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# *N*-Methyl-*N*-(2-pyridiniomethyl)-2-[*N*-(2-pyridiniomethyl)methylamino]-*N*-(2-pyridylmethyl)ethanaminium tris(perchlorate)

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The crystal structure of the title compound, $C_{22}H_{30}N_5^{3+}\cdot 3ClO_4^-$, at 180 K contains intramolecular N—H···N and intermolecular N—H···O hydrogen bonds.

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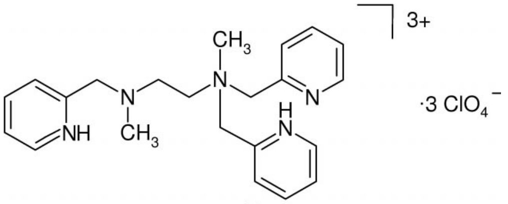
**Key indicators**

Single-crystal X-ray study
*T* = 180 K
Mean $\sigma$(C–C) = 0.004 Å
*R* factor = 0.056
*wR* factor = 0.154
Data-to-parameter ratio = 14.9

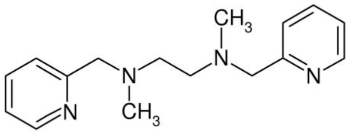
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

## Comment

We have used the tetradentate $N_4$ capping ligand 'mebispicen' [*N*,*N*′-dimethyl-*N*,*N*′-bis(2-pyridylmethyl)ethane-1,2-diamine] on numerous occasions to prepare mononuclear and dinuclear transition metal complexes [for examples, see Hazell *et al.* (1995), Glerup *et al.* (1994) and Egdal *et al.* (2003)]. From a reaction mixture incorporating mebispicen and manganese(II), single crystals of the title compound, (I), were isolated. The formation of a quaternary ammonium centre by addition of a third 2-pyridylmethyl group side-arm is not unprecedented; we have previously observed similar reactivity for the pentadentate $N_5$ ligand *N*′-methyl-*N*,*N*,*N*′-tris(2-pyridylmethyl)ethane-1,2-diamine in the presence of iron(III) (Jensen *et al.*, 1997).



(I)



**mebiscipen**

In the solid state at 180 K, the organic cation of the title compound (Fig. 1) exhibits an intramolecular N—H···N hydrogen bond between the N atoms of the 2-pyridylmethyl pendant arms bound to the quaternary N atom (Table 1). The pyridyl ring of the 2-pyridylmethyl group bound to the tertiary N atom is also protonated and forms intermolecular N—H···O hydrogen bonds (bifurcated at the donor) to two perchlorate anions (Table 1). The third perchlorate anion in the asymmetric unit is not involved in hydrogen bonding.

## Experimental

The tetradentate $N_4$ ligand 'mebispicen' [*N*,*N*′-dimethyl-*N*,*N*′-bis(2-pyridylmethyl)ethane-1,2-diamine] was prepared according to the method of Toftlund *et al.* (1984). Crystals were obtained from a solution of mebispicen and manganese(II) perchlorate hexahydrate in equimolar quantities in water. The possibility of contamination of the mebispicen starting material was excluded by $^1$H NMR.

### Crystal data

$C_{22}H_{30}N_5^{3+}\cdot 3ClO_4^-$
$M_r = 662.86$
Monoclinic, $P2_1/n$
$a = 13.6004$ (3) Å
$b = 8.7668$ (2) Å
$c = 23.5440$ (5) Å
$\beta = 98.493$ (1)°
$V = 2776.41$ (11) Å$^3$
$Z = 4$
$D_x = 1.586$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 8693 reflections
$\theta = 2.8$–26.4°
$\mu = 0.40$ mm$^{-1}$
$T = 180$ (2) K
Block, colourless
0.25 × 0.20 × 0.20 mm

### Data collection

Bruker–Nonius X8APEX-II CCD diffractometer
Thin-slice $\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.841$, $T_{\max} = 0.924$
55950 measured reflections
5653 independent reflections
4549 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 26.4°$
$h = -16 \rightarrow 16$
$k = -10 \rightarrow 10$
$l = -29 \rightarrow 29$

### Refinement

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.154$
$S = 1.06$
5653 reflections
379 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 5.5837P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.25$ e Å$^{-3}$
$\Delta\rho_{\min} = -1.10$ e Å$^{-3}$

**Table 1**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$B$···O5$^{\rm i}$ | 0.88 | 2.53 | 3.051 (4) | 118 |
| N1—H1$B$···O9$^{\rm i}$ | 0.88 | 2.16 | 2.951 (5) | 149 |
| N4—H4$B$···N5 | 0.88 | 1.88 | 2.718 (4) | 159 |

Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

H atoms bound to C atoms were positioned geometrically and allowed to ride during subsequent refinement, with C—H = 0.95 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for the pyridyl rings, C—H = 0.99 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for the methylene groups, and C—H = 0.98 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for the methyl groups. H atoms bound to pyridyl N atoms were apparent in difference Fourier maps, but were positioned geometrically in the final model and allowed to ride, with N—H = 0.88 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(N). The highest peak and deepest hole are associated with one perchlorate anion (containing Cl3), indicative of some degree of orientational disorder which was not modelled.

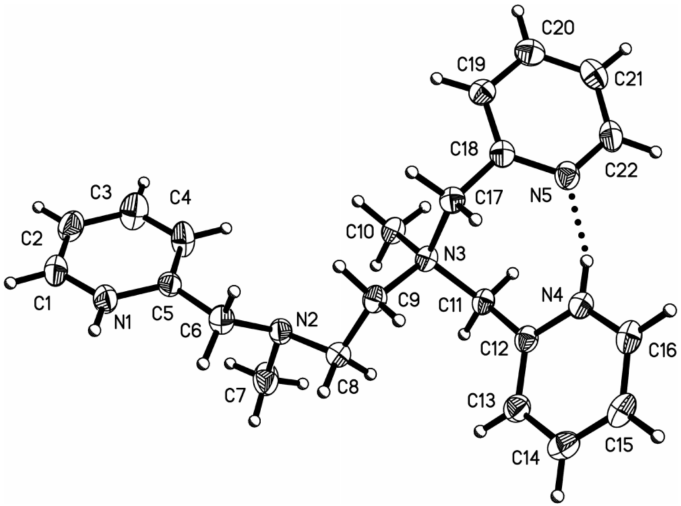


**Figure 1**
The organic cation in the title compound, showing displacement ellipsoids at the 50% probability level for non-H atoms. H atoms are shown as spheres of arbitrary radius. The intramolecular N—H···N hydrogen bond is indicated by a dotted line.

Data collection: *APEX*2 (Bruker–Nonius, 2004); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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## References

Bruker-Nonius (2004). *APEX*2. Version 1.0–22. Bruker-Nonius BV, Delft, The Netherlands.

Bruker (2003). *SAINT*. Version 7.06a. Bruker AXS Inc., Madison, Wisconsin, USA.

Egdal, R. K., Hazell, A., Larsen, F. B., McKenzie, C. J. & Scarrow, R. C. (2003). *J. Am. Chem. Soc.* **125**, 32–33.

Glerup, J., Goodson, P. A., Hazell, A., Hazell, R., Hodgson, D. J., McKenzie, C. J., Michelsen, K., Rychlewska, U. & Toftlund, H. (1994). *Inorg. Chem.* **33**, 4105–4111.

Hazell, R., Jensen, K. B., McKenzie, C. J. & Toftlund, H. (1995). *J. Chem. Soc. Dalton Trans.* pp. 707–717.

Jensen, K. B., McKenzie, C. J., Simonsen, O., Toftlund, H. & Hazell, A. (1997). *Inorg. Chim. Acta*, **257**, 163–172.

Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Toftlund, H., Pedersen, E. & Yde-Andersen, S. (1984). *Acta Chem. Scand. Ser. A*, **38**, 693–697.

**o1988** Annette Rompel *et al.* • $C_{22}H_{30}N_5^{3+}\cdot 3ClO_4^-$ *Acta Cryst.* (2004). E**60**, o1987–o1988