structure: *SHELXL*93. Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1199). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 2409–2412

Addition of Methanol to Copper(II)-Coordinated Dicyanonitrosomethanide: (3-Amino-3-methoxy-2-nitrosoacrylonitrilato-N^2,N^3)(bipyridine-*N*,*N'*)(dicyanamido-*N*)copper(II)

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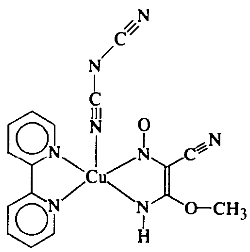
(*Received 29 March 1996; accepted 19 July 1996*)

Abstract

A new ligand formation was observed in the title complex, $[Cu(C_4H_4N_3O_2)\{N(CN)_2\}(C_{10}H_8N_2)]$, as a result of the nucleophilic addition reaction between methanol and dicyanonitrosomethanide in the inner coordination sphere of a Cu^{II} atom. This new ligand, 3-amino-3-methoxy-2-nitrosoacrylonitrilate (add), $[NHC(OMe)C(CN)NO]^-$, is coordinated as a chelate, forming a five-membered metallocycle with Cu^{II}. The distorted tetragonal-pyramidal coordination is completed by one chelate-coordinated molecule of 2,2′-bipyridine (bipy) and one end-coordinated dicyanamide anion, $[N(CN)_2]^-$. The four N atoms of the two metallocycles form the base and a fifth N atom, from the dicyanamide anion, forms the apex of the pyramid, creating a $\{CuN_5\}$ chromophore. The structure of $[Cu(add)(bipy)\{N(CN)_2\}]$ consists of discrete molecules.

Comment

The title compound, (I), was prepared and its crystal structure solved as a part of our study concerning changes in the shape of the $[Cu(N,N\text{-chel})_2X]^+$ cation resulting from alteration of the out-of-sphere anion Y^- [*N*,*N*-chel = 1,10-phenanthroline (phen) or 2,2′-bipyridine (bipy), X^- = the linear and non-linear pseudo-halide anions cyanide(1−), thiocyanate(1−) and dicyanamide(1−), and Y^- = the non-linear pseudo-halide anions tricyanomethanide(1−) and dicyanonitrosomethanide(1−)].



(I)

We have recently published several structures of Cu^{II} coordination compounds with phen, X^- and Y^-; $[Cu(phen)_2\{N(CN)_2\}_2]$ [(II); Potočňák, Dunaj-Jurčo, Mikloš, Kabešová & Jäger, 1995*a*], $[Cu(phen)_2\{N(CN)_2\}][C(CN)_3]$ [(III); Potočňák, Dunaj-Jurčo, Mikloš & Jäger, 1996*c*], $[Cu(phen)_2(NCS)][C(CN)_3]$ [(IV); Potočňák, Dunaj-Jurčo, Mikloš & Jäger, 1996*b*], $[Cu(phen)_2(CN)][C(CN)_3].2H_2O$ [(V); Potočňák, Dunaj-Jurčo, Mikloš & Jäger, 1996*a*] and $[Cu(phen)_2(NCS)][ONC(CN)_2]$ [(VI); Potočňák, Dunaj-Jurčo, Mikloš, Kabešová & Jäger, 1995*b*]. Dicyanamide anions complete the sixfold coordination of Cu^{II} in compound (II) and the fivefold coordination of Cu^{II} in (III). Tricyanomethanide and dicyanonitrosomethanide do not enter the inner coordination sphere of Cu^{II} in any of the above compounds. Also, no creation of new ligands in the coordination sphere of Cu^{II} has been observed in the above compounds.

On the other hand, when phen was replaced by bipy we obtained the title compound, $[Cu(bipy)\{CH_3OH.$-

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ISSN 0108-2701 © 1996

$ONC(CN)_2\}\{N(CN)_2\}]$, (I), where $CH_3OH.ONC(CN)_2$ represents the adduct of methanol and dicyanonitrosomethanide, which was created by nucleophilic addition of the methanol molecule to the coordinated dicyanonitrosomethanide, *i.e.* to the β-C site of the non-linear pseudo-halide, activated by its coordination to Cu^{II}. The same reaction between the above molecules was described for the coordination of Ni^{II} in $[Ni\{CH_3OH.ONC(CN)_2\}_2(H_2O)_2]$ [(VII); Hvastijová, Kožíšek & Kohout, 1995]. To confirm the hypothesis that the nucleophilic addition reaction described above resulted in compound (I), we performed the X-ray structure determination of the product obtained.

The crystal structure of (I) consists of discrete molecules held together by van der Waals forces. A molecule of (I) with the atomic labelling scheme is shown in Fig. 1.

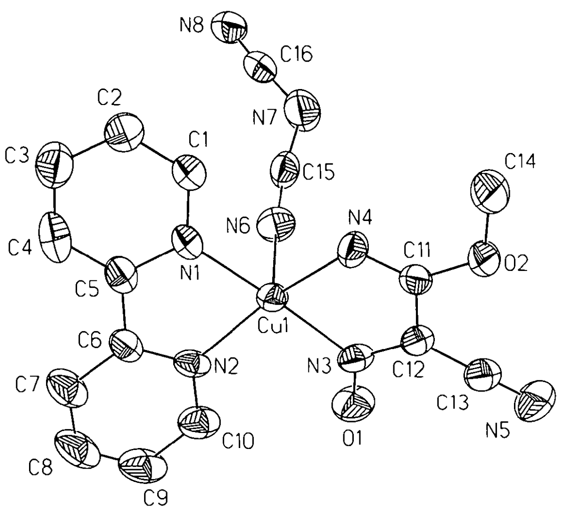

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the formula unit of (I) showing the labelling of the atoms. Displacement ellipsoids are plotted at the 40% probability level.

The Cu^{II} atom is fivefold coordinated by one bipy molecule, one add(1−) anion and one $[N(CN)_2]^-$ anion. The bipy and add(1−) ligands form two five-membered metallocycles with Cu^{II}. The distorted tetragonal-pyramidal coordination is completed by one end-bonded dicyanamide anion. The trigonality criterion τ, which gives values of 100 and 0 for an ideal trigonal bipyramid and an ideal tetragonal pyramid, respectively (Addison, Rao, Reedijk, van Rijn & Verschoor, 1984) is 18.7 for compound (I) and 69.2 for (III). Both pentacoordinate moieties in (I) and (III) are very similar with respect to their composition, *i.e.* they have $\{CuN_5\}$ chromophores, two five-membered metallocycles and one end-bonded $[N(CN)_2]^-$ ligand, but the coordination polyhedra are of different types. In compound (I), the coordination polyhedron is a distorted tetragonal pyramid, while in (III), it is a distorted trigonal bipyramid. This difference is caused by the rigidity of the phenanthroline molecule and the flexibility of the bipyridine molecule. The distances in the add(1−) ligand of the Cu(add), (I), and Ni(add), (VII), metallocycles may be considered to be the same [N3—C12 1.314 (5) and 1.310 (4) Å, N4—C11 1.264 (6) and 1.265 (4) Å, C13—N5 1.142 (6) and 1.130 (5) Å, C14—O2 1.430 (7) and 1.443 (4) Å, in compounds (I) and (VII), respectively], except for the distances C11—C12 [1.435 (6) in (I) and 1.471 (4) Å in (VII)] and C12—C13 [1.408 (6) in (I) and 1.425 (5) Å in (VII)], which are significantly shorter in (I) due to the increased bond multiplicity.

The mode of bonding in the dicyanamide ligand and its bond to the Cu^{II} atom in compound (I) may be compared with those in compound (III) [data for compound (III) are given in square brackets]: N6—C15 1.133 (6) [1.126 (6)] and N8—C16 1.119 (6) Å [1.123 (6) Å] for the cyano groups, and N7—C15 1.291 (7) [1.282 (7)] and N7—C16 1.294 (7) Å [1.282 (6) Å] for the amide bonds. The lengths of all corresponding bonds in the dicyanamide ligands in (I) and (III) are equal within 2σ. The expected values for the single, double and triple N—C bonds are 1.345, 1.270 and 1.153 Å, respectively. The observed values in compounds (I) and (III) correspond to double and triple N—C bonds; there is no single N—C bond present. Therefore, none of the three canonical formulae proposed to describe the mode of bonding in the dicyanamide ligand (Golub, Köhler & Skopenko, 1986) describes properly the bonding mode in the dicyanamide ligands in compounds (I) and (III). Higher multiplicity of the amide N—C bonds is supported by the fact that the whole dicyanamide ligand is planar in (I) and (III) [the largest deviation from the mean plane being 0.011 (6) for (I) and 0.003 (6) Å for (III)]. According to Golub, Köhler & Skopenko (1986), the bonding mode of $[N(CN)_2]^-$ to Cu^{II} can be considered as angular [Cu—N6—C15 145.4 (4) for (I) and 159.0 (4)° for (III)], with Cu—N6 distances of 2.188 (4) and 1.982 (4) Å, and C15—N7—C16 angles of 122.1 (5) and 128 (4)°, for (I) and (III), respectively].

Experimental

Crystals of (I) were prepared by mixing 10 ml of a 0.1 *M* water solution of $Cu(NO_3)_2$ with 20 ml of a 0.1 *M* methanol solution of bipy. A mixture of 5 ml of a 0.2 *M* water solution of $KN(CN)_2$ and 5 ml of a 0.2 *M* water solution of $NaONC(CN)_2$ was added. Dark green prismatic crystals appeared after two weeks. The density D_m was measured by flotation in benzene/bromoform solution.

Crystal data

$[Cu(C_4H_4N_3O_2)(C_2N_3)(C_{10}H_8N_2)]$	Mo $K\alpha$ radiation
$M_r = 411.88$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 21 reflections
$P2_1/n$	$\theta = 5.55–9.60°$

$a = 9.1800(9)$ Å
$b = 7.3440(7)$ Å
$c = 25.385(3)$ Å
$\beta = 91.22(2)°$
$V = 1711.0(3)$ Å^3
$Z = 4$
$D_x = 1.599$ Mg m^{-3}
$D_m = 1.579$ Mg m^{-3}

$\mu = 1.308$ mm^{-1}
$T = 293(2)$ K
Prism
$0.50 \times 0.35 \times 0.30$ mm
Dark green

Data collection

Syntex $P2_1$ diffractometer
θ–2θ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.546$, $T_{max} = 0.749$
3189 measured reflections
3002 independent reflections
1675 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.0516$
$\theta_{max} = 25°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 9$
$l = -32 \rightarrow 33$
2 standard reflections monitored every 98 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0436$
$wR(F^2) = 0.1078$
$S = 0.929$
2983 reflections
292 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.002$
$\Delta\rho_{max} = 0.451$ e Å^{-3}
$\Delta\rho_{min} = -0.374$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu1	0.18911 (6)	0.21593 (7)	0.36308 (2)	0.0490 (2)
O1	0.2323 (4)	0.5653 (5)	0.30539 (14)	0.0775 (11)
O2	0.5433 (3)	0.0981 (4)	0.27650 (12)	0.0581 (9)
N1	0.0948 (4)	0.0113 (5)	0.40278 (14)	0.0506 (10)
N2	−0.0116 (4)	0.3210 (5)	0.37086 (13)	0.0481 (10)
N3	0.2730 (4)	0.4030 (5)	0.31278 (14)	0.0525 (10)
N4	0.3495 (5)	0.0738 (7)	0.3344 (2)	0.0570 (13)
N5	0.5250 (5)	0.5174 (7)	0.2190 (2)	0.089 (2)
N6	0.3054 (4)	0.3252 (6)	0.4323 (2)	0.0676 (12)
N7	0.4995 (5)	0.2646 (9)	0.4958 (2)	0.115 (2)
N8	0.4826 (4)	0.1999 (7)	0.5885 (2)	0.0794 (13)
C1	0.1601 (6)	−0.1395 (8)	0.4201 (2)	0.0630 (14)
C2	0.0921 (6)	−0.2653 (8)	0.4503 (2)	0.071 (2)
C3	−0.0474 (6)	−0.2373 (9)	0.4643 (2)	0.076 (2)
C4	−0.1161 (6)	−0.0838 (9)	0.4465 (2)	0.067 (2)
C5	−0.0437 (5)	0.0406 (7)	0.4159 (2)	0.0491 (12)
C6	−0.1057 (4)	0.2106 (7)	0.3952 (2)	0.0517 (12)
C7	−0.2496 (6)	0.2559 (10)	0.4003 (2)	0.073 (2)
C8	−0.2983 (8)	0.4161 (11)	0.3795 (3)	0.086 (2)
C9	−0.2052 (8)	0.5271 (10)	0.3549 (2)	0.082 (2)
C10	−0.0613 (6)	0.4746 (8)	0.3510 (2)	0.0673 (15)
C11	0.4259 (5)	0.1548 (6)	0.3008 (2)	0.0441 (11)
C12	0.3843 (5)	0.3375 (6)	0.2872 (2)	0.0475 (12)
C13	0.4612 (5)	0.4380 (7)	0.2496 (2)	0.0597 (13)
C14	0.5900 (9)	−0.0848 (11)	0.2862 (3)	0.089 (2)
C15	0.3906 (5)	0.2967 (7)	0.4641 (2)	0.0624 (13)
C16	0.4824 (5)	0.2287 (7)	0.5452 (2)	0.0622 (13)

Table 2. *Selected geometric parameters* (Å, °)

Cu1—N4	1.958 (4)	N2—C6	1.344 (6)
Cu1—N2	2.011 (3)	N3—C12	1.314 (5)
Cu1—N1	2.015 (4)	N4—C11	1.264 (6)
Cu1—N3	2.038 (4)	N5—C13	1.142 (6)
Cu1—N6	2.188 (4)	N6—C15	1.133 (6)
O1—N3	1.262 (4)	N7—C15	1.291 (7)
O2—C11	1.321 (5)	N7—C16	1.294 (7)
O2—C14	1.430 (7)	N8—C16	1.119 (6)
N1—C1	1.330 (6)	C5—C6	1.464 (6)
N1—C5	1.338 (5)	C11—C12	1.435 (6)
N2—C10	1.313 (6)	C12—C13	1.408 (6)
N4—Cu1—N2	159.9 (2)	O1—N3—C12	120.2 (4)
N4—Cu1—N1	97.1 (2)	O1—N3—Cu1	128.0 (3)
N2—Cu1—N1	80.38 (15)	C12—N3—Cu1	111.8 (3)
N4—Cu1—N3	80.2 (2)	C11—N4—Cu1	115.6 (4)
N2—Cu1—N3	99.3 (2)	C15—N6—Cu1	145.4 (4)
N1—Cu1—N3	171.22 (15)	C15—N7—C16	122.1 (5)
N4—Cu1—N6	97.8 (2)	N1—C5—C6	114.4 (4)
N2—Cu1—N6	102.28 (15)	N2—C6—C5	115.5 (4)
N1—Cu1—N6	94.5 (2)	N4—C11—O2	129.6 (4)
N3—Cu1—N6	94.1 (2)	N4—C11—C12	117.0 (4)
C11—O2—C14	117.4 (4)	O2—C11—C12	113.4 (4)
C1—N1—C5	118.4 (4)	N3—C12—C13	123.3 (4)
C1—N1—Cu1	126.2 (3)	N3—C12—C11	115.4 (4)
C5—N1—Cu1	115.1 (3)	C13—C12—C11	121.3 (4)
C10—N2—C6	118.2 (4)	N5—C13—C12	178.9 (6)
C10—N2—Cu1	127.1 (4)	N6—C15—N7	172.8 (5)
C6—N2—Cu1	114.3 (3)	N8—C16—N7	172.9 (5)

Data collection and cell refinement were carried out using Syntex $P2_1$ software. Intensities were corrected for Lorentz and polarization factors using *XP*21 (Pavelčík, 1993). The structure was solved by the heavy-atom method with *XFPS* (Pavelčík, Rizzoli & Andreetti, 1990) and subsequent Fourier syntheses using *SHELXL*93 (Sheldrick, 1993). Anisotropic displacement parameters were refined for all non-H atoms. All H atoms were included in the refinement in calculated positions and then independently isotropically refined. Geometric analysis was performed using *PARST* (Nardelli, 1983) and *SHELXL*93. *ORTEP* (Johnson, 1965) was employed to produce the figure.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1203). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 2412–2414

Perchlorotriphenylcarbenium Hexachloroantimonate(V)

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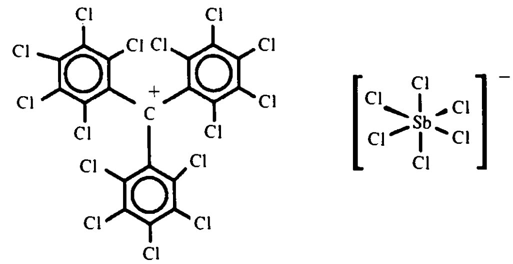
(*Received 12 January 1996; accepted 4 June 1996*)

Abstract

The title compound, $C_{19}Cl_{15}^{+}.SbCl_6^{-}$, consists of perchlorotriphenylcarbenium cations and hexachloroantimonate anions. The central Sb and C atoms occupy special positions of symmetry 32 along the *c* axis. The cation shows a symmetrical propeller conformation. Four Cl atoms of the $SbCl_6^{-}$ anion are disordered.

Comment

The so-called 'inert carbon-free radicals' belong mainly to the perchlorodiphenylmethyl (Ballester & Riera, 1954), perchlorotriphenylmethyl (Ballester, 1967; Ballester, Riera, Castañer, Badía & Monsó, 1971) and 9-phenylfluorenyl radical (Ballester, Castañer & Pujadas, 1971) classes. They have chemical and thermal stabilities which are higher than those of the majority of normal tetrahedral carbon compounds and materials. Such passivity is due to steric shielding of the molecular sites, where most of the radical reactivity normally resides (sites of high spin density), and to the strength of their valence bonds. Yet they are active in single-electron transfers yielding stable isolable carbenium (Ballester, Riera-Figueras & Rodríguez-Siurana, 1970) and carboanion (Ballester & De la Fuente, 1970) salts. As part of a systematic investigation of overcrowded polyhalogenated aromatic species, we report here the structural study of perchlorotriphenylcarbenium hexachloroantimonate, (I), obtained from the perchlorotriphenylmethyl radical by single-electron oxidation (Ballester *et al.*, 1982). This carbenium salt possesses a highly symmetrical structure.



(I)

The overall conformation of the perchlorotriphenylcarbenium ion (Fig. 1) is described by the angle between the mean planes of the aromatic rings [77.2 (2)°] and the angle between these planes and the plane defined by the C1 and three C2 atoms [46.1 (2)°]. The mean values of the bond lengths and angles in the pentachlorophenyl group are C—C 1.397 (7), C—Cl 1.700 (6) Å, C—C—C 119.9 (6) and C—C—Cl 119.9 (4)°. These values do not differ significantly from those found in other structures containing the carbenium cation (Miravitlles, Molins, Solans, Germain & Declercq, 1985; Veciana, Carilla, Miravitlles & Molins, 1987). The most significant difference is that in the present compound, the three perchlorophenyl groups of the cation are crystallographically equivalent, while in the cited structures, these three groups are not. In the present structure, the perchlorotriphenylcarbenium ion adopts the highest symmetrical conformation so far described. Due to the intercalated packing, the shortest intermolecular Cl···Cl interactions are between the anions and cations [Cl6···Cl7($x + \frac{1}{3}$, $y + \frac{2}{3}$, $z - \frac{1}{3}$) 3.121 (2) Å].

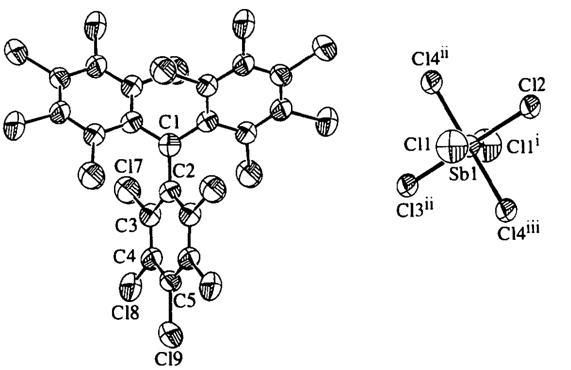


Fig. 1. The molecular structure and atomic labelling of the perchlorotriphenylcarbenium cation and hexachloroantimonate anion. Only one of the six components of the disordered $SbCl_6^{-}$ anion is shown [symmetry codes: (i) $y, x, -z$; (ii) $-y, x - y, z$; (iii) $-x, y - x, -z$]. Displacement ellipsoids are drawn at the 50% probability level.

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