hydrogens between the geminal methyl groups on one quaternary carbon and a third Cp hydrogen between the two CMe_2 groups on different pinacolate ligands. We find the same to be true of $(CpTiCl_2)_2O_2C_2Me_4$ when space-filling models are examined. This leads to the general conclusion that these molecules, containing both Cp and tertiary alkoxide ligands, are rather crowded.

We commented earlier that the Ti–O–C angle of 166.2° in $[CpTiCl_2]_2O_2C_2Me_4$ (Table IV) was the largest then recorded. Compound I provides two additional values, one a very large 170.9 (2)°, reinforcing the idea of very strong π-donation from oxygen. Additional large angles have been reported recently at the alkoxide oxygen of tartrate attached to Ti(IV).[5] Note that near-linearity at oxygen aggravates nonbonded repulsions between pinacol methyl groups and the Cp rings in I compared to what they might have been had the 10-membered ring in I adopted the conformation of cyclodecane.[4]

Finally, we return to the question of the driving force for the dimerization portrayed in eq 1. Why does the 5-membered ring of the kinetic product, $CpTiCl(\eta^2\text{-}O_2C_2Me_4)$, rearrange spontaneously (albeit not instantaneously) to a 10-membered ring? We suggest that it involves unfavorable nonbonded repulsions between the Cp ring and the two syn methyl groups in the monomer. It is evident from Figure 3 that such interactions are incipient in I. Since the O–Ti–O angle will be quite constrained in $CpTiCl(\eta^2\text{-}O_2C_2Me_4)$, the degrees of freedom in this 5-membered ring will be limited and Cp/methyl interactions will be increased. Constraints within the 5-membered ring will also have the effect of decreasing the Ti–O–C angle;[6] to the extent that π-donation by oxygen contributes thermodynamic stability (a point established by the structure we report here), this represents a second factor that explains the thermodynamics of eq 1.

Acknowledgment. We thank the National Science Foundation for support of this work and Scott Horn for skilled technical assistance.

Registry No. I, 76971-95-4; $CpTiCl(OCMe_2CMe_2O)$, 62422-44-0.

Supplementary Material Available: A listing of anisotropic thermal parameters (1 page); a listing of observed and calculated structure factors (27 pages). Ordering information is given on any current masthead page.

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Contribution from the Department of Chemistry,
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Crystal Structure and Electronic and EPR Spectra of a Compound Containing a Symmetric Square-Planar $CuCl_4^{2-}$ Ion

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Square-planar $CuCl_4^{2-}$ ions are exceedingly rare, notwithstanding the large number of ions with this stoichiometry that have been reported in the literature.[1,2] In fact most of the $CuCl_4^{2-}$ anions have distorted tetrahedral geometries with trans Cl–Cu–Cl angles close to 130°. The way to obtain isolated square-planar ions is to stabilize them through hydrogen bonds, and indeed, the only nearly idealized square-planar complexes reported so far have been obtained with organic cations that can form stable hydrogen bonds with the $CuCl_4^{2-}$ ions. However, although planar, these compounds have the ions slightly asymmetric, with two different Cu–Cl bond distances. This is an unfortunate circumstance, because the square-planar $CuCl_4^{2-}$ ion is a very interesting reference for the spectral properties of the tetrachlorocuprate complexes, which have been often used as models for copper(II) metalloproteins.

Table I. Crystallographic Data and Summary of Intensity Data Collection

(A) Crystallographic Parameters

cryst syst: monoclinic
space group: $P2_1/c$ (C_{2h}^5, No. 14) uniquely determined on the basis of systematic absences
molecular formula: $C_{14}H_{14}Cl_4CuN_4S_2$
$a = 7.053$ (2) Å $b = 9.747$ (2) Å
$c = 14.029$ (2) Å $\beta = 98.48$ (2)°
$V = 953.89$ Å^3 $Z = 2$
$d_{calcd} = 1.77$ g/cm^3 mol wt = 507.78
$F(000) = 510.0$ $d_{obsd} = 1.75$ g/cm^3 (by flotation)

(B) Measurement of Diffraction Data

radiation (λ, Å): graphite-monochromated Mo Kα (0.710 69)
reflcns measd: $\pm h,+k,+l$
scan type: ω–2θ
θ range: 2–27°
scan speed range: 2.1–5.5° min^{-1}
scan width: $(1.3 + 0.35 \tan\theta)$°
no. of std reflcns: 3 every 4 h (no significant changes)
no. of collcd reflcns: 2287
no. of obsd. reflcns: 1359 with $I > 3.0\sigma(I)$ (after merge, $R = 0.046$)
cryst size: 0.2 × 0.2 × 0.5 mm
abs coeff: 18.3 (abs cor applied: $0.99 < T_{factor} < 0.80$)

Table II. Final Positional Parameters

atom	*x/a*	*y/b*	*z/c*
Cu	0.0	0.0	0.0
Cl(1)	−0.1257 (3)	0.0429 (2)	0.1369 (1)
Cl(2)	−0.2239 (2)	0.1376 (2)	−0.0850 (1)
S	−0.1938 (3)	0.6256 (2)	0.1381 (1)
N(1)	−0.2154 (7)	0.4158 (5)	0.0271 (3)
N(2)	−0.1115 (8)	0.3620 (6)	0.1879 (4)
C(1)	−0.1694 (8)	0.4517 (6)	0.1198 (4)
C(2)	−0.2789 (8)	0.6497 (6)	0.0148 (4)
C(3)	−0.2768 (7)	0.5258 (6)	−0.0336 (4)
C(4)	−0.3335 (8)	0.5182 (7)	−0.1332 (4)
C(5)	−0.3924 (9)	0.6395 (8)	−0.1796 (5)
C(6)	−0.3981 (9)	0.7610 (8)	−0.1315 (5)
C(7)	−0.3417 (9)	0.7688 (7)	−0.0323 (5)

We have now isolated a nearly idealized square-planar $CuCl_4^{2-}$ complex with the organic cation 2-aminobenzothiazolium, which has four Cu–Cl bonds whose lengths are equal within experimental error, and we wish to report here the crystal structure and the electronic and EPR spectra.

Experimental Section

Complex Preparation. All chemicals were reagent grade and used as received. The complex was prepared by adding diethyl ether to a methanolic solution of 2-aminobenzothiazole (L) (2 mM), $CuCl_2 \cdot 2H_2O$ (1 mM) and HCl 37% (16 mM). After 24 h, single green crystals suitable for X-ray were collected by filtration, washed with methanol/HCl (v/v 90/10) and then diethyl ether, and dried in vacuo over silica gel. Anal. Calcd. for $C_{14}H_{14}CuN_4S_2Cl_4$: C, 33.12; H, 2.78; N, 11.03; S, 12.63. Found: C, 33.29; H, 2.77; N, 11.04; S, 12.58.

Spectral Measurements. The electronic spectrum of the solid compound was recorded as a mull transmission spectrum with a Cary 2300 Varian spectrophotometer. EPR spectra were recorded on a Varian E spectrometer on polycrystalline sample with diphenylpicrylhydrazyl (dpph, $g = 2.0036$) as the calibrating field marker.

X-ray Structure Determination. The intensity data were collected at room temperature on a CAD 4 Enraf-Nonius automatic diffractometer. The crystals were shaped as irregular prisms. Details on crystal data, intensity collection, and refinement are reported in Table I.

The structure was solved by conventional Patterson and Fourier methods and refined through full-matrix least-squares calculations, with $\sum w(|F_o| - |F_c|)^2$ being minimized. After isotropic refinement of nonhydrogen atoms, all the hydrogen atoms were located from difference Fourier maps; they were then treated as fixed contributors with assigned isotropic thermal parameters 1.0 Å^2 higher than those of the bonded

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Table III. Bond Distances (Å) and Bond Angles (deg) with Esd's in Parentheses

Cu–Cl(1)	2.269 (2)	N(1)–C(3)	1.397 (7)	C(3)–C(4)	1.398 (8)
Cu–Cl(2)	2.271 (1)	N(2)–C(1)	1.315 (8)	C(4)–C(5)	1.383 (9)
S–C(1)	1.726 (6)	C(2)–C(3)	1.386 (8)	C(5)–C(6)	1.366 (10)
S–C(2)	1.762 (6)	C(2)–C(7)	1.376 (9)	C(6)–C(7)	1.392 (9)
N(1)–C(1)	1.340 (7)				
Cl(2)–Cu–Cl(1)	90.4 (1)	C(2)–S–C(1)	90.7 (3)		
C(3)–N(1)–C(1)	113.6 (5)	C(2)–C(3)–N(1)	113.3 (5)		
N(1)–C(1)–S	112.7 (4)	C(4)–C(3)–N(1)	125.8 (5)		
N(2)–C(1)–S	124.9 (5)	C(4)–C(3)–C(2)	120.9 (6)		
N(2)–C(1)–N(1)	122.4 (6)	C(5)–C(4)–C(3)	116.5 (6)		
C(3)–C(2)–S	109.7 (4)	C(6)–C(5)–C(4)	122.5 (6)		
C(7)–C(2)–S	128.5 (5)	C(7)–C(6)–C(5)	121.1 (6)		
C(7)–C(2)–C(3)	121.8 (5)	C(6)–C(7)–C(2)	117.3 (6)		

atoms, in the subsequent anisotropic refinements of non-hydrogen atoms.

This model converged to $R = 0.045$ and $R_w = 0.049$, $w = 0.8/[\sigma^2(F) + 0.0003F_o^2]$. There was no evidence for secondary extinction. Complex neutral-atom scattering factors, including anomalous dispersion terms, were taken from ref 4; major calculations were carried out on a Vax 11/750 computer by using the SHELX 76 program package[5] and the ORTEP plotting program.[6]

Final fractional coordinates for non-hydrogen atoms are given in Table II. Bond distances and bond angles are given in Table III.

Results and Discussion

Description of the Structure. The crystal structure of $(HL)_2CuCl_4$, (HL = 2-aminobenzothiazolium monocation), Figure 1, consists of a tetrachlorocuprate anion and two protonated 2-aminobenzothiazole cations held together by a combination of hydrogen-bonded contacts and aromatic ring stacking interactions between the organic cations. The anion has crystallographically imposed planar geometry with the two independent Cu–Cl bond lengths equal within two standard deviations.

In chlorocuprates with known structures Cu–Cl bond distances fall in the range 2.18–2.30 Å.[7-15] The average value of 2.270 Å observed in the present complex is just the sum of the covalent radii (2.27 Å).[16] In the present complex the closest neighbor in axial positions is a carbon atom at 3.281 Å, which cannot be even regarded as a weak interaction.

Tetrahalogenocuprate(II) complexes generally assume distorted tetrahedral geometry, square-planar coordination being less common.[17] Some examples of discrete, planar $CuCl_4^{2-}$ are known;[18-21] only three of them are nearly idealized square planar without axial interactions,[22-24] as we find in our compound.

The two ring moieties of the HL cation are planar within +0.015 and +0.013 Å, respectively, for the benzene and the five-membered ring, and the dihedral angle between the two planes is 1.8°. The aminic nitrogen atom deviates 0.048 Å from the mean five-membered ring plane. Corresponding values have been obtained in the 2-(methylamino)benzothiazole free ligand.[25] The C–S distances of 1.726 and 1.762 Å can be considered as intermediate bonds between the pure single bond and the double bond.[25] Our bond distances and bond angles compare very well with those reported for 2-substituted benzothiazoles.[26,27] This means that the organic molecule is not affected by the complex formation.

Hydrogen bonds occur between the four chlorine atoms and the four nitrogen atoms of the two 2-aminobenzothiazolium cations, as depicted in Figure 1, leading to discrete $HL\cdots CuCl_4\cdots HL$ units. These discrete units are involved in aromatic ring stacking interactions of the type $\cdots HL'\cdots HL\cdots HL''\cdots$, which occur between symmetry related, and hence parallel, HL cations. The shortest interatomic $HL'\cdots HL$ and $HL\cdots HL''$ distances are 3.434 and 3.513 Å, respectively, and the distances between mean planes are 3.405 and 3.464 Å, respectively. The distances $N(1)\cdots Cl(2)$ and $N(2)\cdots Cl(1)$ are 3.15 and 3.22 Å, respectively, with $N–H\cdots Cl$ of 165°. These quite strong $Cl\cdots N$ interactions may be one of the factors contributing to the maximum distortion of the $CuCl_4^{2-}$ ion from the tetrahedral configuration.

As reported in the literature, the hydrogen-bonds and the cation dimension are decisive factors for the $CuCl_4^{2-}$ geometry; in fact with weak hydrogen bonds and small counterions a "flattened" tetrahedral coordination is observed.[9-12] In our compound, besides the hydrogen bonds, which involve all the chlorines, even the aromatic stacking interactions could be a further stabilizing factor for a planar $CuCl_4^{2-}$ unit, which is in any case uncommon. A dihedral angle of 132.8° is found between the cation mean plane and the coordination plane.

Electronic Spectrum. In the spectral region 20 000–5000 cm^{-1} there is one band, with a maximum at 13 500 cm^{-1} and two shoulders at 15 500 and 11 500 cm^{-1}, respectively. These bands can be assigned to d–d transitions by using data previously reported.[28] As compared to $(C_6H_5CH_2CH_2NH_2CH_3)CuCl_4$[29] all the bands are shifted to lower frequencies. The ground state is $x^2 - y^2$, and the order of the excited states is such that the 11 500-cm^{-1} band corresponds to the $x^2 - y^2 \rightarrow xy$ transition, while the 13 500 cm^{-1} band is the $x^2 - y^2 \rightarrow xz, yz$ transition and the band at 15,500 cm^{-1} corresponds to $x^2 - y^2 \rightarrow z^2$. The energies of these transitions can be expressed in the angular overlap model at the simplest level, as

$$E(xy) = 3e_\sigma - 4e_\pi$$

$$E(xz,yz) = 3e_\sigma - 2e_\pi$$

$$E(z^2) = 2e_\sigma$$

Using the first two equations, we calculate $e_\sigma = 5166$ cm^{-1} and $e_\pi = 1000$ cm^{-1}, in good agreement with the values previously reported for other square-planar $CuCl_4$ complexes. The e_π/e_σ ratio is here smaller than that previously used for pseudotetrahedral complexes, in which the metal chloride distance is shorter (223 pm vs 227 pm).[29]

The energy of the $x^2 - y^2 \rightarrow z^2$ transition is calculated as 10 232 cm^{-1} in this scheme, much smaller than the experimental value.

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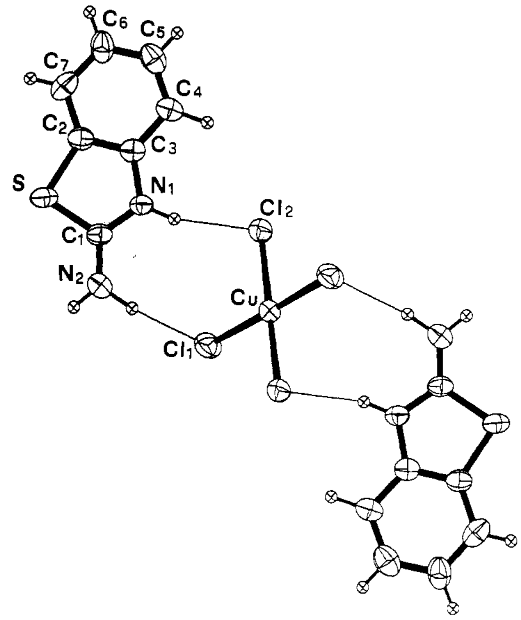

Figure 1. ORTEP view of the complex showing the atom numbering and thermal motion ellipsoids (50%) for the non-hydrogen atoms. The hydrogen atoms are represented as spheres of arbitrary radius.

This is a well-known result for square planar complexes when the ligand field models systematically underestimate the $x^2 - y^2 \rightarrow z^2$ separation. This has been attributed to d–s mixing, or to the residual electron density on the z axis, and it has been found to raise the energy of the $x^2 - y^2 \rightarrow z^2$ transition by 5000 cm^{-1} as in the present case.

The charge transfer bands are observed at 21 700 cm^{-1} as a shoulder, and at 26 100 cm^{-1}, respectively, comparing well with values previously reported, with essentially no change.[30]

EPR Spectrum. The EPR spectrum is axial; $g_{\parallel} = 2.13$, and $g_{\perp} = 2.05$. These values are crystal values, which correspond to averaged molecular values, due to the misalignment of the molecules in the unit cell and the presence of exchange narrowing effects, evidenced by the shape of the EPR line. If we assume axial symmetry for the chromophore, in agreement with the observed geometry of the $CuCl_4^{2-}$ anion, the molecular g values can be calculated and turn out to be $g_{\parallel} = 2.21$ and $g_{\perp} = 2.05$, in good accordance with the values previously reported for square-planar copper chloride complexes.[2] The fact that the two signals of the magnetically non-equivalent sites in the unit cell are averaged, indicates that the intermolecular exchange is larger than 0.02 cm^{-1}.[31]

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Registry No. $(HL)_2CuCl_4$, 114058-87-6.

Supplementary Material Available: Tables SI–SV, listing thermal parameters, positional and thermal parameters for hydrogen atoms, bond distances and angles involving hydrogen atoms, selected least-squares planes, and interatomic distances, respectively (5 pages); tables of calculated and observed structure factors (4 pages). Ordering information is given on any current masthead page.

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Tetranuclear Molybdenum Compounds Containing a μ_4-Disulfide Linkage

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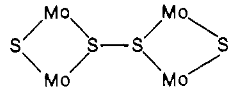
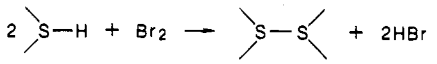
The addition of carboxylic acids[1] to an equilibrium mixture of cubane-like $[Mo(NC_6H_4CH_3)(\mu_3\text{-}S)(S_2P(OC_2H_5)_2)]_4$ and its dinuclear dissociation product[2] yields $[Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)]_2(\mu\text{-}S)(\mu\text{-}SH)(\mu\text{-}O_2CR)$ (R = alkyl). This type of compound is significant because of the bridging sulfhydryl ligand and because it resembles a thiol with moderate reactivity. Alkylation affords S-alkyl derivatives,[3] while oxidation with peroxides apparently leads[4] to products with the rare[5] μ_4-disulfide linkage



In this paper, we demonstrate alternate syntheses based on oxidation with Br_2 in CH_2Cl_2, we obtain the molecular structure of $[Mo_2(NC_6H_4CH_3)_2(S_2P(OC_2H_5)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)\cdot 2CH_2Cl_2$ by X-ray methods, and we explore the reduction of the μ_4-disulfide linkage with dithiocarbamate ions.

Results and Discussion

Synthesis. The preparation of $[Mo_2(NC_6H_4CH_3)_2(S_2P(OC_2H_5)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)$ (**1**) was accomplished easily by oxidizing the sulfhydryl-bridged compound with Br_2 in CH_2Cl_2:



The acid was scavenged with N,N-dimethylaniline. The selection of this base allowed a particularly easy separation of the desired compound from the ammonium salt. In contrast, this was not the case with other bases, such as triethylamine.

When the synthesis of $[Mo_2(NC_6H_4CH_3)_2(S_2CN(i\text{-}C_4H_9)_2)_2(\mu\text{-}S)(\mu\text{-}O_2CCF_3)]_2(\mu_4\text{-}S_2)$ (**2**) was attempted by this method, an infrared spectrum suggested that considerable hydrolysis of the arylimide ligands had occurred. Presumably, the water necessary for hydrolysis was present as an impurity in the trifluoroacetic acid. The acid-assisted replacement of arylimide groups with oxo ligands has been studied previously.[6] The problem did not occur, however, when the sulfhydryl-bridged compound was replaced with its conjugate base.

Structure. An ORTEP drawing of the molecular structure of **1** is shown in Figure 1. A crystallographically imposed center of inversion is located at the midpoint of the $\mu_4\text{-}S_2$ ligand. The drawing does not show the two molecules of CH_2Cl_2 that accompany each molybdenum-containing molecule even though they were located readily. This solvent of crystallization was not found during chemical analysis because it is easily lost and completely removed by vacuum. We are confident that the crystal selected for structural determination is representative of the bulk because it was taken from a sample that was very uniform in appearance.

Distances and angles within the dithiophosphate, trifluoroacetate, and arylimide ligands are unexceptional and do not merit

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