$4F_o^2/[\sigma(F_o^2)]^2$. Max. $\Delta/\sigma = 0.106$; max. residual density 0.22 e Å^{-3}. No correction for secondary extinction was applied. Atomic scattering factors and anomalous-dispersion coefficients were used as quoted in the *SPD/PDP* software package (Enraf–Nonius, 1985) operating on a PDP11/44 computer. Final fractional coordinates for non-H atoms are given in Table 1.* The interatomic distances and angles are summarized in Table 2. The atom-numbering and hydrogen-bonding schemes are shown in Fig. 1.

Related literature. Products of the reaction of orthophosphoric acid with mono-, oligo- and polymeric caprolactam are of interest to industry (Mladenov & Vladkova, 1977). They have been characterized by X-ray powder and infrared spectroscopy methods (Mladenov, Vladkova, Fakirov & Mitzulov, 1978), by morphological and kinetic studies (Mladenov, Vladkova & Fakirov, 1980; Mladenov, Vladkova, Tzenkova & Fakirov, 1980), and by thermal analysis (Vladkova, 1981). A powder pattern of the title compound has been evaluated and submitted to the PDF database (Macíček, 1991).

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54411 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1′,1‴-Dimethyl-1,1″-tetramethylenedi(4,4′-bipyridinium) Tetraperchlorate

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Abstract. $C_{26}H_{30}N_4^{4+}.4ClO_4^-$, $M_r = 796.36$, orthorhombic, *Pbca*, $a = 9.791$ (2), $b = 15.908$ (4), $c = 21.288$ (5) Å, $V = 3315.7$ Å^3, $Z = 4$, $D_x = 1.595$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 4.399$ cm^{-1}, $F(000) = 1640$, $T = 295$ (1) K, $R = 0.052$ for 2072 unique observed reflections with $I > 3.0\sigma(I)$ and 226 parameters. 1′,1‴-Dimethyl-1,1″-tetramethylenedi(4,4′-bipyridinium) belongs to the vio-

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B_{eq}
Cl(1)	0·1311 (1)	0·03338 (7)	0·67162 (5)	4·66 (2)
Cl(2)	0·6671 (1)	0·23919 (7)	0·58763 (5)	5·00 (2)
O(1)	0·0364 (5)	0·0925 (3)	0·6476 (2)	9·2 (1)
O(2)	0·2393 (4)	0·0257 (2)	0·6282 (2)	7·18 (9)
O(3)	0·1793 (5)	0·0617 (3)	0·7304 (2)	8·9 (1)
O(4)	0·0661 (4)	−0·0458 (2)	0·6801 (2)	7·6 (1)
O(5)	0·6102 (5)	0·3070 (2)	0·6211 (2)	8·2 (1)
O(6)	0·7822 (5)	0·2117 (3)	0·6218 (2)	9·3 (1)
O(7)	0·5758 (4)	0·1696 (2)	0·5843 (2)	7·5 (1)
O(8)	0·7020 (6)	0·2627 (3)	0·5275 (2)	10·6 (1)
N(1)	−0·8058 (3)	−0·2502 (2)	0·7027 (2)	4·02 (7)
N(2)	−0·2888 (3)	−0·0138 (2)	0·5608 (2)	3·76 (7)
C(1)	−0·9104 (5)	−0·3051 (3)	0·7325 (2)	5·4 (1)
C(2)	−0·7356 (5)	−0·1953 (3)	0·7366 (2)	4·5 (1)
C(3)	−0·6373 (5)	−0·1471 (3)	0·7109 (2)	4·27 (9)
C(4)	−0·6063 (4)	−0·1538 (2)	0·6476 (2)	4·92 (9)
C(5)	−0·6824 (6)	−0·2100 (3)	0·6137 (2)	6·0 (1)
C(6)	−0·7805 (6)	−0·2560 (3)	0·6415 (2)	6·4 (1)
C(7)	−0·4950 (4)	−0·1030 (2)	0·6175 (2)	4·81 (9)
C(8)	−0·4693 (5)	−0·1103 (3)	0·5537 (2)	4·24 (9)
C(9)	−0·3676 (6)	−0·0654 (3)	0·5272 (2)	4·4 (1)
C(10)	−0·3111 (5)	−0·0056 (3)	0·6229 (2)	4·36 (9)
C(11)	−0·4124 (5)	−0·0491 (3)	0·6513 (2)	4·33 (9)
C(12)	−0·1755 (5)	0·0322 (3)	0·5297 (2)	4·4 (1)
C(13)	−0·0565 (4)	−0·0247 (3)	0·5159 (2)	3·78 (9)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

N(1)—C(1)	1·488 (6)	C(1)—N(1)—C(2)	120·8 (4)
N(1)—C(2)	1·326 (6)	C(1)—N(1)—C(6)	120·3 (4)
N(1)—C(6)	1·329 (6)	C(2)—N(1)—C(6)	118·8 (4)
C(2)—C(3)	1·347 (6)	N(1)—C(2)—C(3)	121·7 (4)
C(3)—C(4)	1·384 (5)	C(2)—C(3)—C(4)	120·6 (4)
C(4)—C(5)	1·371 (6)	N(1)—C(6)—C(5)	121·8 (5)
C(5)—C(6)	1·346 (7)	C(6)—C(5)—C(4)	120·8 (4)
C(4)—C(7)	1·500 (5)	C(3)—C(4)—C(5)	116·3 (4)
C(7)—C(8)	1·386 (5)	C(3)—C(4)—C(7)	122·3 (4)
C(7)—C(11)	1·381 (5)	C(5)—C(4)—C(7)	121·4 (3)
C(8)—C(9)	1·349 (6)	C(4)—C(7)—C(8)	120·4 (4)
N(2)—C(9)	1·335 (5)	C(4)—C(7)—C(11)	122·5 (3)
N(2)—C(10)	1·346 (5)	C(8)—C(7)—C(11)	117·1 (4)
N(2)—C(12)	1·485 (5)	C(7)—C(8)—C(9)	120·0 (4)
C(10)—C(11)	1·352 (6)	C(8)—C(9)—N(2)	122·0 (4)
C(12)—C(13)	1·505 (6)	C(7)—C(11)—C(10)	121·0 (4)
C(13)—C(13)	1·517 (6)	C(11)—C(10)—N(2)	120·5 (4)
		C(9)—N(2)—C(12)	119·8 (3)
		C(10)—N(2)—C(12)	120·8 (3)
		C(9)—N(2)—C(10)	119·4 (4)
		N(2)—C(12)—C(13)	111·7 (3)
		C(12)—C(13)—C(13)	109·9 (3)
Cl(1)—O(1)	1·416 (4)	O(1)—Cl(1)—O(2)	108·2 (2)
Cl(1)—O(2)	1·413 (4)	O(1)—Cl(1)—O(3)	109·1 (3)
Cl(1)—O(3)	1·411 (4)	O(1)—Cl(1)—O(4)	109·9 (3)
Cl(1)—O(4)	1·423 (4)	O(2)—Cl(1)—O(3)	110·9 (2)
Cl(2)—O(5)	1·407 (4)	O(2)—Cl(1)—O(4)	110·0 (2)
Cl(2)—O(6)	1·413 (4)	O(3)—Cl(1)—O(4)	108·7 (2)
Cl(2)—O(7)	1·425 (4)	O(5)—Cl(2)—O(6)	107·0 (3)
Cl(2)—O(8)	1·376 (6)	O(5)—Cl(2)—O(7)	111·9 (3)
		O(5)—Cl(2)—O(8)	111·1 (3)
		O(6)—Cl(2)—O(7)	106·6 (2)
		O(6)—Cl(2)—O(8)	111·4 (3)
		O(7)—Cl(2)—O(8)	108·7 (3)

logen compounds, which undergo reversible one-electron reduction to colored radical cations. This property is utilized in the mediation of electron-transfer processes. The structure determination was undertaken to establish the stereochemistry of the compound. The dihedral angle between the least-squares planes of the two pyridine rings is 3·3°, indicating that the two rings are essentially coplanar. One half of the molecule is related to the other half by a crystallographic inversion center at the middle of the butanediyl group in the unit cell. The anions display an approximate tetrahedral geometry.

Experimental. The title compound was synthesized by the method reported previously (Yu & Wang, 1985). Yellow crystal, prismatic, recrystallized by slow evaporation from an aqueous solution at 323 (2) K with dimensions 0·4 × 0·4 × 0·3 mm. Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters from least-squares refinement of 25 reflections with $9 < \theta < 13°$. 3310 reflections measured, 2908 unique, $R_{int} = 0.027$, using ω–2θ scan technique within ranges $1 \le \theta \le 25°$, $0 \le h \le 11$, $0 \le k \le 18$, $0 \le l \le 25$, $(\sin\theta/\lambda)_{max} = 0{\cdot}5941$ Å^{-1}. Three standard reflections measured every hour showed a variation of less than 2·4%. 2072 independent reflections with $I > 3{\cdot}0\sigma(I)$. Lorentz and polarization corrections, no absorption correction.

The structure was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refinement was by full-matrix least squares on F; all non-H atoms were refined anisotropically. A difference Fourier synthesis calculated at this stage of the refinement revealed all H-atom positions; these were included in the difference-map positions with arbitrary isotropic temperature factors $B = 4{\cdot}0$ Å^2 in the subsequent calculations, but not refined. Final $R = 0{\cdot}052$ and $wR = 0{\cdot}049$ for 226 variables with $w = 1/\sigma^2(F_o)$ and $S = 1{\cdot}71$; $(\Delta/\sigma)_{max} = 0{\cdot}04$ in final refinement cycle, the difference electron density map was essentially featureless with $\Delta\rho_{max} = 0{\cdot}53$ and $\Delta\rho_{min} = -0.46$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations on a PDP11/44 computer using the Enraf–Nonius *SDP-Plus Structure Determination Package* (Frenz, 1985) and *ORTEP*II (Johnson, 1976). Final coordinates are given in Table 1, with bond lengths and angles in Table 2.* A perspective view of the cation is shown in Fig. 1, the molecular packing is illustrated in Fig. 2.

* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54382 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

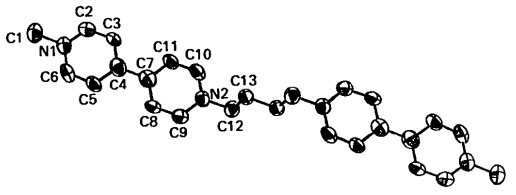

Fig. 1. Perspective view of the cation.

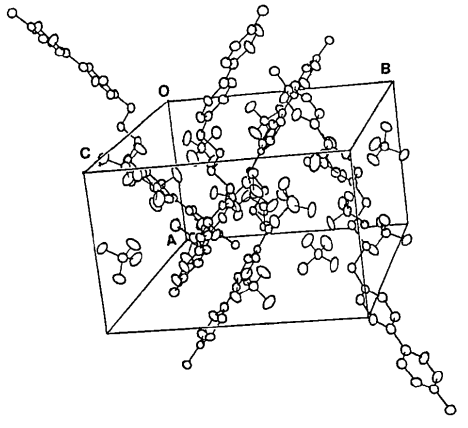

Fig. 2. Unit-cell packing diagram.

Related literature. The photoreduction behaviour and radical reaction of the title compound have been reported by Yu & Wang (1985) and Atherton, Tsukahara & Wilkins (1986). Crystal structure analysis of related compounds: *N,N'*-dimethyl-4,4'-bipyridinium tetrachlorocuprate (Russell & Wallwork, 1969); *N,N'*-dimethyl-4,4'-bipyridinium 2-dicyanomethylene-1,1,3,3-tetracyanopropanediide (Nakamura, Kai, Yasuoka & Kasai, 1981).

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Structures of 1,2-Ethanediammonium Dinitrate (1) and 1,3-Propanediammonium Dinitrate (2)

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Abstract. (1): $C_2H_{10}N_2^{2+}.2NO_3^-$, $M_r = 186.1$, triclinic, $P\bar{1}$, $a = 5.068$ (1), $b = 5.514$ (4), $c = 7.185$ (3) Å, $\alpha = 105.03$ (3), $\beta = 90.16$ (3), $\gamma = 93.58$ (4)°, $V = 193.5$ (2) Å^3, $Z = 1$, $D_x = 1.597$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.15$ mm^{-1}, $F(000) = 98$, $T = 295$ K, final $R = 0.057$, $wR = 0.057$ for 379 independent observed reflections. (2): $C_3H_{12}N_2^{2+}.2NO_3^-$, $M_r = 200.2$, monoclinic, Cc, $a = 8.161$ (1), $b = 8.724$ (2), $c = 13.048$ (2) Å, $\beta = 95.86$ (1)°, $V = 924.2$ (2) Å^3, $Z = 4$, $D_x = 1.439$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 0.12$ mm^{-1}, $F(000)$ 424, $T = 295$ K, final $R = 0.041$, $wR = 0.053$ for 649 independent observed reflections. In (1) there is an inversion center midway between the two C atoms, and only half of the dication and one nitrate are crystallographically unique. In both (1) and (2) the dications are in a *trans* configuration and there is extensive intermolecular hydrogen bonding present, with each of the diammonium H atoms participating in moderate to weak hydrogen bonds.

Experimental. Colorless 0.12 × 0.23 × 0.34 mm (1) and 0.23 × 0.35 × 0.45 mm (2) data crystals were provided by Dr Robert McKenney of Air Force Armament Laboratory, Eglin AFB, FL. Hereafter, values for (2) where different will be given in square brackets [ ]. Automated Siemens *R3m/V* diffractometer with incident-beam monochromator. 25 centered reflections within $18 \le 2\theta \le 28°$ [$36 \le 2\theta \le 56°$] used for determining lattice parameters. $(\sin\theta/\lambda)_{max} = 0.54$ [0.55] Å^{-1}, range of *hkl*: $-5 \le h \le 2$, $-5 \le k \le 5$, $-7 \le l \le 7$ [$0 \le h \le 8$, $0 \le k \le 9$, $-13 \le l \le 13$].

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