

Fig. 3. ORTEP diagram and atom-labeling scheme of Bi_2Ph_4 , tetragonal form, showing 50% probability ellipsoids.

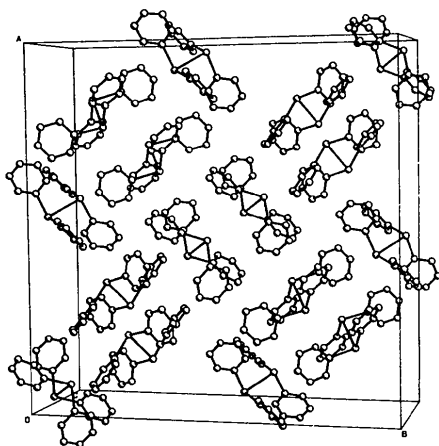


Fig. 4. Packing diagram of Bi_2Ph_4 , tetragonal form.

were included in calculated positions but not refined. The data were corrected for Lorentz-polarization effects and absorption (*DIFABS*, transmission range 0.901–1.238). Refinement of 115 variables (Molecular Structure Corporation, 1988) was carried out on *F* with final $R = 0.038$, $wR = 0.039$, $S = 1.21$ and a maximum Δ/σ of 0.0001. The final difference map showed peaks $\Delta\rho_{\min} = -0.84$, $\Delta\rho_{\max} = 0.93 \text{ e } \text{\AA}^{-3}$. Scattering factors were taken from *International Tables for X-ray Crystallography*. Atomic coordinates are given in Table 3 and selected bond metrics in Table 4. An ORTEP diagram is presented in Fig. 3 and a packing diagram in Fig. 4.

Related literature. Bond metrics are similar to those reported for Bi_2Ph_4 in a triclinic cell (Calderazzo, Morvillo, Pelizzi & Poli, 1983; Calderazzo, Poli & Pelizzi, 1984).

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References

- CALDERAZZO, F., MORVILLO, A., PELIZZI, G. & POLI, R. (1983). *J. Chem. Soc. Chem. Commun.* pp. 507–508.
 CALDERAZZO, F., POLI, R. & PELIZZI, G. (1984). *J. Chem. Soc. Dalton Trans.* pp. 2365–2369.
 CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, p. 71 (scattering factors) and p. 148 (anomalous dispersion). Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
 GILMORE, C. J. (1983). *MITHRIL*. A computer program for the automatic solution of crystal structures from X-ray data. Univ. of Glasgow, Scotland.
 JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Molecular Structure Corporation (1988). *TEXSAN. TEXRAY Structure Analysis Package*. Version 2.1. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 WALKER, N. & STUART, D. (1983). *Acta Cryst. A39*, 158–166.

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Structure of 2-Chloroimidazolium Aquatrichlorocuprate(II)

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Abstract. $\text{C}_3\text{H}_4\text{ClN}_2^+ \cdot [\text{CuCl}_3(\text{H}_2\text{O})]^-$, $M_r = 291.5$, monoclinic, $P2_1/c$, $a = 9.023$ (1), $b = 13.881$ (2), $c = 7.341$ (1) Å, $\beta = 97.8$ (2)°, $V = 910.9$ (5) Å³, $Z = 4$,

$D_x = 2.13 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.7107 \text{ \AA}$, $\mu = 33.72 \text{ cm}^{-1}$, $F(000) = 572$, room temperature, $R = 0.053$ for 1106 reflections with $F > 7\sigma(F)$. The struc-

ture consists of rows of $[\text{CuCl}_3(\text{H}_2\text{O})]^-$ anions and rows of $\text{C}_3\text{H}_4\text{ClN}_2^+$ cations parallel to the c axis. The nearly planar $[\text{CuCl}_3(\text{H}_2\text{O})]^-$ groups are linked by $\text{Cu}\cdots\text{Cl}$ bonds giving each Cu^{II} ion a 4 + 1 + 1 coordination.

Experimental. A 1:2 mixture of CuCl_2 and 2-chloroimidazole (Takeuchi, Kirk & Cohen, 1979) in concentrated hydrochloric acid was evaporated to dryness; partial dissolution of the residue in acetone followed by diffusion in dichloromethane produced yellow crystals of $\text{C}_3\text{H}_4\text{ClN}_2^+.[\text{CuCl}_3(\text{H}_2\text{O})]^-$ (I). Crystal dimensions approximately $0.1 \times 0.2 \times 0.8$ mm. Cell dimensions and intensity data were measured on a Philips PW 1100 diffractometer; θ - 2θ scan; lattice parameters from 25 reflections with $7 < \theta < 13^\circ$. 2210 unique reflections ($R_{\text{int}} = 0.046$), $5 < 2\theta < 56^\circ$ ($h - 11 \rightarrow 11$; $k 0 \rightarrow 18$; $l 0 \rightarrow 9$), 1106 had $F > 7\sigma(F)$; three standard reflections monitored every 180 reflections, 10% variation (no corrections applied); Lorentz and polarization corrections applied, no absorption correction. The Cu atom was first located from a Patterson map through *SHELXS86* (Sheldrick, 1986); then, a difference Fourier map revealed the other non-H atoms (*SHELX76*; Sheldrick, 1976); one water H atom located from a difference map, other H atoms placed at calculated positions; anisotropic refinement (on F) for all non-H atoms, H atoms not refined; the refinement converged to final $R = 0.053$, $wR = 0.059$, for 100 parameters, $w = 1/[\sigma^2(F) + 0.003969F^2]$; $S = 1.073$, Δ/σ values < 1 except for U_{11} of N(1) (1.58) and N(3) (1.20); $\Delta\rho_{\text{max}} = 0.891$, $\Delta\rho_{\text{min}} = -0.88 \text{ e } \text{\AA}^{-3}$; atomic scattering factors for C, H, N, O, Cl from *SHELX76* and for Cu from *International Tables for X-ray Crystallography* (1974, Vol. IV).

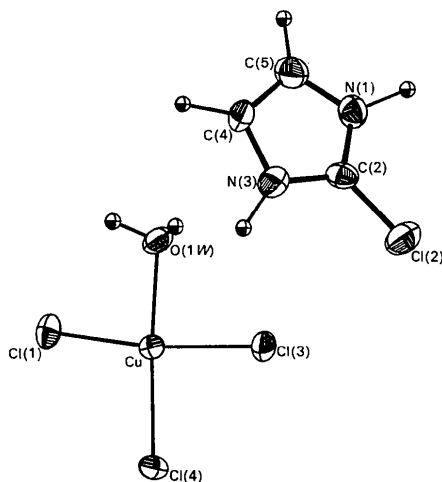


Fig. 1. The asymmetric unit.

Table 1. Fractional coordinates of non-H atoms with equivalent isotropic thermal parameters (\AA^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cu	0.4149 (1)	0.23894 (8)	0.1850 (2)	0.0280 (4)
Cl(1)	0.6021 (2)	0.2589 (2)	0.4193 (3)	0.0341 (7)
Cl(2)	-0.1374 (3)	0.3899 (2)	0.1520 (5)	0.055 (1)
Cl(3)	0.2055 (3)	0.2415 (2)	-0.0262 (3)	0.0335 (7)
Cl(4)	0.4024 (2)	0.0752 (2)	0.2253 (4)	0.0344 (7)
O(1W)	0.4095 (7)	0.3852 (5)	0.1554 (9)	0.034 (2)
N(1)	-0.0488 (9)	0.5618 (6)	0.285 (1)	0.037 (3)
N(3)	0.1333 (9)	0.4658 (7)	0.257 (1)	0.045 (3)
C(2)	-0.014 (1)	0.4755 (7)	0.232 (1)	0.030 (3)
C(4)	0.193 (1)	0.5531 (8)	0.326 (2)	0.043 (4)
C(5)	0.079 (1)	0.6118 (7)	0.344 (2)	0.041 (4)

Table 2. Bond lengths (\AA), angles ($^\circ$) and interatomic contacts (\AA)

Cu—Cl(1)	2.258 (5)	Cu—Cl(3)	2.276 (5)
Cu—Cl(4)	2.296 (3)	Cu—O(1W)	2.041 (6)
Cu \cdots Cl(1 ^b)	2.748 (5)	Cu \cdots Cl(3 ^b)	3.037 (5)
C(2)—Cl(2)	1.68 (1)	N(1)—C(2)	1.31 (1)
N(1)—C(5)	1.36 (1)	N(3)—C(2)	1.32 (1)
N(3)—C(4)	1.39 (1)	C(4)—C(5)	1.33 (2)
Cl(4)—Cu—O(1W)	175.4 (2)	Cl(3)—Cu—O(1W)	84.6 (2)
Cl(3)—Cu—Cl(4)	93.0 (1)	Cl(1)—Cu—O(1W)	87.9 (2)
Cl(1)—Cu—Cl(4)	93.9 (1)	Cl(1)—Cu—Cl(3)	169.4 (1)
C(2)—N(1)—C(5)	109.4 (9)	C(2)—N(3)—C(4)	107.0 (9)
N(1)—C(2)—N(3)	109.2 (9)	Cl(2)—C(2)—N(3)	125.6 (8)
Cl(2)—C(2)—N(1)	125.1 (8)	N(3)—C(4)—C(5)	108 (1)
N(1)—C(5)—C(4)	106.7 (9)		
O(1W) \cdots Cl(4 ^b)	3.197 (7)	N(1) \cdots Cl(4 ^b)	3.187 (9)
O(1W) \cdots Cl(4 ⁱⁱⁱ)	3.194 (7)	N(1) \cdots Cl(3 ^{iv})	3.468 (10)
N(3) \cdots O(1W)	2.920 (11)	N(3) \cdots Cl(3 ^b)	3.309 (10)

Symmetry code: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (iv) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$.

The asymmetric unit is shown in Fig. 1 with the atomic numbering. Positional parameters for non-H atoms are given in Table 1, bond lengths and angles in Table 2.*

The structure consists of rows of $[\text{CuCl}_3(\text{H}_2\text{O})]^-$ anions and rows of $\text{C}_3\text{H}_4\text{ClN}_2^+$ cations parallel to the c axis. The configuration about the Cu atom is square-planar with slight pyramidal distortion. The $[\text{CuCl}_3(\text{H}_2\text{O})]^-$ groups are linked by semicoordinate $\text{Cu}\cdots\text{Cl}$ bonds giving each Cu^{II} ion a 4 + 1 + 1 coordination. The repeat distance between Cu atoms is 3.683 (2) \AA .

Related literature. The 2-chloroimidazole complex $[\text{CuCl}_2(\text{C}_3\text{H}_3\text{ClN}_2)_2]$ (II) has been reported previously (Valle, Sánchez González & Ettore,

* Lists of structure factors, anisotropic thermal parameters, least-squares planes, possible interatomic contacts, possible hydrogen-bond geometries and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54721 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0278]

1991). The compounds $[\text{Cu}_2\{3,6\text{-bis}(3,5\text{-dimethyl-1-pyrazolyl})\text{pyridazine}\}(\text{OH})\text{Cl}_2][\text{CuCl}_3(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}$ (III) (Thompson, Woon, Murphy, Gabe, Lee & Le Page, 1985), $[\text{N},\text{N}'\text{-dimethylpiperazinium}][\text{CuCl}_3(\text{H}_2\text{O})]$ (IV) and $[2\text{-aminopyrimidinium}][\text{CuCl}_3(\text{H}_2\text{O})]$ (V) (Manfredini *et al.*, 1990) have been described. The configuration of the anion in (III) is intermediate between tetrahedral and square-planar. Compounds (IV) and (V) contain planar $[\text{CuCl}_3(\text{H}_2\text{O})]^-$ groups linked by $\text{Cu}\cdots\text{Cl}$ bonds. The contact between $[\text{CuCl}_3(\text{H}_2\text{O})]^-$ groups is closer for (I) than for (IV) [$\text{Cu}\cdots\text{Cl} = 3.106$ (2), 3.110 (2) Å] or (V) [$\text{Cu}\cdots\text{Cl} = 2.996$ (1), 3.169 (1) Å].

References

- MANFREDINI, T., PELLACANI, G. C., BONAMARTINI-CORRADI, A., BATTAGLIA, L. P., GUARINI, G. G. T., GELSOMINI GIUSTI, J., PON, G., WILLETT, R. D. & WEST, D. X. (1990). *Inorg. Chem.* **29**, 2221–2228.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
- TAKEUCHI, Y., KIRK, K. L. & COHEN, L. A. (1979). *J. Org. Chem.* **44**, 4240–4242.
- THOMPSON, L. K., WOON, T. C., MURPHY, D. B., GABE, E. J., LEE, F. L. & LE PAGE, Y. (1985). *Inorg. Chem.* **24**, 4719–4725.
- VALLE, G., SÁNCHEZ GONZÁLEZ, A. & ETTORRE, R. (1991). *Acta Cryst. C* **47**, 1392–1394.

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Structure of Tetracarbonyl[(2,3,7,8,12,13,17,18-octaethylporphinato-germanio(IV))iron]

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Abstract. $[\text{FeGe}(\text{C}_{36}\text{H}_{44}\text{N}_4)(\text{CO})_4]$, $[(\text{oep})\text{GeFe}(\text{CO})_4]$, $M_r = 773.25$, triclinic, $P\bar{1}$, $a = 12.123$ (2), $b = 13.851$ (3), $c = 15.028$ (3) Å, $\alpha = 59.99$ (2), $\beta = 61.53$ (2), $\gamma = 69.15$ (2)°, $V = 1897.9$ (7) Å³, $Z = 2$, $D_x = 1.357$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 12.05$ cm⁻¹, $F(000) = 804$, $R(F) = 0.0410$ for 5098 reflections. $[(\text{oep})\text{GeFe}(\text{CO})_4]$ has two coordinated metal units, which are linked by a double bond; the Ge—Fe bond distance is 2.370 (2) Å. The average Ge—N distance is 2.082 (6) \pm 0.01 Å and the Ge atom lies 0.684 (1) Å above the four-N-atom plane towards the Fe atom, the Fe atom is in an axial position with Ge—Fe—C(53) = 178.9 (1)°, the average Fe—CO distance is 1.77 (1) \pm 0.005 Å.

Experimental. Crystals were prepared according to Barbe, Guillard, Lecomte & Gerardin (1984). A black crystal, $0.25 \times 0.18 \times 0.12$ mm, of $[(\text{oep})\text{GeFe}(\text{CO})_4]$ recrystallized from toluene/heptane was mounted on an Enraf–Nonius CAD-4F diffractometer. Unit-cell

dimensions at room temperature were obtained from accurate angle values of 25 reflections with $10 < \theta < 24^\circ$ using monochromated Mo $K\alpha$ radiation. 9054 reflections were measured up to $(\sin\theta)/\lambda = 0.66 \text{ \AA}^{-1}$ at room temperature ($-14 < h < 14$, $-16 < k < 16$, $0 < l < 17$); standard reflections 200, $\bar{2}\bar{1}\bar{5}$, $3\bar{1}4$ monitored every 3 h; ω - 2θ scan; scan width $\Delta\omega = 0.9^\circ + 0.35^\circ \tan\theta$; scan speed 0.6 to $1.55^\circ \text{ min}^{-1}$. No decay was observed and no absorption correction was applied. 5098 reflections [$I \geq 3\sigma(I)$], corrected for Lorentz and polarization effects, structure solved by interpretation of the Patterson map; all non-H atoms were refined anisotropically (*SHELX76*; Sheldrick, 1976); H atoms were found in difference Fourier maps and refined isotropically. At convergence, $\Delta/\sigma_{\text{max}} = -0.31$ for U_{11} of C(53), a residual Fourier map gave a maximum peak of 0.52 e \AA^{-3} . Weighting scheme $w^{-1} = \sigma^2(F) + 0.0003F^2$. Atomic scattering factors were taken from *SHELX76* and from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final residuals are $R(F) = 0.0410$, $wR(F) = 0.0372$, $\text{GOF} = 1.421$. Fractional coordinates and

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