0.022 (3)	−0.005 (2)
C8B	0.070 (3)	0.089 (4)	0.070 (3)	0.000 (3)	0.012 (2)	−0.015 (3)
C9B	0.054 (2)	0.075 (3)	0.064 (3)	0.006 (2)	0.020 (2)	0.002 (2)
C10B	0.076 (3)	0.102 (4)	0.061 (3)	0.026 (3)	0.023 (3)	0.012 (3)
C11B	0.086 (4)	0.101 (4)	0.102 (4)	0.028 (3)	0.046 (3)	0.041 (4)
C12B	0.080 (3)	0.076 (4)	0.124 (5)	0.010 (3)	0.039 (3)	0.026 (4)
C13B	0.068 (3)	0.072 (3)	0.087 (4)	−0.005 (3)	0.016 (3)	0.002 (3)

Geometric parameters (Å, °)

Cl1—O12B	1.375 (5)	C4—C3B	1.385 (5)
Cl1—O11A	1.377 (5)	C4—H4A	0.9300
Cl1—O14	1.379 (5)	C5A—H5AA	0.9700
Cl1—O13A	1.380 (5)	C5A—H5AB	0.9700
Cl1—O12A	1.382 (5)	C6A—H6AA	0.9600
Cl1—O13	1.382 (5)	C6A—H6AB	0.9600
Cl1—O13B	1.385 (5)	C6A—H6AC	0.9600
Cl1—O12	1.386 (5)	C7A—C8A	1.503 (5)
Cl1—O11	1.386 (5)	C7A—H7AA	0.9700
Cl1—O11B	1.387 (5)	C7A—H7AB	0.9700
Cl1—O14B	1.387 (5)	C8A—C9A	1.503 (6)
Cl1—O14A	1.396 (5)	C8A—H8AA	0.9700
Cl2—O23	1.391 (5)	C8A—H8AB	0.9700
Cl2—O24	1.392 (5)	C9A—C10A	1.391 (6)
Cl2—O24B	1.393 (5)	C10A—C11A	1.358 (7)
Cl2—O21A	1.393 (5)	C10A—H10B	0.9300
Cl2—O22B	1.394 (5)	C11A—C12A	1.373 (7)
Cl2—O22A	1.397 (5)	C11A—H11B	0.9300
Cl2—O24A	1.397 (5)	C12A—C13A	1.374 (6)
Cl2—O21	1.397 (5)	C12A—H12B	0.9300
Cl2—O21B	1.400 (5)	C13A—H13A	0.9300
Cl2—O23B	1.403 (5)	C2B—C3B	1.372 (6)
Cl2—O23A	1.404 (4)	C2B—H2BA	0.9300
Cl2—O22	1.411 (5)	C3B—C5B	1.506 (5)
N1A—C6A	1.491 (5)	C5B—H5BA	0.9700

N1A—C5A	1.496 (5)	C5B—H5BB	0.9700
N1A—C7A	1.500 (5)	C6B—H6BA	0.9600
N1A—H1NA	0.90 (4)	C6B—H6BB	0.9600
N2A—C13A	1.321 (5)	C6B—H6BC	0.9600
N2A—C9A	1.342 (5)	C7B—C8B	1.523 (5)
N1B—C6B	1.489 (5)	C7B—H7BA	0.9700
N1B—C5B	1.491 (5)	C7B—H7BB	0.9700
N1B—C7B	1.496 (5)	C8B—C9B	1.503 (6)
N1B—H1NB	0.90 (4)	C8B—H8BA	0.9700
N2B—C9B	1.319 (5)	C8B—H8BB	0.9700
N2B—C13B	1.331 (5)	C9B—C10B	1.384 (5)
C1—C2B	1.367 (6)	C10B—C11B	1.368 (6)
C1—C2A	1.372 (6)	C10B—H10A	0.9300
C1—H1A	0.9300	C11B—C12B	1.366 (7)
C2A—C3A	1.381 (5)	C11B—H11A	0.9300
C2A—H2AA	0.9300	C12B—C13B	1.372 (6)
C3A—C4	1.390 (5)	C12B—H12A	0.9300
C3A—C5A	1.499 (5)	C13B—H13B	0.9300
O11A—Cl1—O13A	110.5 (2)	H6AB—C6A—H6AC	109.5
O11A—Cl1—O12A	110.6 (2)	N1A—C7A—C8A	114.2 (4)
O13A—Cl1—O12A	109.5 (2)	N1A—C7A—H7AA	108.7
O14—Cl1—O13	109.8 (3)	C8A—C7A—H7AA	108.7
O12B—Cl1—O13B	110.5 (2)	N1A—C7A—H7AB	108.7
O14—Cl1—O12	109.4 (3)	C8A—C7A—H7AB	108.7
O13—Cl1—O12	109.3 (3)	H7AA—C7A—H7AB	107.6
O14—Cl1—O11	109.6 (3)	C7A—C8A—C9A	114.6 (4)
O13—Cl1—O11	109.4 (3)	C7A—C8A—H8AA	108.6
O12—Cl1—O11	109.2 (3)	C9A—C8A—H8AA	108.6
O12B—Cl1—O11B	110.0 (2)	C7A—C8A—H8AB	108.6
O13B—Cl1—O11B	108.8 (3)	C9A—C8A—H8AB	108.6
O12B—Cl1—O14B	109.4 (3)	H8AA—C8A—H8AB	107.6
O13B—Cl1—O14B	109.2 (3)	N2A—C9A—C10A	121.0 (5)
O11B—Cl1—O14B	108.9 (3)	N2A—C9A—C8A	118.1 (4)
O11A—Cl1—O14A	109.1 (2)	C10A—C9A—C8A	120.9 (4)
O13A—Cl1—O14A	108.5 (2)	C11A—C10A—C9A	119.2 (5)
O12A—Cl1—O14A	108.5 (2)	C11A—C10A—H10B	120.4
O23—Cl2—O24	110.3 (3)	C9A—C10A—H10B	120.4
O24B—Cl2—O22B	110.2 (3)	C10A—C11A—C12A	120.2 (6)
O21A—Cl2—O22A	109.8 (2)	C10A—C11A—H11B	119.9
O21A—Cl2—O24A	109.6 (2)	C12A—C11A—H11B	119.9
O22A—Cl2—O24A	109.0 (2)	C11A—C12A—C13A	117.1 (6)
O23—Cl2—O21	109.8 (3)	C11A—C12A—H12B	121.4
O24—Cl2—O21	109.8 (3)	C13A—C12A—H12B	121.4
O24B—Cl2—O21B	109.7 (3)	N2A—C13A—C12A	124.2 (5)
O22B—Cl2—O21B	109.5 (3)	N2A—C13A—H13A	117.9
O24B—Cl2—O23B	109.5 (3)	C12A—C13A—H13A	117.9
O22B—Cl2—O23B	109.1 (3)	C1—C2B—C3B	121.0 (5)
O21B—Cl2—O23B	108.9 (3)	C1—C2B—H2BA	119.5
O21A—Cl2—O23A	109.6 (2)	C3B—C2B—H2BA	119.5

O22A—Cl2—O23A	109.3 (2)	C2B—C3B—C4	118.1 (4)
O24A—Cl2—O23A	109.4 (2)	C2B—C3B—C5B	121.0 (4)
O23—Cl2—O22	109.2 (2)	C4—C3B—C5B	120.8 (4)
O24—Cl2—O22	108.9 (2)	N1B—C5B—C3B	113.4 (3)
O21—Cl2—O22	108.7 (3)	N1B—C5B—H5BA	108.9
C6A—N1A—C5A	110.6 (4)	C3B—C5B—H5BA	108.9
C6A—N1A—C7A	111.9 (3)	N1B—C5B—H5BB	108.9
C5A—N1A—C7A	112.5 (3)	C3B—C5B—H5BB	108.9
C6A—N1A—H1NA	107 (2)	H5BA—C5B—H5BB	107.7
C5A—N1A—H1NA	112 (2)	N1B—C6B—H6BA	109.5
C7A—N1A—H1NA	102 (2)	N1B—C6B—H6BB	109.5
C13A—N2A—C9A	118.2 (4)	H6BA—C6B—H6BB	109.5
C6B—N1B—C5B	110.2 (3)	N1B—C6B—H6BC	109.5
C6B—N1B—C7B	112.6 (4)	H6BA—C6B—H6BC	109.5
C5B—N1B—C7B	112.1 (3)	H6BB—C6B—H6BC	109.5
C6B—N1B—H1NB	109 (2)	N1B—C7B—C8B	112.8 (4)
C5B—N1B—H1NB	113 (2)	N1B—C7B—H7BA	109.0
C7B—N1B—H1NB	99 (2)	C8B—C7B—H7BA	109.0
C9B—N2B—C13B	117.8 (4)	N1B—C7B—H7BB	109.0
C2B—C1—C2A	120.7 (5)	C8B—C7B—H7BB	109.0
C2B—C1—H1A	119.6	H7BA—C7B—H7BB	107.8
C2A—C1—H1A	119.6	C9B—C8B—C7B	115.0 (4)
C1—C2A—C3A	120.1 (5)	C9B—C8B—H8BA	108.5
C1—C2A—H2AA	120.0	C7B—C8B—H8BA	108.5
C3A—C2A—H2AA	120.0	C9B—C8B—H8BB	108.5
C2A—C3A—C4	118.3 (4)	C7B—C8B—H8BB	108.5
C2A—C3A—C5A	120.7 (4)	H8BA—C8B—H8BB	107.5
C4—C3A—C5A	120.9 (4)	N2B—C9B—C10B	121.7 (4)
C3B—C4—C3A	121.7 (4)	N2B—C9B—C8B	116.5 (4)
C3B—C4—H4A	119.1	C10B—C9B—C8B	121.7 (4)
C3A—C4—H4A	119.1	C11B—C10B—C9B	118.8 (5)
N1A—C5A—C3A	112.8 (3)	C11B—C10B—H10A	120.6
N1A—C5A—H5AA	109.0	C9B—C10B—H10A	120.6
C3A—C5A—H5AA	109.0	C12B—C11B—C10B	120.6 (5)
N1A—C5A—H5AB	109.0	C12B—C11B—H11A	119.7
C3A—C5A—H5AB	109.0	C10B—C11B—H11A	119.7
H5AA—C5A—H5AB	107.8	C11B—C12B—C13B	116.1 (5)
N1A—C6A—H6AA	109.5	C11B—C12B—H12A	122.0
N1A—C6A—H6AB	109.5	C13B—C12B—H12A	122.0
H6AA—C6A—H6AB	109.5	N2B—C13B—C12B	124.9 (5)
N1A—C6A—H6AC	109.5	N2B—C13B—H13B	117.5
H6AA—C6A—H6AC	109.5	C12B—C13B—H13B	117.5
C2B—C1—C2A—C3A	−0.5 (8)	C2A—C1—C2B—C3B	0.6 (8)
C1—C2A—C3A—C4	0.1 (7)	C1—C2B—C3B—C4	−0.3 (7)
C1—C2A—C3A—C5A	177.4 (4)	C1—C2B—C3B—C5B	−179.7 (4)
C2A—C3A—C4—C3B	0.2 (6)	C3A—C4—C3B—C2B	−0.1 (6)
C5A—C3A—C4—C3B	−177.1 (3)	C3A—C4—C3B—C5B	179.3 (3)
C6A—N1A—C5A—C3A	173.1 (4)	C6B—N1B—C5B—C3B	−167.5 (4)
C7A—N1A—C5A—C3A	−61.0 (5)	C7B—N1B—C5B—C3B	66.3 (5)

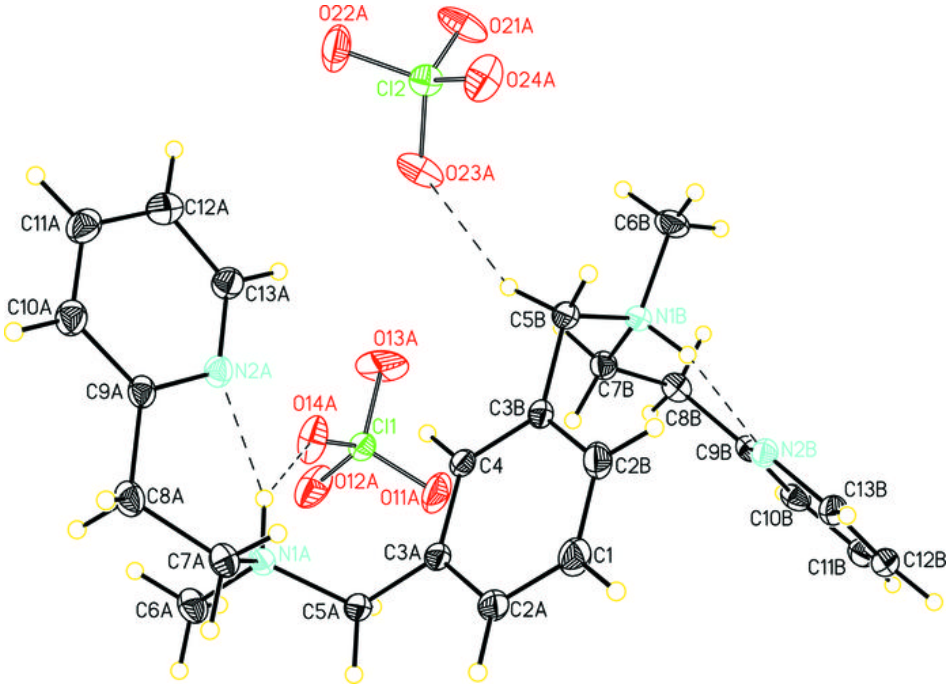
C2A—C3A—C5A—N1A	118.5 (4)	C2B—C3B—C5B—N1B	82.5 (5)
C4—C3A—C5A—N1A	−64.3 (5)	C4—C3B—C5B—N1B	−96.9 (5)
C6A—N1A—C7A—C8A	−63.2 (5)	C6B—N1B—C7B—C8B	69.2 (5)
C5A—N1A—C7A—C8A	171.6 (4)	C5B—N1B—C7B—C8B	−165.9 (4)
N1A—C7A—C8A—C9A	−58.1 (5)	N1B—C7B—C8B—C9B	60.0 (5)
C13A—N2A—C9A—C10A	1.3 (6)	C13B—N2B—C9B—C10B	−0.3 (6)
C13A—N2A—C9A—C8A	−179.8 (4)	C13B—N2B—C9B—C8B	−179.8 (4)
C7A—C8A—C9A—N2A	18.6 (6)	C7B—C8B—C9B—N2B	−24.7 (5)
C7A—C8A—C9A—C10A	−162.5 (4)	C7B—C8B—C9B—C10B	155.8 (4)
N2A—C9A—C10A—C11A	1.0 (7)	N2B—C9B—C10B—C11B	0.0 (6)
C8A—C9A—C10A—C11A	−177.9 (4)	C8B—C9B—C10B—C11B	179.5 (4)
C9A—C10A—C11A—C12A	−2.5 (7)	C9B—C10B—C11B—C12B	−0.2 (7)
C10A—C11A—C12A—C13A	1.7 (7)	C10B—C11B—C12B—C13B	0.6 (7)
C9A—N2A—C13A—C12A	−2.1 (7)	C9B—N2B—C13B—C12B	0.8 (6)
C11A—C12A—C13A—N2A	0.6 (7)	C11B—C12B—C13B—N2B	−1.0 (7)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1A—H1NA···N2A	0.90 (4)	1.95 (4)	2.721 (5)	143 (3)
N1A—H1NA···O14A	0.90 (4)	2.45 (4)	3.056 (11)	125 (3)
N1A—H1NA···O11B	0.90 (4)	2.72 (4)	3.180 (17)	113 (3)
N1B—H1NB···N2B	0.90 (4)	1.90 (4)	2.704 (5)	148 (3)
C5A—H5AA···O14	0.97	2.10	2.823 (12)	130
C6A—H6AB···O12^{i}	0.96	2.48	3.40 (2)	160
C6A—H6AC···O14	0.96	2.50	3.14 (2)	124
C6A—H6AC···O11B	0.96	2.46	3.085 (11)	122
C7A—H7AB···O13^{i}	0.97	2.42	3.27 (2)	145
C12B—H12A···O11A^{ii}	0.93	2.47	3.266 (8)	143
C4—H4A···O14A	0.93	2.56	3.406 (14)	151
C5A—H5AB···O11B^{i}	0.97	2.34	3.307 (15)	178
C6B—H6BC···O13A^{iii}	0.96	2.46	3.223 (18)	136
C8B—H8BB···O13A^{iii}	0.97	2.44	3.394 (7)	168
C5B—H5BA···O22B^{iv}	0.97	2.18	2.944 (19)	135
C5B—H5BB···O21	0.97	2.55	3.266 (15)	131
C8B—H8BA···O22^{v}	0.97	2.53	3.252 (16)	131
C8B—H8BA···O24B^{v}	0.97	2.56	3.51 (3)	167
C10B—H10A···O21B^{v}	0.93	2.37	3.25 (2)	158
C6B—H6BA···O23B	0.96	1.93	2.578 (15)	123
C6B—H6BB···O24B^{iv}	0.96	2.52	3.44 (3)	161
C8A—H8AA···O24^{vi}	0.97	2.29	3.146 (13)	147

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

Fig. 1


O22A
O21A
Cl2
O24A
O23A
C6B
C11A
C12A
C13A
C10A
C5B
N1B
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N2A
C9A
C7B
C3B
C8B
O14A
Cl1
C9B
N2B
C4
C2B
C13B
C8A
O12A
O11A
C10B
C7A
N1A
C3A
C1
C11B
C12B
C6A
C5A
C2A

Fig. 2