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The Crystal Structure of Catena-tri-μ_2-(1,12-dodecanedinitrile)copper(II) Hexachloroantimonate(V), $Cu(C_{12}H_{20}N_2)_3(SbCl_6)_2$

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The crystal structure of $Cu(C_{12}H_{20}N_2)_3(SbCl_6)_2$ has been determined by single-crystal X-ray diffraction techniques. The compound is triclinic, space group $P\bar{1}$ with $a=8{\cdot}181$ (1), $b=13{\cdot}147$ (1), $c=14{\cdot}314$ (2) Å, $\alpha=112{\cdot}54$ (8), $\beta=99{\cdot}84$ (7), $\gamma=101{\cdot}24$ (6)° and $Z=1$. Low-temperature (100K) data to $(\sin\theta)/\lambda=0{\cdot}59$ Å^{-1} (Mo $K\alpha$ radiation) were collected with a three-circle diffractometer and the structure was solved by means of the Patterson synthesis and refined by least-squares methods to a final R value of 0·055 for the 4643 independent reflexions measured. The compound is a two-dimensional network, in which the Cu atoms are linked in two different ways by the ligands. The coordination polyhedron around copper shows Jahn–Teller deformation and consists of six N atoms.

Introduction

Previously it was shown (Zuur, Eversteyn & Groeneveld, 1975), that the C≡N stretching frequency of 2246 cm^{-1} in free 1,12-dodecanedinitrile is shifted towards higher frequencies (2271, 2299 cm^{-1}) in the complex $Cu(C_{12}H_{20}N_2)_3(SbCl_6)_2$. The absence of the non-bonded nitrile stretching frequency indicates that the ligand acts as a bidentate, in spite of its size and shape. There are three possible ways in which the ligand may act as a bidentate: by surrounding a single Cu atom, or by forming a two- or three-dimensional network. In order to get more information about the way the ligand is bonded the crystal structure of $Cu(C_{12}H_{20}N_2)_3(SbCl_6)_2$ has been determined.

Unit cell and space group

Crystals of the title compound were prepared as described previously (Zuur, Eversteyn & Groeneveld, 1975). Single crystals were obtained by slowly cooling a saturated solution in nitromethane, containing 20% ligand, from 20 to 0°C. The crystals decompose rapidly when exposed to air. Approximate cell parameters were determined from zero- and first-level Weissenberg photographs. The diffraction symmetry $\bar{1}$ points to the space group $P1$ or $P\bar{1}$. The initial choice of space group $P\bar{1}$ is justified by the consistency of the results. Precise unit-cell parameters were determined on a single-crystal diffractometer at 100K with Mo $K\alpha$ radiation ($\lambda=0{\cdot}71069$ Å). The parameters, $a=8{\cdot}181$ (1), $b=13{\cdot}147$ (1), $c=14{\cdot}314$ (2) Å, $\alpha=112{\cdot}54$ (8), $\beta=99{\cdot}84$ (7) and $\gamma=101{\cdot}24$ (6)° were obtained from θ, φ and χ measurements of 32 $h00$, $0k0$ and $00l$ reflexions. Due to the instability of the crystals it was not possible to determine the density. The calculated density for $Z=1$ and a molecular weight of 1342·4 is 1·620; $F(000)=653{\cdot}66$ on an absolute scale.

Collection and reduction of X-ray diffraction data

An irregular fragment of approximate size 0·6 × 0·14 × 0·25 mm was mounted in a glass capillary on a Nonius three-circle diffractometer. Intensities were recorded at 100K by the ω-scan method for all reflexions with θ between 4 and 35°. Mo $K\alpha$ radiation monochromatized by graphite was used. The scan width varied according to $\Delta\omega=1{\cdot}1+0{\cdot}64\tan\theta$. The mean counting time was 28 s for each background and 56 s for each scan. In all, 4998 reflexions were measured. After averaging the symmetry-related reflexions 4643 independent reflexions were obtained, including 232 having intensities smaller than twice their standard deviations σ_I. The σ_I's were calculated from counting statistics. The intensities were corrected for Lorentz and polarization effects. The linear absorption coefficient [μ(Mo $K\alpha$)= 20·4 cm^{-1}] is rather high. Due to the irregular shape of the crystal it was impossible to calculate precise transmission factors. For this reason no absorption correction was applied. An azimuth scan of the reflexion $\bar{2}41$ showed intensity variations from 85 to 115 (arbitrary scale). In accordance with these variations the σ_I's were recalculated with the formula $\sigma_I^2=\sigma_{st}^2+\sigma_{abs}^2+\sigma_{pol}^2$, where σ_{st} is the standard deviation from counting statistics, σ_{abs} the estimated standard error due to the neglect of the absorption effects and σ_{pol} the standard deviation due to variation in the intensity during the measurement. After reduction of the intensities to F values, a Wilson plot was calculated from which approximate values of the scale factor and the initial overall isotropic thermal parameter B were obtained.

Solution and refinement

The analysis of the three-dimensional Patterson synthesis gave positions for all atoms except H. A block-diagonal least-squares refinement of positional parameters and an overall thermal parameter for the 29 non-H atoms with the Cu atom at the centre of symmetry converged to $R=0·140$ for the reflexions greater than twice their standard deviations. Two additional cycles with individual isotropic thermal parameters resulted in $R=0·122$. Five more cycles with individual anisotropic thermal parameters led to $R=0·086$. A difference synthesis did not reveal all H atoms. For the following cycles of least-squares refinement the H atoms were placed on calculated positions at a C–H distance of 0·95 Å.

Three more cycles of block-diagonal least squares followed, refining the parameters of the non-H atoms and a single thermal parameter for the H atoms. After each cycle the positions of the H atoms were recalculated. At this stage ($R=0·0585$) extinction correction was applied to all 4683 independent reflexions. Three additional cycles of refinement, again recalculating the positions of the H's after each cycle, led to a final R value of 0·0555 ($R_w=0·1042$) for all 4643 independent reflexions. In the last cycle the calculated shifts were less than $\frac{1}{8}$ of the estimated standard deviations.

Atomic parameters are listed in Tables 1 and 2. The anisotropic thermal parameters for the non-H atoms are given in Table 3.* The final isotropic thermal par-

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31528 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional coordinates of the non-hydrogen atoms* ($\times 10^4$)

E.s.d.'s in the least significant digits are in parentheses for this and following tables. Special positions are marked with an asterisk. The numbering of the atoms is depicted in Figs. 1 and 2.

	x	y	z
Cu*	0	0	0
Cl(1)	3895 (2)	7722 (1)	8309 (1)
Cl(2)	2199 (2)	3702 (1)	7145 (1)
Cl(3)	291 (2)	5802 (1)	7955 (1)
Cl(4)	5851 (2)	5661 (1)	7539 (1)
Cl(5)	2102 (2)	5460 (1)	5975 (1)
Cl(6)	3959 (2)	5924 (1)	9469 (1)
Sb	3059 (0)	5711 (0)	7716 (0)
N(1,2)	240 (6)	1060 (4)	1500 (3)
N(1,1)	10909 (6)	1399 (4)	9801 (3)
N(3,2)	7291 (6)	247 (4)	9506 (3)
C(1,1)	11176 (6)	2262 (5)	9744 (3)
C(1,2)	11469 (7)	3342 (4)	9637 (3)
C(1,3)	9724 (7)	3475 (5)	9143 (4)
C(1,4)	8773 (7)	2521 (5)	8049 (4)
C(1,5)	7345 (7)	2830 (5)	7496 (4)
C(1,6)	6727 (7)	2079 (5)	6306 (4)
C(1,7)	5388 (9)	2469 (5)	5751 (4)
C(1,8)	4922 (10)	1796 (7)	4565 (5)
C(1,9)	3867 (9)	2316 (6)	3945 (5)
C(1,10)	2106 (10)	2281 (6)	4158 (5)
C(1,11)	1011 (9)	2621 (5)	3415 (4)
C(1,12)	549 (7)	1756 (5)	2325 (4)
C(3,7)	368 (6)	361 (4)	5585 (3)
C(3,8)	1872 (6)	14 (5)	6051 (3)
C(3,9)	2554 (6)	657 (5)	7247 (3)
C(3,10)	4049 (7)	270 (5)	7668 (3)
C(3,11)	4671 (7)	874 (5)	8857 (3)
C(3,12)	6153 (7)	529 (5)	9249 (3)

Table 2. *Fractional coordinates* (*calculated*) *of the hydrogen atoms* ($\times 10^3$)

The numbers in parentheses are those of the C atom to which the H atoms are attached.

	x	y	z
H1(1,2)	1197	397	1031
H2(1,2)	1212	335	920
H3(1,3)	899	349	959
H4(1,3)	995	419	910
H5(1,4)	959	238	765
H6(1,4)	828	184	811
H7(1,5)	637	276	776
H8(1,5)	774	361	758
H9(1,6)	769	211	603
H10(1,6)	622	132	620
H11(1,7)	436	237	598
H12(1,7)	584	327	593
H13(1,8)	596	177	436
H14(1,8)	425	104	438
H15(1,9)	450	309	414
H16(1,9)	371	188	321
H17(1,10)	154	152	405
H18(1,10)	226	280	487
H19(1,11)	−2	270	362
H20(1,11)	164	334	347
H21(3,7)	76	114	572
H22(3,7)	−53	27	592
H23(3,8)	150	−79	586
H24(3,8)	279	15	575
H25(3,9)	294	146	744
H26(3,9)	164	51	755
H27(3,10)	367	−53	745
H28(3,10)	498	45	739
H29(3,11)	500	168	908
H30(3,11)	375	67	913

Table 3. *Anisotropic temperature factors of non-hydrogen atoms* ($Å^2 \times 10^4$)

The general anisotropic temperature factor has the form $\exp[-2\pi^2(\sum_i\sum_j U_{ij}h_ih_ja_i^*a_j^*)]$.

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{31}$
Cu	216 (4)	198 (4)	164 (4)	169 (7)	133 (6)	−83 (6)
Cl(1)	595 (10)	200 (6)	298 (6)	181 (12)	154 (10)	74 (13)
Cl(2)	514 (9)	213 (6)	466 (8)	180 (12)	183 (12)	218 (14)
Cl(3)	242 (6)	544 (8)	379 (7)	432 (12)	538 (13)	103 (11)
Cl(4)	255 (7)	506 (8)	420 (8)	376 (12)	505 (13)	139 (12)
Cl(5)	370 (8)	497 (8)	218 (6)	214 (13)	231 (12)	−109 (11)
Cl(6)	296 (7)	400 (7)	276 (6)	215 (12)	350 (11)	−89 (11)
Sb	188 (2)	177 (2)	199 (2)	148 (3)	119 (3)	−48 (3)
N(1,2)	280 (20)	290 (20)	170 (20)	310 (40)	150 (40)	−80 (40)
N(1,1)	290 (20)	270 (20)	250 (20)	280 (40)	260 (40)	−120 (40)
N(3,2)	310 (20)	240 (20)	180 (20)	190 (40)	100 (30)	−110 (40)
C(1,1)	140 (20)	310 (30)	200 (30)	210 (40)	120 (40)	−100 (40)
C(1,2)	290 (30)	220 (20)	170 (20)	180 (40)	80 (40)	−100 (40)
C(1,3)	270 (30)	290 (30)	210 (30)	230 (40)	240 (40)	−20 (40)
C(1,4)	330 (30)	230 (30)	260 (30)	300 (40)	250 (40)	50 (40)
C(1,5)	340 (30)	330 (30)	260 (30)	240 (50)	230 (50)	−140 (50)
C(1,6)	300 (30)	380 (30)	260 (30)	370 (50)	170 (50)	−70 (50)
C(1,7)	410 (30)	460 (30)	250 (30)	490 (60)	150 (50)	−200 (50)
C(1,8)	530 (40)	730 (50)	220 (50)	800 (80)	40 (60)	−260 (60)
C(1,9)	440 (40)	630 (40)	350 (30)	400 (70)	350 (60)	10 (60)
C(1,10)	550 (40)	550 (40)	270 (30)	540 (70)	330 (60)	−12 (60)
C(1,11)	580 (40)	360 (30)	210 (30)	570 (60)	190 (50)	40 (50)
C(1,12)	340 (30)	270 (30)	260 (20)	420 (40)	320 (40)	120 (40)
C(3,7)	220 (20)	220 (20)	180 (20)	240 (40)	100 (40)	−90 (30)
C(3,8)	220 (20)	260 (20)	180 (20)	210 (40)	110 (40)	−60 (40)
C(3,9)	240 (30)	270 (20)	190 (20)	240 (40)	160 (40)	−70 (40)
C(3,10)	270 (30)	240 (20)	140 (20)	230 (40)	−20 (40)	−150 (40)
C(3,11)	320 (30)	310 (30)	190 (20)	260 (20)	210 (40)	−100 (40)
C(3,12)	360 (30)	230 (30)	140 (20)	240 (50)	80 (40)	−60 (40)

ameter of the H atoms was 2·58 $Å^2$. Scattering factors, taken from Cromer & Waber (1965) and from Stewart, Davidson & Simpson (1965), were used. Only the real part of the anomalous dispersion $\Delta f'$ was taken into account. The function minimized during the least-squares refinement was: $\sum w(|F_o|-|F_c|)^2$ with the weighting scheme $w=1/\sigma_F^2$. Agreement indices refer to: $R=\sum||F_o|-|F_c||/\sum|F_o|$ and $R_w=(\sum w(|F_o|-|F_c|)^2/\sum w|F_o|^2)^{1/2}$.

The molecular structure

Intramolecular distances and their estimated standard deviations (e.s.d.'s) are shown in Table 4 and bond

Table 4. *Intramolecular distances* (Å) *and their e.s.d.'s*

The atoms marked with ′ or ″ are related to the atoms with the same number by the centres of symmetry on $\frac{1}{2},0,\frac{1}{2}$ and $0,0,\frac{1}{2}$ respectively.

Sb——Cl(1)	2·357 (2)	C(1,4)—C(1,5)	1·507 (9)
Sb——Cl(2)	2·385 (2)	C(1,5)—C(1,6)	1·534 (9)
Sb——Cl(3)	2·365 (2)	C(1,6)—C(1,7)	1·526 (10)
Sb——Cl(4)	2·351 (2)	C(1,7)—C(1,8)	1·519 (11)
Sb——Cl(5)	2·352 (2)	C(1,8)—C(1,9)	1·560 (12)
Sb——Cl(6)	2·381 (2)	C(1,9)—C(1,10)	1·519 (11)
Cu——N(1,2)	2·011 (5)	C(1,10)-C(1,11)	1·526 (10)
Cu——N(1,1)′	1·989 (5)	C(1,11)-C(1,12)	1·463 (9)
Cu——N(3,2)′	2·331 (5)	C(3,12)-C(3,11)	1·471 (8)
N(1,1)-C(1,1)	1·150 (8)	C(3,11)-C(3,10)	1·516 (8)
N(1,2)-C(1,12)	1·129 (7)	C(3,10)-C(3,9)	1·530 (8)
N(3,2)-C(3,12)	1·124 (7)	C(3,9)—C(3,8)	1·529 (8)
C(1,1)-C(1,2)	1·463 (8)	C(3,8)—C(3,7)	1·529 (7)
C(1,2)-C(1,3)	1·554 (8)	C(3,7)—C(3,7)″	1·511 (10)
C(1,3)-C(1,4)	1·526 (8)		

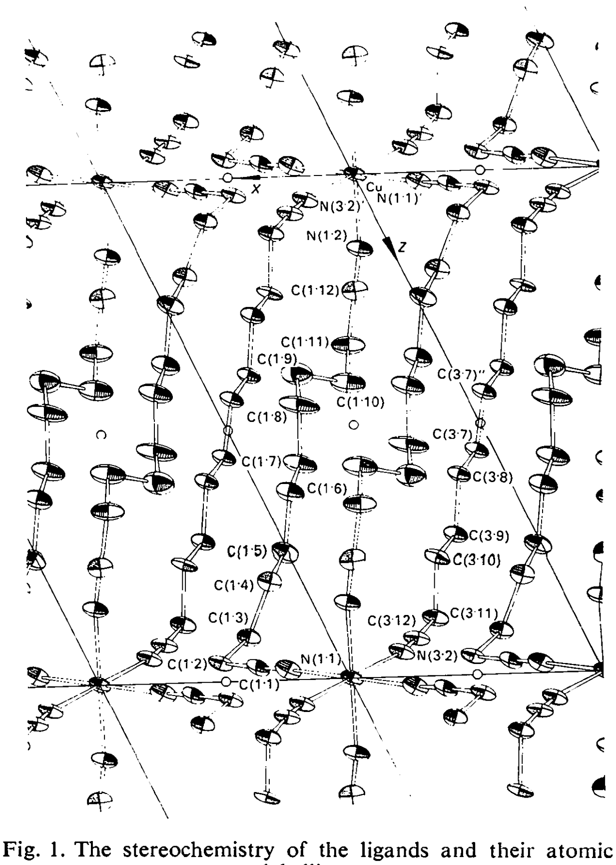

Fig. 1. The stereochemistry of the ligands and their atomic labelling.

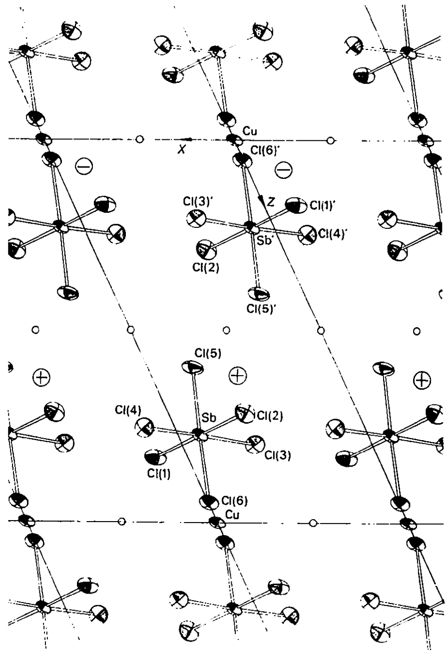

Fig. 2. The atomic labelling of the anions. Anions above the XZ plane are depicted with a plus sign, those below with a minus sign.

angles with e.s.d.'s in Table 5. The stereochemistry of the ligands and the atomic labelling are depicted in Figs. 1 and 2. Each Cu atom is surrounded by six N atoms. From the possibilities mentioned in the introduction we see in Fig. 1 that the ligands form a two-dimensional network linking the Cu atoms. The first two C chains are centrosymmetric to each other, the third is centrosymmetric in itself. The layers are parallel to the XZ plane of the unit cell. The space between two of these C–N–Cu layers is filled by two layers of $(SbCl_6)^-$ units related to each other by the centre of symmetry at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$, as shown in Fig. 2.

The coordination of Cu shows Jahn–Teller deformation, the Cu–N distances being 2·011 [N(1,2)], 1·989 [N(1,1)] and 2·331 Å [N(3,2)] respectively.

The authors are indebted to A. P. Zuur, C. Romers and J. Reedijk for their interest in this study. All crystallographic calculations were performed on the Leiden University IBM 370/158 computer.

Table 5. *Bond angles* (°) *of the* $(SbCl_6)^-$ *anion and* Cu *polyhedron and ligands*

(*a*) $(SbCl_6)^-$ anion (e.s.d.'s are 0·06°).

Cl(1)–Sb–Cl(2)	179·02
Cl(1)–Sb–Cl(3)	88·86
Cl(1)–Sb–Cl(4)	90·16
Cl(1)–Sb–Cl(5)	90·42
Cl(1)–Sb–Cl(6)	90·75
Cl(2)–Sb–Cl(3)	90·45
Cl(2)–Sb–Cl(4)	90·51
Cl(2)–Sb–Cl(5)	90·25
Cl(2)–Sb–Cl(6)	88·56
Cl(3)–Sb–Cl(4)	178·05
Cl(3)–Sb–Cl(5)	90·09
Cl(3)–Sb–Cl(6)	88·89
Cl(4)–Sb–Cl(5)	91·60
Cl(4)–Sb–Cl(6)	89·43
Cl(5)–Sb–Cl(6)	178·43

(*b*) Cu polyhedron and ligands

N(1,2)—Cu——N(1,1)	92·9 (2)
N(1,1)—Cu——N(3,2)	84·9 (2)
N(1,2)—Cu——N(3,2)	90·5 (2)
Cu——N(1,2)—C(1,12)	170·5 (5)
Cu——N(1,1)—C(1,1)	167·4 (5)
Cu——N(3,2)—C(3,12)	167·4 (5)
N(1,1)—C(1,1)—C(1,2)	178·1 (6)
C(1,1)—C(1,2)—C(1,3)	110·3 (5)
C(1,2)—C(1,3)—C(1,4)	114·0 (5)
C(1,3)—C(1,4)—C(1,5)	112·0 (5)
C(1,4)—C(1,5)—C(1,6)	113·2 (5)
C(1,5)—C(1,6)—C(1,7)	112·3 (5)
C(1,6)—C(1,7)—C(1,8)	111·7 (6)
C(1,7)—C(1,8)—C(1,9)	114·1 (6)
C(1,8)—C(1,9)—C(1,10)	111·2 (6)
C(1,9)—C(1,10)–C(1,11)	109·6 (6)
C(1,10)–C(1,11)–C(1,12)	111·5 (6)
C(1,11)–C(1,12)–N(1,2)	176·8 (6)
C(3,7)″–C(3,7)—C(3,8)	112·4 (5)
C(3,7)—C(3,8)—C(3,9)	114·0 (4)
C(3,8)—C(3,9)—C(3,10)	111·6 (4)
C(3,9)—C(3,10)–C(3,11)	111·4 (5)
C(3,10)–C(3,11)–C(3,12)	110·8 (5)
C(3,11)–C(3,12)–N(3,2)	177·1 (6)

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