2.47 Å and four longer ones of about 2.76 Å. Na(5) atoms are included in more distorted polyhedra with Na—F distances ranging from 2.265 to 2.787 Å. Other compounds (Na_2CoFeF_7 and a second modification of Na_2CuFeF_7) exhibiting similar structural features have been investigated by Welsch & Babel (1992). Their results are in good agreement with ours.

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Structure of Methanol(nitrato)(2,2′:6′,2″-terpyridyl)copper(II) Perchlorate

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Abstract. $[Cu(NO_3)(C_{15}H_{11}N_3)(CH_3OH)]ClO_4$, $M_r = 490.3$, triclinic, $P\bar{1}$, $a = 9.105$ (2), $b = 9.588$ (2), $c = 11.753$ (3) Å, $\alpha = 78.21$ (2), $\beta = 80.45$ (2), $\gamma = 69.72$ (1)°, $V = 937.1$ (6) Å^3, $Z = 2$, $D_x = 1.737$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.36$ mm^{-1}, $F(000) = 498$, room temperature, $R = 0.054$ for 3051 independent reflections. The compound consists of discrete mononuclear $[Cu(terpy)(CH_3OH)(NO_3)]^+$ cationic units and non-coordinated perchlorate groups. The Cu atom is hexacoordinated and its configuration corresponds to a tetragonally distorted octahedron. A structural comparison is made with other terpyridyl-containing Cu^{II} complexes and the influence of the counterion in this series of complexes is anlayzed.

Introduction. Six- and five-coordinate complexes of formula $Cu(terpy)X_2.nH_2O$ (X = counterion and terpy = 2,2′:6′,2″-terpyridine) are interesting examples for investigating various aspects of vibronic coupling and the Jahn–Teller effect (Reinen & Friebel, 1979). These mono(terpyridine) complexes

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can give rise not only to mononuclear species but also to polynuclear ones when X can act as a bridging ligand. So when $X = Cl^-$, OH^-, CN^-, oxalate, squarate and chloranilate, dinuclear complexes (Castro, Faus, Julve & Gleizes, 1991; Solans, Aguiló, Gleizes, Faus, Julve & Verdaguer, 1990; Folgado, Coronado, Beltrán-Porter, Rojo & Fuertes, 1989; Folgado, Ibáñez, Coronado, Beltrán, Savariault & Galy, 1988; Rojo, Arriortúa, Ruiz, Darriet, Villeneuve & Beltrán-Porter, 1987), tetranuclear complexes (Folgado, Gómez-Romero, Sapiña, Beltrán-Porter, 1990) and one-dimensional chains (Solans, Aguiló, Gleizes, Faus̆, Julve & Verdaguer, 1990; Anderson, Packard & Wicholas, 1976) have been obtained and structurally characterized.

Although single crystals of the mono(terpyridine) complexes Cu(terpy)X_2.$n$$H_2O$ have been obtained for a great variety of X groups, the crystal structures of the nitrate and perchlorato complexes could not be determined. In the former case the translucent blue needles of Cu(terpy)$(NO_3)_2$.$n$$H_2O$ become opaque without mother liquor. The perchlorate complex crystallizes as blue sheet-like crystals which are not suitable for X-ray diffraction. In this paper, we report the crystal structure of the complex [Cu(terpy)$(CH_3OH)(NO_3)$]ClO_4 which contains both nitrate and perchlorate anions as well as semicoordinated methanol. A structural comparison with related mono(tridentate N-donor ligand)copper(II) complexes focusing on the structural role of the X group is also presented.

Table 1. *Fractional coordinates* ($\times 10^4$ *for* N, C *and* O *atoms*, $\times 10^5$ *for* Cu) *and equivalent isotropic temperature factors* (Å^2) *of the non-*H *atoms with e.s.d.'s in parentheses*

$$U_{eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu(1)	16443 (4)	13081 (4)	15633 (3)	2.49 (2)
N(1)	2427 (3)	2962 (3)	605 (2)	2.56 (11)
C(2)	3671 (4)	2786 (5)	−195 (3)	3.08 (14)
C(3)	4120 (5)	3999 (5)	−815 (4)	3.64 (16)
C(4)	3256 (5)	5399 (5)	−581 (3)	3.38 (15)
C(5)	1962 (4)	5601 (4)	264 (3)	3.17 (14)
C(6)	1579 (4)	4356 (4)	841 (3)	2.55 (12)
N(7)	152 (3)	3010 (3)	2200 (2)	2.42 (10)
C(8)	263 (4)	4389 (3)	1766 (3)	2.37 (12)
C(9)	−797 (5)	5630 (4)	2215 (3)	3.18 (14)
C(10)	−1927 (5)	5414 (5)	3095 (3)	3.45 (16)
C(11)	−2031 (5)	4000 (5)	3536 (3)	3.23 (14)
C(12)	−959 (4)	2796 (4)	3039 (3)	2.68 (13)
N(13)	377 (4)	246 (3)	2769 (2)	2.68 (11)
C(14)	−835 (4)	1189 (4)	3368 (3)	2.71 (13)
C(15)	−1830 (5)	647 (5)	4210 (4)	3.56 (16)
C(16)	−1532 (6)	−880 (5)	4482 (4)	4.11 (18)
C(17)	−286 (5)	−1856 (5)	3898 (4)	3.88 (18)
C(18)	625 (5)	−1236 (4)	3018 (3)	3.39 (15)
O(19)	2748 (3)	−357 (3)	664 (2)	3.50 (11)
N(20)	2083 (4)	−99 (3)	−298 (3)	3.44 (13)
O(21)	955 (4)	1058 (3)	−460 (3)	4.71 (14)
O(22)	2614 (5)	−1006 (4)	−952 (3)	6.12 (19)
O(23)	3698 (4)	657 (4)	2663 (4)	5.18 (15)
C(24)	4878 (6)	−689 (6)	2960 (5)	5.65 (23)
Cl(1)	−6404 (1)	4064 (1)	3658 (1)	3.20 (4)
O(1)	−5501 (36)	3258 (32)	2660 (24)	3.05 (66)†
O(1′)	−5852 (19)	3392 (20)	2694 (16)	8.45 (76)†
O(2)	−8019 (5)	4074 (7)	3950 (5)	2.99 (23)
O(3)	−5603 (50)	3534 (37)	4612 (45)	5.05 (108)‡
O(3′)	−5736 (38)	3125 (71)	4478 (49)	3.99 (134)‡
O(3″)	−5375 (94)	3164 (81)	4829 (64)	4.00 (119)‡
O(4)	−6653 (35)	5521 (33)	3261 (33)	4.48 (107)†
O(4′)	−6230 (17)	5583 (19)	3452 (15)	4.29 (46)†

† S.o.f. = 0.5.
‡ S.o.f. = 0.33.

Experimental. [Cu(terpy)$(CH_3OH)(NO_3)$]ClO_4 was prepared by adding sodium perchlorate (0.25 mmol) to a hot methanolic solution of the complex Cu(terpy)$(NO_3)_2$.H_2O (0.25 mmol, 60 cm^3) (Castro, Faus, Julve & Gleizes, 1991) with stirring. Polyhedral blue crystals were obtained by slow evaporation at room temperature.

A crystal of dimensions 0.1 × 0.1 × 0.2 mm was mounted on an Enraf–Nonius CAD-4 automatic diffractometer, equipped with a graphite-monochromator and Mo $K\alpha$ radiation. The unit-cell parameters were determined from a least-squares fit of the angular setting of 25 reflections in the range $12 \leq \theta \leq 18°$. The intensities of 3323 reflections were measured ($-10 \leq h \leq 10$, $-11 \leq k \leq 11$, $0 \leq l \leq 13$) by using the $\omega/2\theta$ scan technique to $\theta_{max} = 25°$. 3051 of these were assumed to be observed applying the condition $I > 2.5\sigma(I)$. Three standard reflections were remeasured every 2 h and no significant fluctuation was observed in their intensities. The data were corrected for Lorentz and polarization effects, but not for absorption.

The structure was solved by direct methods using the *MULTAN*84 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1984) system of computer programs and refined by full-matrix least-squares methods, using *SHELX*76 (Sheldrick, 1976). The function minimized was $\sum w||F_o| - |F_c||^2$, where $w = (\sigma^2|F_o| + 0.0019|F_o|^2)^{-1}$. Three O atoms of the perchlorate group were found to be disordered over two [(O(1) and O(4)] or three [O(3)] sites. Occupancy factors of 0.5 [O(1) and O(4)] and 0.33 [O(3)] were assigned to each site according to the Fourier map height of the respective peaks. H atoms were located from a difference synthesis, except H(17) and H(24) for which positions consistent with normal stereochemistry were computed. All H atoms were refined with an overall isotropic temperature factor using a riding model for computed atoms. The remaining atoms were refined anisotropically. At convergence the final R (wR) value was 0.054 (0.058). The number of parameters refined was 335. The minimum and maximum heights in the final Fourier difference synthesis were −0.3 and 0.4 e Å^{-3}, respectively, and the maximum Δ/σ was 0.1. The effect of anomalous dispersion was included for all non-H atoms. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Geometrical calculations were performed with *XANADU* (Roberts & Sheldrick, 1975) and molecular illustrations were drawn with *ORTEP* (Johnson, 1965) and *SCHAKAL* (Keller, 1987).

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Cu(1)—N(1)	2.020 (3)	C(12)—C(14)	1.479 (5)
Cu(1)—N(7)	1.921 (3)	N(13)—C(14)	1.357 (5)
Cu(1)—N(13)	2.014 (3)	N(13)—C(18)	1.336 (5)
Cu(1)—O(19)	1.975 (2)	C(14)—C(15)	1.374 (6)
Cu(1)—O(21)	2.634 (3)	C(15)—C(16)	1.372 (7)
Cu(1)—O(23)	2.284 (3)	C(16)—C(17)	1.378 (6)
N(1)—C(2)	1.332 (5)	C(17)—C(18)	1.385 (6)
N(1)—C(6)	1.351 (5)	N(20)—O(19)	1.309 (5)
C(2)—C(3)	1.391 (6)	N(20)—O(21)	1.229 (4)
C(3)—C(4)	1.358 (7)	N(20)—O(22)	1.200 (5)
C(4)—C(5)	1.395 (6)	O(23)—C(24)	1.385 (6)
C(5)—C(6)	1.373 (5)	Cl(1)—O(1)	1.50 (2)
C(6)—C(8)	1.474 (5)	Cl(1)—O(1′)	1.35 (2)
N(7)—C(8)	1.349 (4)	Cl(1)—O(2)	1.451 (5)
N(7)—C(12)	1.330 (5)	Cl(1)—O(3)	1.35 (6)
C(8)—C(9)	1.385 (5)	Cl(1)—O(3′)	1.25 (7)
C(9)—C(10)	1.371 (6)	Cl(1)—O(3″)	1.70 (8)
C(10)—C(11)	1.379 (6)	Cl(1)—O(4)	1.33 (3)
C(11)—C(12)	1.391 (5)	Cl(1)—O(4′)	1.49 (2)

N(7)—Cu(1)—N(1)	80.5 (1)	C(12)—N(7)—C(8)	122.3 (3)
N(13)—Cu(1)—N(1)	160.8 (1)	N(7)—C(8)—C(6)	112.8 (3)
N(13)—Cu(1)—N(7)	80.4 (1)	C(9)—C(8)—C(6)	127.8 (3)
O(19)—Cu(1)—N(1)	100.9 (1)	C(9)—C(8)—N(7)	119.3 (3)
O(19)—Cu(1)—N(7)	165.5 (1)	C(10)—C(9)—C(8)	118.7 (3)
O(19)—Cu(1)—N(13)	98.0 (1)	C(11)—C(10)—C(9)	121.6 (4)
O(21)—Cu(1)—N(1)	83.9 (1)	C(12)—C(11)—C(10)	117.4 (4)
O(21)—Cu(1)—N(7)	112.4 (1)	C(11)—C(12)—N(7)	120.6 (3)
O(21)—Cu(1)—N(13)	104.7 (1)	C(14)—C(12)—N(7)	112.2 (3)
O(21)—Cu(1)—O(19)	53.8 (1)	C(14)—C(12)—C(11)	127.1 (3)
O(23)—Cu(1)—N(1)	88.4 (1)	C(14)—N(13)—Cu(1)	113.7 (2)
O(23)—Cu(1)—N(7)	102.7 (1)	C(18)—N(13)—Cu(1)	126.9 (3)
O(23)—Cu(1)—N(13)	94.5 (1)	C(18)—N(13)—C(14)	119.4 (3)
O(23)—Cu(1)—O(19)	91.8 (1)	N(13)—C(14)—C(12)	114.4 (3)
O(23)—Cu(1)—O(21)	142.1 (1)	C(15)—C(14)—C(12)	124.5 (4)
C(2)—N(1)—Cu(1)	126.3 (3)	C(15)—C(14)—N(13)	121.1 (3)
C(6)—N(1)—Cu(1)	114.3 (2)	C(16)—C(15)—C(14)	118.8 (4)
C(6)—N(1)—C(2)	119.4 (3)	C(17)—C(16)—C(15)	120.8 (4)
C(3)—C(2)—N(1)	122.1 (4)	C(18)—C(17)—C(16)	117.6 (4)
C(4)—C(3)—C(2)	118.2 (4)	C(17)—C(18)—N(13)	122.1 (4)
C(5)—C(4)—C(3)	120.4 (4)	N(20)—O(19)—Cu(1)	109.1 (2)
C(6)—C(5)—C(4)	118.4 (4)	O(21)—N(20)—O(19)	117.3 (3)
C(5)—C(6)—N(1)	121.4 (3)	O(22)—N(20)—O(19)	117.8 (3)
C(8)—C(6)—N(1)	114.0 (3)	O(22)—N(20)—O(21)	124.9 (4)
C(8)—C(6)—C(5)	124.6 (3)	N(20)—O(21)—Cu(1)	79.8 (2)
C(8)—N(7)—Cu(1)	118.4 (2)	C(24)—O(23)—Cu(1)	132.8 (3)
C(12)—N(7)—Cu(1)	119.3 (2)		

Discussion. The atomic positional and equivalent isotropic thermal parameters are listed in Table 1. Bond lengths and angles are given in Table 2. The structure of the compound is depicted in Fig. 1 together with the atomic numbering system.*

The structure consists of cationic mononuclear $[Cu(terpy)(NO_3)(CH_3OH)]^+$ units and noncoordinated perchlorate ions. The coordination polyhedron around the Cu atom can be described as a tetragonally distorted octahedron. The N(1), N(7) and N(13) N atoms of the terpy ligand and the O(19) atom of the nitrate group occupy the equatorial positions, whereas the O(23) and O(21) atoms of the methanol molecule and nitrate anion, respectively, occupy the axial sites. The N(1) and N(13) atoms of the terminal pair of pyridine rings in the terpy ligand form Cu—N bonds of practically identical length [2.020 (3) and 2.014 (3) Å], whereas the central Cu—N(7) bond is shorter [1.921 (3) Å]. This feature is a result of geometrical constraints of terpy as a ligand and has been observed previously (Anderson, Packard & Wicholas, 1976). The Cu atom is displaced by 0.134 Å from the N(1), N(7), N(13), O(19) mean plane toward the axially semi-coordinated O atom of the methanol [Cu(1)—O(23) = 2.284 (3) Å]. An O atom of the nitrato group occupies the sixth coordination position at a longer distance [Cu(1)—O(21) = 2.634 (3) Å]. The angles subtended at the Cu atom by the donor N atoms are less than the ideal 90° value [80.5 (1) and 80.4 (1)° for N(1)—Cu(1)—N(7) and N(7)—Cu(1)—N(13), respectively].

The individual pyridine rings of the terpy ligand are planar to within 0.02 Å. However, the ligand itself is not planar: the dihedral angles between the middle and outer pyridyl rings are 1.97 and 3.21°. Average C—C bonds within the rings (1.38 Å), C—N bonds (1.34 Å) and C—C inter-ring bonds (1.48 Å) are unexceptional, as are interbond angles.

The nitrate ion is planar to within 0.007 Å. Its coordination to the Cu atom in an asymmetrical bidentate fashion through O(19) (*xy* plane) and O(21) (off the *z* axis) O atoms induces significant lengthening of the N(20)—O(19) [1.309 (5) Å] and N(20)—O(21) [1.229 (4) Å] bond distances [1.200 (5) Å for N(20)—O(22)]. This structural pattern involving ligand atoms positioned both in the *xy* plane and off the *z* axis in Cu^{II} complexes has been thoroughly discussed elsewhere (Hathaway, 1973).

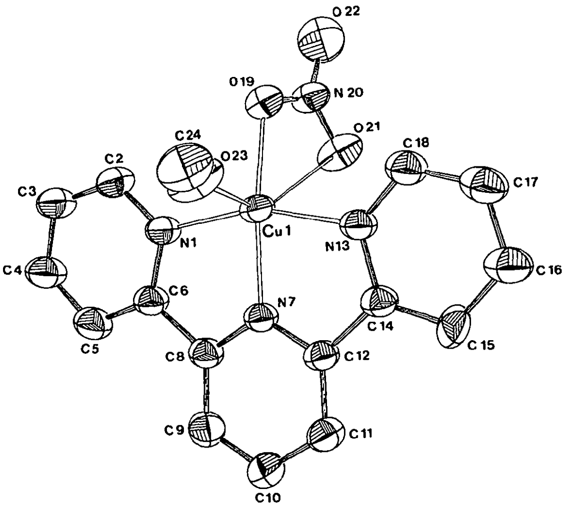

Fig. 1. Perspective view of the molecule showing atom labelling. Thermal ellipsoids are drawn at the 50% probability level.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond distances and angles involving H atoms, and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55309 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0283]

The disordered perchlorate anion is hydrogen bonded to the apical methanol molecule [2.830 (5) Å for O(23)···O(1)].

The shortest intermolecular Cu···Cu^{i} distance is 6.433 (3) Å [symmetry code: (i) = $-x$, $-y$, $-z$].

Close examples to the structure that we report herein are the complexes [Cu(terpy)(NO$_2$)(H$_2$O)]NO$_3$.H$_2$O (Savariault, Rojo, Arriortúa & Galy, 1983), [Cu(dien)(HCO$_2$)]HCO$_2$ (Davey & Stephens, 1971) and [Cu(dien)(CH$_3$COO)]ClO$_4$ (Towle, Hoffmann, Hatfield, Singh & Chaudhuri, 1988), where dien is diethylenetriamine. The Cu environment is also tetragonally distorted octahedral in these three complexes and three of the four equatorial positions are filled by the N atoms of the tridenate terpy and dien ligands. Formate and acetate act as bridges in an *anti-syn* fashion between Cu^{II} ions yielding one-dimensional chains of metal ions. The aqua complex is monomeric: the nitrite anion acts as an asymmetrically bidentate ligand, its two O atoms occupying one equatorial and one axial position, the remaining axial site being filled by a water molecule. In the title complex the nitrate and methanol play the role of the nitrite and water, respectively. The presence of the semicoordinated CH$_3$OH and water molecules in [Cu(terpy)(CH$_3$OH)(NO$_3$)]ClO$_4$ and [Cu(terpy)(NO$_2$)(H$_2$O)]NO$_3$.H$_2$O block the one-dimensional μ-bridging found in [Cu(dien)(HCO$_2$)]HCO$_2$ and [Cu(dien)(CH$_3$COO)]ClO$_4$. The structures of the nitrito and nitrato complexes show that it is possible to grow single crystals of closely related complexes by using mixed counterions.

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Chloro[diphenyl(diphenylphosphino-κP-methyl)phosphine selenide]gold(I) Dichloromethane Hemisolvate at 178 K

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Abstract. [AuCl(C$_{25}$H$_{22}$P$_2$Se)].$\frac{1}{2}$CH$_2$Cl$_2$, $M_r = 738.2$, monoclinic, $P2_1/c$, $a = 9.790$ (3), $b = 18.050$ (4), $c = 15.118$ (4) Å, $\beta = 105.86$ (3)°, $V = 2570$ Å^3, $Z = 4$, $D_x = 1.91$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 7.49$ mm^{-1}, $F(000) = 1412$, $T = 178$ K. The structure was refined to $R = 0.046$ for 3339 unique observed

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