the sense of rotation changes chain by chain alternately just as in $[N(CH_3)_4]_2ZnBr_4$ (Asahi *et al.*, 1988) and in $[N(CH_3)_4]_2MnCl_4$ (Mashiyama & Koshiji, 1989).

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Structures of Nitratobis(4-nitropyridine *N*-oxide)silver(I) and Dinitratobis(4-nitropyridine *N*-oxide)copper(II)

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Abstract. $[Ag(NO_3)(C_5H_4N_2O_3)_2]$ (I), $M_r = 450{\cdot}1$, monoclinic, $C2/c$, $a = 24{\cdot}073\ (4)$, $b = 5{\cdot}295\ (1)$, $c = 23{\cdot}396\ (4)$ Å, $\beta = 98{\cdot}73\ (2)°$, $V = 2948\ (1)$ Å^3, $Z = 8$, $D_x = 2{\cdot}03$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}41$ mm^{-1}, $F(000) = 1776$, $T = 299\ (1)$ K, final $R = 0{\cdot}042$ for 2309 observed unique reflections. $[Cu(NO_3)_2(C_5H_4N_2O_3)_2]$ (II), $M_r = 467{\cdot}8$, monoclinic, $P2_1$, $a = 10{\cdot}670\ (2)$, $b = 8{\cdot}1249\ (9)$, $c = 9{\cdot}699\ (2)$ Å, $\beta = 104{\cdot}71\ (2)°$, $V = 813{\cdot}3\ (2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}91$ Mg m^{-3}, $\mu = 1{\cdot}43$ mm^{-1}, $F(000) = 470$, $T = 299\ (1)$ K, final $R = 0{\cdot}031$ for 1715 reflections. In (I), the Ag atom exhibits a distorted tetrahedral geometry to two *N*-oxide O atoms and two O atoms of a nitrate ion. In (II), the two *N*-oxide O atoms and the two O atoms of two nitrate ions coordinate to the Cu atom forming a square-planar *cis* complex. There are no contacts between the metal atoms and the nitro group of 4-nitropyridine *N*-oxide.

Introduction. Certain nitrobenzene derivatives have recently attracted much attention as organic nonlinear optical materials (Twieg & Jain, 1983). If the crystal structure has a center of symmetry, optical second harmonic generation cannot be expected. Accordingly, the control of the molecular arrangement in crystals is desirable. A solution to this problem may be polynuclear metal complex formation. In the present study, the structures of two metal complexes involving 4-nitropyridine *N*-oxide have been studied. Metal cations, Ag^+ and Cu^{2+}, were selected because of their relatively small coordination numbers.

Experimental. (I): Yellow needle-like crystals were obtained from ethanol solution of silver nitrate and 4-nitropyridine *N*-oxide (molar ratio 1/1). A crystal of dimensions $0{\cdot}2 \times 0{\cdot}3 \times 0{\cdot}35$ mm was mounted on a Rigaku AFC-5 four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. Laue group $2/m$, systematic absences hkl, $h + k$ odd; $h0l$, h or l odd (Cc or $C2/c$), cell parameters refined by least squares for 25 2θ values ($20 < 2\theta < 30°$); intensity

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measurement performed to $2\theta = 55°$ ($h\ 0 \rightarrow 31$, $k\ -6 \rightarrow 0$, $l\ -30 \rightarrow 30$), θ–2θ scan, scan speed 6° min^{-1} in θ. Variation of five standard reflections, $0{\cdot}98 \leq \sum(|F_o|/|F_o|_{\rm initial})/5 \leq 1{\cdot}00$. 3456 reflections measured, 2366 observed reflections with $|F_o| > 3\sigma(|F_o|)$; absorption correction ($0{\cdot}66 < A < 0{\cdot}74$). With the assumption of space group $C2/c$ and $Z = 8$ the structure was successfully solved by Patterson and Fourier methods. Non-H atoms were refined with anisotropic thermal parameters. All H atoms were located on a difference synthesis and refined isotropically. $\sum w||F_o| - |F_c||^2$ minimized, $w^{-1} = \sigma^2(|F_o|) + (0{\cdot}015|F_o|)^2$. Final $R = 0{\cdot}042$, $wR = 0{\cdot}053$, $S = 2{\cdot}15$ for 2309 unique reflections. Reflection/parameter ratio 8·9, $(\Delta/\sigma) < 0{\cdot}04$, $-0{\cdot}73 \leq \Delta\rho \leq 0{\cdot}67$ e Å^{-3}. Complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). *UNICS*III program system (Sakurai & Kobayashi, 1979), FACOM M-380R computer of this university.

(II): Green polyhedral crystals were obtained from the filtrate of a mixture of copper nitrate and 4-nitropyridine *N*-oxide (molar ratio 1/1) ethanol solutions. A crystal of dimensions 0·2 × 0·5 × 0·5 mm, Laue group $2/m$, systematic absences $0k0$, k odd ($P2_1$ or $P2_1/m$); intensity measurement performed to $2\theta = 55°$ ($h\ -13 \rightarrow 13$, $k\ 0 \rightarrow 10$, $l\ 0 \rightarrow 12$), $1{\cdot}00 \leq \sum(|F_o|/|F_o|_{\rm initial})/5 \leq 1{\cdot}01$, 2127 reflections measured, 1806 observed, $0{\cdot}58 < A < 0{\cdot}74$. With the assumption of space group $P2_1$ and $Z = 2$, the structure could be solved successfully. Five out of eight H atoms were located by difference synthesis and others were calculated. Final $R = 0{\cdot}031$, $wR = 0{\cdot}041$, $S = 1{\cdot}93$ for 1715 unique reflections.* Reflection/parameter ratio 5·8, $(\Delta/\sigma) < 0{\cdot}3$, $-0{\cdot}51 \leq \Delta\rho \leq 0{\cdot}45$ e Å^{-3}. Structural chirality (Glazer & Stadnicka, 1989) was determined by the anomalous-scattering technique ($R = 0{\cdot}036$ and $wR = 0{\cdot}046$ for enantiomeric structure).

Discussion. Final atomic coordinates and interatomic distances and bond angles are presented in Tables 1 and 2.* Both complexes are mononuclear. The Ag^+ ion in (I) is coordinated by the two *N*-oxide O atoms and two O atoms of a nitrate ion as shown in Fig. 1. The Ag—O distances range from 2·318 (3) to 2·573 (6) Å. The four ligating atoms form a distorted tetrahedron, the basal plane being formed by the O(1), O(10) and O(20) atoms. As seen from Fig. 2, the Cu^{2+} ion in (II) is coordinated by the two *N*-oxide O atoms and two O atoms of two nitrate ions. The Cu—O distances range from 1·910 (3) to 1·963 (3) Å and the coordination geometry is nearly square planar with the two nitrate O atoms in *cis* positions. In both crystals, metal—O—N(*N*-oxide) angles are close to 120° and such geometry is observed in other pyridine *N*-oxide complexes, for example, $[ZnCl_2(C_5H_5NO)_2]$ (McConnell, Day & Wood, 1986), $[TlBr_3(C_5H_5NO)_2]$ (Hiller, Garcia-Fernandez, Bermejo & Castano, 1986), $[M(C_5H_5NO)_6](ClO_4)_2$ (M = Cu, Co and Fe, Taylor, 1978), $[M(C_5H_5NO)_6]X_2$ (M = Cu, Zn; X = ClO_4, BF_4, O'Connor, Sinn & Carlin, 1977). The mean N—O

Table 1. *Positional parameters* ($\times 10^4$, $\times 10^5$ *for* Ag *and* Cu) *and equivalent isotropic temperature factors* (*Hamilton*, 1959)

	x	y	z	B_{eq}(Å$^2 \times 10$)
(I)				
Ag	41 (2)	6199 (10)	8558 (2)	48
O(10)	−978 (1)	560 (8)	854 (2)	47
O(11)	−1819 (2)	1085 (8)	3205 (2)	48
O(12)	−2153 (2)	4575 (8)	2820 (2)	54
N(11)	−1164 (2)	1121 (8)	1332 (2)	34
N(12)	−1880 (2)	2637 (9)	2817 (2)	37
C(12)	−1526 (2)	3067 (11)	1340 (2)	38
C(13)	−1756 (2)	3628 (10)	1823 (2)	35
C(14)	−1610 (2)	2134 (9)	2306 (2)	29
C(15)	−1234 (2)	203 (10)	2311 (2)	40
C(16)	−1010 (2)	−250 (10)	1815 (3)	41
O(20)	366 (1)	−2009 (7)	211 (1)	43
O(21)	2352 (1)	−9432 (7)	437 (2)	45
O(22)	2715 (1)	−6463 (8)	1001 (2)	50
N(21)	833 (1)	−3291 (8)	325 (2)	29
N(22)	2326 (1)	−7372 (8)	673 (2)	36
C(22)	1232 (2)	−2544 (9)	764 (2)	32
C(23)	1728 (2)	−3860 (9)	876 (2)	32
C(24)	1803 (2)	−5938 (9)	550 (2)	28
C(25)	1390 (2)	−6715 (9)	111 (2)	31
C(26)	908 (2)	−5339 (10)	0 (2)	33
O(1)	625 (2)	3372 (11)	1512 (2)	79
O(2)	−160 (2)	4929 (11)	1310 (3)	92
O(3)	438 (3)	7173 (14)	1776 (3)	119
N	303 (2)	5144 (9)	1558 (2)	47
(II)				
Cu	2609 (5)	0*	81445 (5)	24
O(10)	2091 (3)	518 (4)	8686 (4)	35
N(11)	2474 (3)	2086 (5)	8835 (4)	28
C(12)	2610 (5)	2926 (7)	7680 (5)	31
C(13)	3064 (5)	4502 (7)	7823 (5)	30
C(14)	3382 (4)	5193 (8)	9171 (4)	26
C(15)	3228 (5)	4340 (7)	10338 (5)	31
C(16)	2768 (4)	2771 (7)	10135 (5)	31
O(11)	4120 (4)	7547 (6)	8309 (4)	52
O(12)	4146 (4)	7476 (6)	10538 (4)	51
N(12)	3915 (4)	6867 (6)	9343 (4)	34
O(20)	−14 (3)	2159 (4)	7346 (4)	34
N(21)	−1172 (4)	2802 (5)	6727 (4)	26
C(22)	−1421 (4)	3147 (6)	5316 (5)	29
C(23)	−2538 (5)	3963 (6)	4655 (5)	28
C(24)	−3378 (4)	4385 (6)	5450 (5)	25
C(25)	−3137 (5)	3974 (6)	6882 (5)	29
C(26)	−2005 (5)	3181 (6)	7505 (5)	30
O(21)	−4750 (4)	5660 (6)	3528 (4)	46
O(22)	−5283 (4)	5689 (6)	5528 (4)	48
N(22)	−4566 (4)	5317 (5)	4781 (4)	31
O(31)	−1531 (3)	−716 (4)	7600 (3)	30
O(32)	−2937 (4)	−1937 (6)	5935 (6)	66
O(33)	−1276 (4)	−782 (6)	5446 (3)	48
N(3)	−1933 (4)	−1165 (5)	6265 (4)	34
O(41)	846 (4)	−2123 (4)	9047 (4)	51
O(42)	80 (5)	−4483 (5)	9374 (4)	54
O(43)	−30 (4)	−3682 (6)	7246 (4)	50
N(4)	272 (4)	−3452 (5)	8551 (4)	28

* This parameter was used to define the unit-cell origin along y and is listed without e.s.d.

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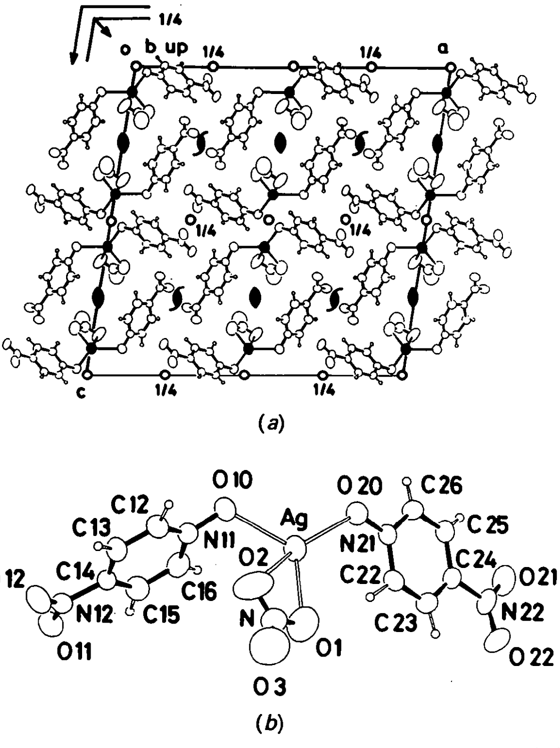
* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms and those in the 4-nitropyridine *N*-oxide moieties and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52539 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected interatomic distances* (Å) *and angles* (°)

(I)			
Ag—O(10)	2·364 (3)	O(20)—N(21)	1·306 (4)
Ag—O(20)	2·318 (3)	O(21)—N(22)	1·228 (6)
Ag—O(1)	2·453 (5)	O(22)—N(22)	1·216 (5)
Ag—O(2)	2·573 (6)	O(1)—N	1·232 (7)
O(10)—N(11)	1·301 (7)	O(2)—N	1·181 (7)
O(11)—N(12)	1·217 (6)	O(3)—N	1·212 (9)
O(12)—N(12)	1·219 (7)		
O(10)—Ag—O(20)	117·7 (1)	Ag—O(1)—N	100·1 (3)
O(10)—Ag—O(1)	121·6 (1)	Ag—O(2)—N	95·5 (4)
O(10)—Ag—O(2)	78·3 (1)	O(11)—N(12)—O(12)	124·6 (5)
O(20)—Ag—O(1)	120·7 (1)	O(21)—N(22)—O(22)	123·2 (3)
O(20)—Ag—O(2)	153·9 (2)	O(1)—N—O(2)	116·3 (5)
O(1)—Ag—O(2)	48·1 (2)	O(1)—N—O(3)	125·4 (6)
Ag—O(10)—N(11)	118·0 (3)	O(2)—N—O(3)	117·7 (6)
Ag—O(20)—N(21)	125·2 (2)		
(II)			
Cu—O(10)	1·935 (3)	O(21)—N(22)	1·213 (6)
Cu—O(20)	1·910 (3)	O(22)—N(22)	1·218 (6)
Cu—O(31)	1·939 (3)	O(31)—N(3)	1·308 (5)
Cu—O(41)	1·963 (3)	O(32)—N(3)	1·212 (6)
O(10)—N(11)	1·335 (5)	O(33)—N(3)	1·226 (6)
O(11)—N(12)	1·213 (6)	O(41)—N(4)	1·274 (5)
O(12)—N(12)	1·226 (6)	O(42)—N(4)	1·210 (6)
O(20)—N(21)	1·335 (5)	O(43)—N(4)	1·238 (5)
O(10)—Cu—O(20)	87·2 (1)	Cu—O(41)—N(4)	120·6 (3)
O(10)—Cu—O(31)	175·1 (1)	O(11)—N(12)—O(12)	124·2 (5)
O(10)—Cu—O(41)	83·4 (1)	O(21)—N(22)—O(22)	124·9 (4)
O(20)—Cu—O(31)	97·2 (1)	O(31)—N(3)—O(32)	116·8 (4)
O(20)—Cu—O(41)	170·6 (1)	O(31)—N(3)—O(33)	118·2 (4)
O(31)—Cu—O(41)	92·2 (1)	O(32)—N(3)—O(33)	124·9 (4)
Cu—O(10)—N(11)	119·8 (3)	O(41)—N(4)—O(42)	118·9 (4)
Cu—O(20)—N(21)	124·8 (3)	O(41)—N(4)—O(43)	119·3 (4)
Cu—O(31)—N(3)	114·0 (3)	O(42)—N(4)—O(43)	121·7 (4)

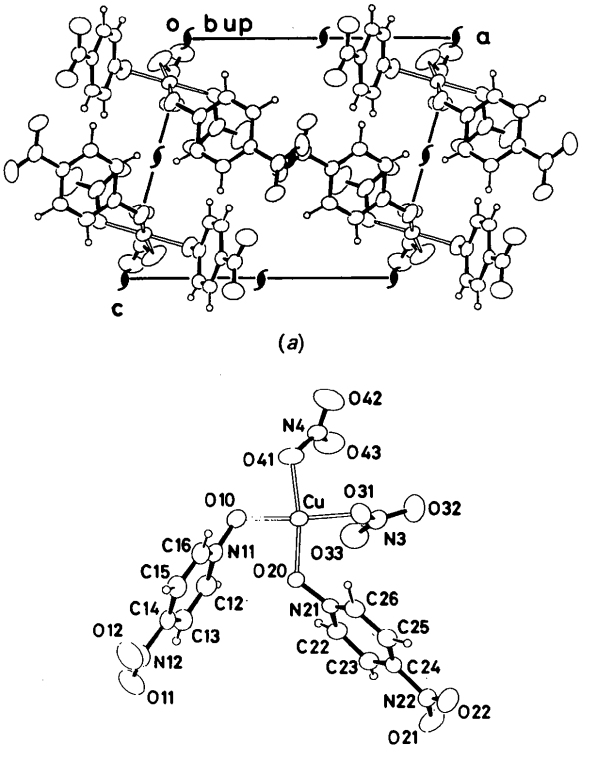
distance of the *N*-oxide moiety, 1·335 (5) Å, of (II) is significantly longer than that of 4-nitropyridine *N*-oxide, 1·291 (2) Å (Coppens & Lehmann, 1976) and that of (I), 1·304 (7) Å. It suggests that the coordination bond in (II) is stronger than in (I). This fact is supported by the longer N—O bond distances involving the coordinated nitrate O atoms, O(31) and O(41), than the others in (II). No close contact of the metal cations to the nitro group is observed. This fact indicates that the attractive interaction of the nitro group with the metal cation is much weaker than that of the *N*-oxide O atoms. However, the reason is not clear. An *ab initio* molecular orbital calculation using the STO-6G basis set showed that nitro and *N*-oxide O atoms carry almost the same effective charge of −0·22 e. The dipole moment of the 4-nitropyridine *N*-oxide is almost zero within the experimental error (Katritzky, Randall & Sutton, 1957). An attempt was also made to prepare the complexes containing *p*-dinitrobenzene. However, the complex could not be isolated. Therefore, the neutral nitro group does not seem to be able to coordinate to the metal atom in contrast to the nitrite ion (Aoyama, Ohba & Saito, 1988, and references therein), although the nitro group can act as a hydrogen-bond acceptor. For example, weak



(*a*)

(*b*)

Fig. 1. (*a*) Projection of the crystal structure of (I) along **b** and (*b*) structure of the complex with thermal ellipsoids scaled at the 50% probability level. H atoms are represented by circles of radius 0·08 Å.



(*a*)

(*b*)

Fig. 2. (*a*) Projection of crystal structure of (II) along **b** and (*b*) structure of the complex.

N—H···O hydrogen bonds involving the nitro O atom are observed in *p*-nitrobenzamide (Di Rienzo, Domenicano & Serantoni, 1977).

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Structure of $[Pt(C_6H_5)_2(btz\text{-}N,N')].CHCl_3$, btz = 2,2′-Bi-5,6-dihydro-4*H*-1,3-thiazine

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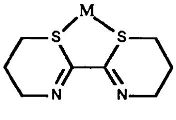
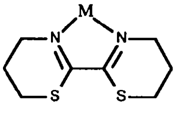
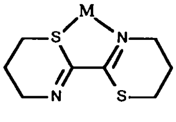
Abstract. (2,2′-Bi-5,6-dihydro-4*H*-1,3-thiazine-*N*,*N*′)-diphenylplatinum trichloromethane solvate, $[Pt(C_6H_5)_2(C_8H_{12}N_2S_2)].CHCl_3$, $M_r = 669{\cdot}0$, monoclinic, $P2_1/n$, $a = 14{\cdot}945\,(2)$, $b = 9{\cdot}841\,(1)$, $c = 16{\cdot}873\,(3)$ Å, $\beta = 109{\cdot}8\,(1)°$, $V = 2334{\cdot}7\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}90$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 6{\cdot}260$ mm^{-1}, $F(000) = 1296$, $T = 298$ K, final $R = 0{\cdot}036$ and $wR = 0{\cdot}038$ for 3123 independent reflections $[I > 3\sigma(I)]$. The $[Pt(C_6H_5)_2(btz\text{-}N,N')]$ complex has square-planar geometry about the Pt atom, with the btz coordinated to the Pt atom through the N atoms of the dihydrothiazine. The average Pt—N and Pt—C bond distances are 2·089 (6) and 2·008 (7) Å. In the solid state there is an interaction between the H atom of the chloroform molecule and the metal atom [Pt···H = 2·48 (1) Å].

Introduction. 2,2′-Bi-5,6-dihydro-4*H*-1,3-thiazine (btz) is a heterocyclic ligand with three possible coordination modes:







Its coordinating properties and those of the parent ligand 2,2′-bi-4,5-dihydrothiazole have been widely investigated (Nelson, Nelson & Perry, 1976). It has been shown that the chelation occurs *via* the α-di-imine group, except in a copper(I) derivative where the ligand is bound to both S and N atoms (Drew, Pearson, Murphy & Nelson, 1983).

We have prepared the title compound in order to achieve a mononuclear precursor for bimetallic systems. The X-ray analysis was undertaken to clarify the coordination mode of the 2,2′-bi-4,5-dihydrothiazine.

Experimental. The title compound was obtained by mixing, in chloroform, equimolecular quantities of *cis*-$[Pt(C_6H_5)_2(Me_2SO)_2]$ and btz. Red prismatic crystals suitable for X-ray investigation were obtained by slow evaporation from a chloroform–hexane solution.

A quasi-spherical single crystal with approximate dimensions 0·15 × 0·15 × 0·16 mm was used for intensity-data collection. Accurate unit-cell dimensions were obtained from least-squares refinement of the angular setting of 20 strong reflections in the range $14 < 2\theta < 30°$. Siemens–Stoe four-circle diffractometer using graphite-monochromated Mo $K\alpha$ radiation, ω–2θ mode, $2\theta_{\max} = 50°$ $(0 \le h \le 13,\ -14 \le k \le 14,\ -16 \le l \le 16)$. Four standard reflections

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