

1978). Full-matrix least squares with *SHELX76* (Sheldrick, 1976); refinement on *F*; anisotropic thermal parameters for non-H atoms except for the disordered C atom of minor component, C10B, which refined isotropically. Occupancy factors of the disordered atoms refined to 0.68 (C10A) and 0.32 (C10B). H atoms placed and fixed in calculated positions. Final agreement parameters: $R = 0.081$, $wR = 0.082$, $w = [\sigma^2(F_o) + 0.001F_o^2]^{-1}$, $(\Delta/\sigma)_{\max} = 0.17$, $\Delta\rho_{\max} = 0.71 \text{ e } \text{\AA}^{-3}$ around the Co atom. Atomic scattering factors including anomalous-dispersion term from *International Tables for X-ray Crystallography* (1974, Vol. IV). The somewhat large *R* value would be caused by the large thermal vibration and the disordered structure. All calculations were performed on a MicroVAX II computer.

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond distances and angles in Table 2. Fig. 1 shows the labelled

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54050 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

molecular structure, and Fig. 2 illustrates the crystal packing.

Related literature. Crystalline state racemization of the cyanoethyl groups has been reported (Ohashi, 1988). The structure of the present complex with a chiral cyanoethyl group has been published (Kojima, Iwasaki, Ohashi, Baba & Ohgo, 1991). The racemic structure produced by X-ray irradiation was compared with the title compound (Uchida, Kojima, Sekine, Ohashi, Ohgo & Baba, 1990).

References

- KOJIMA, Y., IWASAKI, S., OHASHI, Y., BABA, S. & OHGO, Y. (1991). *Acta Cryst.* **C47**, 300–303.
 MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
 OHASHI, Y. (1988). *Acc. Chem. Res.* **21**, 268–274.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 UCHIDA, A., KOJIMA, Y., SEKINE, A., OHASHI, Y., OHGO, Y. & BABA, S. (1990). *Acta Cryst.* **A46**, C179.

Acta Cryst. (1991). **C47**, 1973–1975

Hexamethylethylenediammonium Hexachlorodicuprate(II)

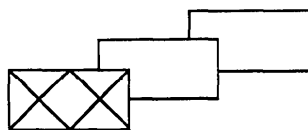
BY MARCUS R. BOND AND ROGER D. WILLETT

Department of Chemistry, Washington State University, Pullman, Washington 99164-4630, USA

(Received 2 July 1990; accepted 11 March 1991)

Abstract. $[\text{C}_8\text{H}_{22}\text{N}_2]^{2+} \cdot [\text{Cu}_2\text{Cl}_6]^{2-}$, $M_r = 486.1$, triclinic, $P\bar{1}$, $a = 6.254$ (1), $b = 8.269$ (2), $c = 9.753$ (2) Å, $\alpha = 103.96$ (2), $\beta = 103.67$ (2), $\gamma = 105.80$ (2)°, $V = 445.7$ (2) Å³, $Z = 1$, $D_x = 1.81 \text{ Mg m}^{-3}$, $\text{Cu } K\alpha$, $\lambda = 1.54182$ Å, $\mu = 11.32 \text{ mm}^{-1}$, $F(000) = 244$, $T = 295 \text{ K}$, $R = 0.049$ for 889 unique observed [$F \geq 3\sigma(F)$] reflections and 85 parameters. The structure consists of separate organic dication and stacks, (I), of quasi-planar

$\text{Cu}_2\text{Cl}_6^{2-}$ anions. The stacking arrangement is such that each Cu^{II} ion assumes a 4 + 1 coordination geometry. Within the dimer, terminal Cu—Cl distances average 2.264 Å and bridging distances average 2.318 Å while the interdimer semicoordinate Cu...Cl distance is 2.665 (1) Å. The bifold angle between the central Cu_2Cl_2 core and the folded CuCl_3 flap of the dimer is 19.1°.



(I)

Experimental. In an attempt to extend the study of the dynamic Jahn–Teller effect in ACuCl_3 salts containing tribridged chains we have sought to prepare analogs of $(\text{CH}_3)_4\text{NCuCl}_3$ (Weenk & Spek, 1976; Willett, Bond, Haije, Soonieus & Maaskant, 1988). One cation selected was the $\text{Me}_6\text{en}^{2+}$ dication

(Me₆en ≡ hexamethylethylenediammonium), which can be visualized as two Me₄N⁺ cations linked through a C—C bond. Red rod-like crystals of (Me₆en)Cu₂Cl₆ were obtained by slow evaporation of a 1:2 stoichiometric mixture of Me₆enCl₂ and CuCl₂·2H₂O dissolved in a hydrochloric acid solution. A block-shaped crystal with dimensions of 0.21 × 0.28 × 0.34 mm was selected for data collection on a Nicolet R3m diffractometer with graphite monochromator. Lattice constants from 25 reflections in the range 65 < 2θ < 69°. Data were collected with ω scans (1.3°); three check reflections monitored every 96 reflections (111, 110 and 011) showed no excursions beyond counting statistics; 1024 total reflections to 2θ = 100°, 915 unique with R_{int} = 0.058; hkl ranges 0 ≤ h ≤ 6, -8 ≤ k ≤ 8, -9 ≤ l ≤ 9 (Campana, Shepard & Litchman, 1981).

Data reduction, structure solution and refinement utilized the SHELXTL crystallographic program package (Sheldrick, 1985). Empirical ψ-scan absorption corrections applied with the routine XEMP assuming an ellipsoidally shaped crystal (0.073 < T < 0.208). A difference synthesis based on the Cu- and Cl-atom positions obtained from the direct methods routine SOLV yielded the C- and N-atom positions. H atoms were constrained to ideal locations (C—H = 0.96 Å) and group isotropic thermal parameters refined for the CH₃ and CH₂ protons. Non-H atoms were refined anisotropically.

The final refinement resulted in R = 0.049 (3σ data set) and 0.050 (all data), wR = 0.066 [|F| > 3σ(F)] and 0.066 (all data) and w = 1/[σ²(F) + g(F)²], with g = 0.00201. The function minimized was Σw(|F_o - |F_c||²). The goodness of fit was 1.39, with |Δσ|_{max} = 0.004 in the final cycle of least squares. The largest peak on the final difference map was 0.7 e Å⁻³ near Cu, while the most negative excursion was -0.9 e Å⁻³. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). An isotropic secondary-extinction correction of the form F* = F_c/[1.0 + 0.002g|F|²/sin(2θ)]^{0.25} with g refined to 0.032 (2) was included in the refinement. Atomic coordinates are listed in Table 1 and bond distances and angles are given in Table 2. A view of the structure is shown in Fig. 1.*

Related literature. The stacking pattern, (I), is just one of a series observed for Cu₂X₆²⁻ type species (Geiser, Willett, Lindbeck & Emerson, 1986; Bond & Willett, 1989). The bifold distortion has been

Table 1. Atomic coordinates (× 10⁴) and isotropic thermal parameters (Å² × 10³)

*U*_{eq} is defined as one third of the trace of the orthogonalized *U*_{ij} tensor.

	x	y	z	<i>U</i> _{eq}
Cu	-3108 (1)	4270 (1)	4185 (1)	28 (1)
Cl(1)	-674 (2)	2790 (2)	4845 (1)	37 (1)
Cl(2)	-4126 (2)	4398 (2)	6339 (1)	48 (1)
Cl(3)	-3413 (2)	3294 (2)	1754 (1)	42 (1)
N(1)	-308 (6)	-1193 (5)	11455 (4)	31 (2)
C(11)	-296 (11)	-2962 (7)	10640 (6)	54 (3)
C(12)	-2277 (8)	-1531 (6)	12113 (6)	42 (2)
C(13)	1939 (9)	-134 (8)	12699 (6)	52 (2)
C(1)	-877 (8)	-168 (6)	10433 (5)	38 (2)

Table 2. Bond distances (Å) and angles (°)

Cu—Cl(1)	2.272 (2)	N(1)—C(11)	1.490 (7)
Cu—Cl(2)	2.322 (2)	N(1)—C(12)	1.510 (7)
Cu—Cl(3)	2.256 (1)	N(1)—C(13)	1.492 (5)
Cu—Cl(2 ^a)	2.315 (2)	N(1)—C(1)	1.498 (7)
Cu—Cl(1 ^b)	2.665 (1)	C(1)—C(1 ⁱⁱⁱ)	1.542 (11)
Cl(1)—Cu—Cl(2)	90.6 (1)	Cu—Cl(2)—Cu ⁱ	96.6 (1)
Cl(1)—Cu—Cl(3)	92.8 (1)	Cu—Cl(1 ^a)—Cu ⁱⁱ	92.0 (1)
Cl(1)—Cu—Cl(2)	172.9 (1)	C(11)—N(1)—C(12)	107.1 (1)
Cl(1)—Cu—Cl(1 ^b)	88.0 (1)	C(11)—N(1)—C(13)	111.6 (4)
Cl(2)—Cu—Cl(3)	158.5 (1)	C(11)—N(1)—C(1)	112.3 (4)
Cl(2)—Cu—Cl(2)	83.4 (1)	C(12)—N(1)—C(13)	108.4 (4)
Cl(2)—Cu—Cl(1 ^b)	101.2 (1)	C(12)—N(1)—C(1)	105.8 (4)
Cl(3)—Cu—Cl(2)	91.4 (1)	C(13)—N(1)—C(1)	111.3 (4)
Cl(3)—Cu—Cl(1 ^b)	100.2 (1)	N(1)—C(1)—C(1 ⁱⁱⁱ)	112.6 (6)
Cl(2)—Cu—Cl(1 ^b)	96.9 (1)		

Symmetry operators: (i) -1 - x, 1 - y, 1 - z; (ii) -x, 1 - y, 1 - z; (iii) -x, -y, 2 - z.

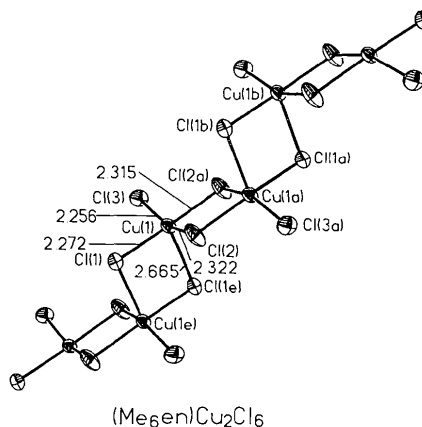


Fig. 1. Illustration of the stacking of the quasi-planar Cu₂Cl₆ anions in Me₆enCu₂Cl₆. Distances in Å.

described previously (Battaglia, Bonamartini-Corradi, Geiser, Willett, Motori, Sandrolini, Antolini, Manfredini, Menabue & Pellacani, 1988).

The structure data agree well with previous structural correlations: Cu...Cl distance vs bifold angle

* Tables of data collection parameters, anisotropic thermal parameters, H-atom parameters and structure factors, as well as a stereoview of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54082 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Willett, 1987); terminal Cu—Cl vs bridging Cu—Cl (Willett, 1988).

Acknowledgement is made to The Boeing Company and to NSF, through grant CHE-8408407, for the establishment of the X-ray diffraction facility. Research supported by NSF grant DMR-88-03382.

References

- BATTAGLIA, L. P., BONAMARTINI-CORRADI, A., GEISER, U., WILLET, R. D., MOTORI, A., SANDROLINI, G., ANTOLINI, L., MANFREDINI, T., MENABUE, L. & PELLACANI, G. C. (1988). *J. Chem. Soc. Dalton Trans.* pp. 265–271.
- BOND, M. R. & WILLETT, R. D. (1989). *Inorg. Chem.* **28**, 3267–3269.
- CAMPANA, C. F., SHEPARD, D. F. & LITCHMAN, W. M. (1981). *Inorg. Chem.* **20**, 4039–4044.
- GEISER, U., WILLETT, R. D., LINDBECK, M. & EMERSON, K. (1986). *J. Am. Chem. Soc.* **108**, 1173–1179.
- SHELDRIK, G. M. (1985). *SHELXTL Users Manual*. Revision 5.1. Nicolet XRD Corporation Madison, Wisconsin, USA.
- WEENK, J. & SPEK, A. L. (1976). *Cryst. Struct. Commun.* **5**, 805–809.
- WILLETT, R. D. (1987). *Trans. Met. Chem. London*, **12**, 410–413.
- WILLETT, R. D. (1988). *Acta Cryst.* **B44**, 503–508.
- WILLETT, R. D., BOND, M. R., HAJE, W. G., SOONIEUS, O. P. M. & MAASKANT, W. J. A. (1988). *Inorg. Chem.* **27**, 614–620.

Acta Cryst. (1991). **C47**, 1975–1976

Structure of Dichloro(η^5 -pentamethylcyclopentadienyl)(trimethylphosphine)iridium 0.25 Diethyl Ether Solvate

BY FREDERICK W. B. EINSTEIN, XIAOQIAN YAN AND DEREK SUTTON

Department of Chemistry, Simon Fraser University, Burnaby, British Columbia, Canada V5A 1S6

(Received 19 November 1990; accepted 11 March 1991)

Abstract. $[\text{Ir}(\text{C}_{10}\text{H}_{15})(\text{Cl})_2\{\text{P}(\text{CH}_3)_3\}] \cdot \frac{1}{4}(\text{C}_2\text{H}_5)_2\text{O}$, $M_r = 492.94$, tetragonal, $I4$, $a = 17.918$ (3), $c = 11.409$ (1) Å, $V = 3662.9$ Å³, $Z = 8$, $D_x = 1.78$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 76.4$ cm⁻¹, $F(000) = 1887.5$, $T = 296$ K, $R = 0.036$, $wR = 0.047$ for 959 observed reflections. The Ir atom is coordinated to an η^5 -pentamethylcyclopentadienyl group, a PMe_3 ligand and two Cl atoms in a three-legged piano-stool type of structure. Selected bond distances and angles are: Ir—C₅Me₅ centroid 1.85, Ir—P 2.28 (1), Ir—Cl(1) 2.37 (1), Ir—Cl(2) 2.39 (1) Å; Cl(1)—Ir—Cl(2) 93.0 (3), Cl(1)—Ir—P 87.6 (2), Cl(2)—Ir—P 86.9 (2), Cl(1)—Ir—C₅Me₅ centroid 124.0, Cl(2)—Ir—C₅Me₅ centroid 123.0, P—Ir—C₅Me₅ centroid 130.8°.

Experimental. Compound isolated from reaction of $[\text{Cp}^*\text{IrCl}_2]_2$ and PMe_3 in CHCl_3 ($\text{Cp}^* = \eta^5\text{-C}_5\text{Me}_5$); red crystals obtained from chloroform and diethyl ether. A cube-shaped crystal $0.28 \times 0.23 \times 0.13$ mm was mounted on a glass fibre. Intensity data were collected at 296 (1) K with an Enraf–Nonius CAD-4F diffractometer using graphite-monochromatized Mo $K\alpha$ radiation. Lattice parameters were determined from 25 reflections ($15 \leq \theta < 20^\circ$). 1206 independent reflections were measured ($1.6 \leq 2\theta \leq 50^\circ$; $h: 0\text{--}18$, $k: 0\text{--}18$, $l: 0\text{--}12$) by using ω - 2θ scans; 959 of the 1206 reflections were classed as

observed $I_o \geq 2\sigma(I_o)$; scan range $(0.85 + 0.35\tan\theta)^\circ$; scan speed $0.72\text{--}2.75^\circ \text{min}^{-1}$; intensities of two standards (701, $\bar{3}81$) were measured every hour of acquisition time and showed no long term change and had an r.m.s. deviation of 1.8%; data reduction included Lorentz and polarization corrections and an analytical absorption correction, ψ -scan checked, transmission 0.355–0.648 (Alcock, 1969). Structure solved by Patterson synthesis and refined by full-matrix least squares and Fourier methods with the z coordinate of Ir restrained to zero (z is the fourfold axis). The assumption that the space group is $I4$ was confirmed by the successful refinement. The positions of some methyl-group H atoms were located in electron density difference Fourier maps (with $\sin\theta/\lambda \leq 0.3$ Å⁻¹) and were used as input to calculate positions [$d(\text{C—H}) = 0.95$ Å] for all H atoms. Disorder of the solvent diethyl ether appeared to include some superposition of oxygen and carbon sites and gave peaks along the fourfold axis. It was modelled by three C atoms (see Table 1). Final refinement included anisotropic thermal parameters for Ir, Cl and P and isotropic thermal parameters for other non-H atoms. H atoms were included in fixed positions in structure-factor calculations. Refinement was considered complete when the shift/e.s.d. ratio was less than 0.002; 94 parameters varied. Final residual $R = 0.036$, $wR = 0.047$, goodness of fit 0.86; quantity