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## Key indicators

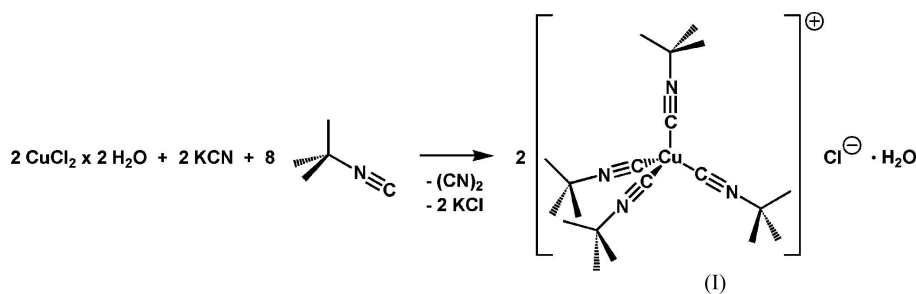
Single-crystal X-ray study  
 $T = 183\text{ K}$   
Mean  $\sigma(\text{N}-\text{C}) = 0.006\text{ \AA}$   
Disorder in main residue  
 $R$  factor = 0.070  
 $wR$  factor = 0.231  
Data-to-parameter ratio = 26.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Tetrakis(*tert*-butyl isocyanide)copper(I) chloride  
monohydrate

The title compound,  $[\text{Cu}(\text{C}_5\text{H}_9\text{N})_4]\text{Cl}\cdot\text{H}_2\text{O}$ , was obtained from the reaction of copper(II) chloride hydrate with KCN in the presence of two equivalents of *tert*-butylisocyanide. The reaction proceeds *via* the reduction of copper and the formation of  $(\text{CN})_2$ . The compound shows a tetrahedrally surrounded  $\text{Cu}^{\text{I}}$  centre, with the  $\text{Cu}^{\text{I}}$  ion and the water O atom being situated on crystallographic twofold axes. The crystal structure contains infinite chains of alternating solvent molecules and  $\text{Cl}^-$  anions, with additional weak  $\text{C}-\text{H}\cdots\text{Cl}$  or  $\text{C}-\text{H}\cdots\text{O}$  interactions with the  $[\text{Cu}(\text{CN}^t\text{Bu})_4]$  cations.

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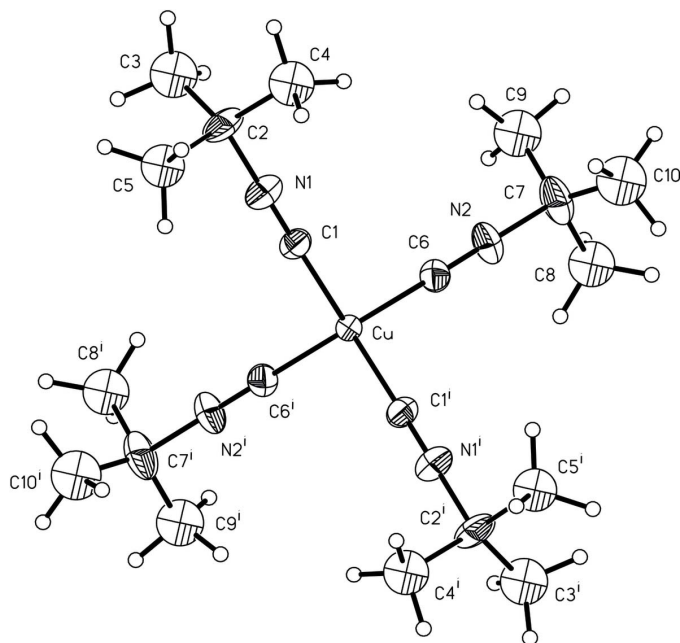
## Comment

In connection with our studies of cyanide-bridged coordination polymers, we recently reported the facile synthesis of *cis*- and *trans*- $[\text{Ru}(\text{CN}^t\text{Bu})_4(\text{CN})_2]$  from  $\text{Ru}_3(\text{CO})_{12}$  and *tert*-butylisocyanide (Imhof & Dönnecke, 2003). Unfortunately, this reaction principle is limited to a small number of transition metal carbonyls. In order to enlarge our repertoire of cyano complexes which may be utilized in the synthesis of cyanide-bridged coordination polymers with a well defined steric arrangement of the metal centres with respect to each other, we are currently looking for a general methodology to obtain *trans*- $[\text{M}(\text{CNR})_4(\text{CN})_2]$  complexes.



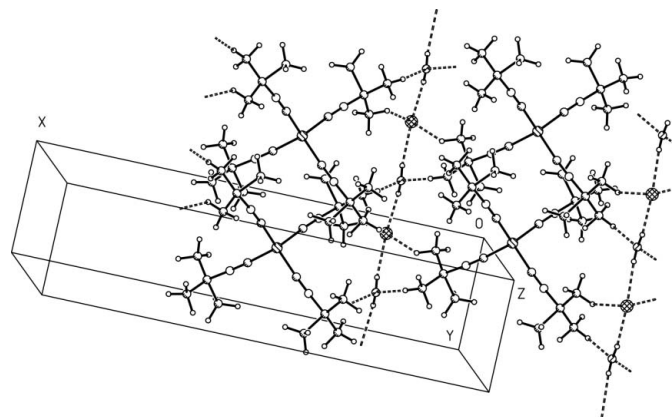
The reaction of  $\text{CuCl}_2\cdot x\text{H}_2\text{O}$  with *tert*-butylisocyanide in the presence of stoichiometric amounts of KCN leads to the almost quantitative formation of the title compound, (I), and the concomitant production of  $(\text{CN})_2$ . The reduction of  $\text{Cu}^{\text{II}}$  in solutions containing cyanide ions is a well known phenomenon, which we tried unsuccessfully to avoid by adding the isocyanide before the corresponding amount of KCN.

The molecular structure of the  $[\text{Cu}(\text{CN}^t\text{Bu})_4]^+$  cation of (I) is shown in Fig. 1, and the most important bond lengths and angles are summarized in Table 1. The  $\text{Cu}^{\text{I}}$  atom and the water O atom are situated on crystallographic twofold axes, resulting in a virtually ideal tetrahedral coordination around the  $\text{Cu}^{\text{I}}$  ion. Bond lengths and angles are typical for tetrahedrally coordinated transition metal isocyanide groups. The isocya-


**Figure 1**

The molecular structure of the  $[\text{Cu}(\text{CN}^t\text{Bu})_4]^+$  cation of (I). Displacement ellipsoids are drawn at the 40% probability level. Symmetry code as in Table 1.

nide moieties show only a slight deviation from linearity. A search of the Cambridge Structural Database (CSD, Version 5.26, November 2004; Allen, 2002) found 36 structures in which a transition metal is surrounded by four isocyanide ligands. Most of these structurally characterized compounds are mononuclear complexes similar to (I) (Gordon *et al.*, 1978; Spek, 1982; Matsubayashi *et al.*, 1982; Plummer *et al.*, 1984; Yamamoto & Yamazaki, 1984; Ashworth *et al.*, 1984; Krause, 1979; Goldberg *et al.*, 1976; Kanters *et al.*, 1989; Bois *et al.*, 1998; Connelly *et al.*, 1992; Ojima *et al.*, 1991; Leach *et al.*, 1994; Deicas *et al.*, 1997; Crossley & Orpen, 1995), but there are also reports of transition metal complexes of diisocyanides yielding either discrete dinuclear compounds (Mann 1981; Exstrom *et al.*, 1996) or coordination polymers (Fortin *et al.*, 1998). Three complexes are also copper(I) compounds, with methylisocyanide (Spek, 1982), 2-methoxyisobutylisocyanide or 2-methoxycarbonyl-2-methylethylisocyanide (Deicas *et al.*, 1997) coordinating the central metal ion. The latter compounds also show a nearly undistorted tetrahedral ligand environment of the Cu atoms. The methylisocyanide derivative (Spek, 1982) with three independent molecules in the asymmetric unit of the unit cell shows Cu—C bond lengths from 1.941 to 2.014 Å, whereas the compounds published by Deicas *et al.* (1997) exhibit Cu—C bond lengths from 1.940 to 1.972 Å. Thus, the Cu—C bond lengths observed in the molecular structure of the title compound [1.952 (4) and 1.950 (4) Å] are in the same range as those reported previously. The same is true for the Cu—C—N bond angles, although values down to 169° have been observed earlier (Deicas *et al.*, 1997), most probably due to steric interactions of the 2-methoxycarbonyl-2-methylethylisocyanide ligands,


**Figure 2**

A packing diagram for (I). Dashed lines indicate hydrogen bonds.

which are even bulkier than the *tert*-butyl groups of the title compound.

The crystal structure of the title compound is depicted in Fig. 2 and the shortest intermolecular contacts are presented in Table 2. Along the *b* axis of the unit cell, infinite chains consisting of  $\text{Cl}^-$  anions and solvent molecules are observed. These chains are held together by quite strong hydrogen bonds between the water H atoms and the  $\text{Cl}^-$  anions. In addition, the  $\text{Cl}^-$ -water chains are connected by weak C—H...O and C—H...Cl contacts (Desiraju & Steiner, 1999) from methyl groups of the isocyanide ligands, building up an infinite three-dimensional network. In Fig. 2, only one plane of the network is shown for clarity.

## Experimental

$\text{CuCl}_2 \cdot x\text{H}_2\text{O}$  (100 mg, 0.587 mmol) was dissolved in anhydrous ethanol (10 ml), resulting in a green solution. *tert*-Butylisocyanide (0.27 ml, 2.348 mmol) was added to the solution using a syringe. After adding KCN (76 mg, 1.174 mmol), the colour of the solution slowly changed from green to colourless and a white precipitate of KCl was formed. The suspension was stirred for 4 h at room temperature. Filtration of the precipitated KCl and evaporation of the solvent *in vacuo* yielded a white solid. Recrystallization of the crude reaction product from acetone produced colourless prisms of (I) which were suitable for this X-ray diffraction study. Spectroscopic analysis: IR (KBr,  $\text{cm}^{-1}$ ):  $\nu(\text{CH})$  2984 (*s*), 2936 (*m*),  $\nu(\text{CN})$  2177 (*vs*),  $\delta(\text{CH})$  1467 (*m*),  $\delta(\text{CCH}_3)$  1395 (*w*), 1370 (*s*), 1240 (*m*), 1193 (*s*); MS (FAB in nitrobenzylalcohol, *m/z*, %): 413 {32,  $[\text{Cu}(\text{CN}^t\text{Bu})_4\text{H}_2\text{O}]$ }, 395 {10,  $[\text{Cu}(\text{CN}^t\text{Bu})_4]$ }, 330 {9,  $[\text{Cu}(\text{CN}^t\text{Bu})_3\text{H}_2\text{O}]$ }, 312 {62,  $[\text{Cu}(\text{CN}^t\text{Bu})_3]$ }, 229 {100,  $[\text{Cu}(\text{CN}^t\text{Bu})_2]$ }.

### Crystal data

$[\text{Cu}(\text{C}_5\text{H}_9\text{N})_4]\text{Cl} \cdot \text{H}_2\text{O}$   
 $M_r = 449.53$   
 Orthorhombic, *Pcca*  
 $a = 22.3933$  (9) Å  
 $b = 5.6174$  (2) Å  
 $c = 22.3033$  (8) Å  
 $V = 2805.58$  (18) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.064$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 1947 reflections  
 $\theta = 1.8$ –27.5°  
 $\mu = 0.89$  mm<sup>-1</sup>  
 $T = 183$  (2) K  
 Prism, colourless  
 0.05 × 0.05 × 0.03 mm

## Data collection

Nonius KappaCCD area-detector diffractometer	1947 reflections with $I > 2\sigma(I)$
$\varphi$ and $\omega$ scans	$R_{\text{int}} = 0.108$
Absorption correction: none	$\theta_{\text{max}} = 27.5^\circ$
25405 measured reflections	$h = -29 \rightarrow 23$
3233 independent reflections	$k = -7 \rightarrow 7$
	$l = -28 \rightarrow 28$

## Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.1248P)^2 + 3.6821P]$
$R[F^2 > 2\sigma(F^2)] = 0.070$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.231$	$(\Delta/\sigma)_{\text{max}} = 0.004$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.98 \text{ e } \text{\AA}^{-3}$
3233 reflections	$\Delta\rho_{\text{min}} = -0.59 \text{ e } \text{\AA}^{-3}$
124 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Cu—C1	1.950 (4)	N1—C2	1.468 (5)
Cu—C6	1.952 (4)	N2—C6	1.152 (5)
N1—C1	1.152 (5)	N2—C7	1.472 (6)
C1—Cu—C1 <sup>i</sup>	110.7 (2)	C6 <sup>i</sup> —Cu—C6	110.9 (2)
C1—Cu—C6 <sup>i</sup>	108.71 (18)	C1—N1—C2	178.1 (4)
C1 <sup>i</sup> —Cu—C6 <sup>i</sup>	108.90 (19)	C6—N2—C7	177.4 (5)
C1—Cu—C6	108.90 (19)	N1—C1—Cu	177.5 (4)
C1 <sup>i</sup> —Cu—C6	108.71 (18)	N2—C6—Cu	176.5 (4)

Symmetry codes: (i)  $-x + 1, y, -z + \frac{3}{2}$ .

Table 2

Geometry of shortest intermolecular contacts ( $\text{\AA}$ ,  $^\circ$ ).

$D-H \cdots A$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C9A—H9AB $\cdots$ O1W <sup>ii</sup>	2.55	3.467	156
C10—H10C $\cdots$ Cl1 <sup>iii</sup>	2.74	3.503	135
O1W—H1OW $\cdots$ Cl1 <sup>iv</sup>	2.38	3.263	166

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

The water H atom was refined freely. All other H atoms were placed in idealized positions, with C—H = 0.98  $\text{\AA}$ , and were refined using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ . The C atoms of the methyl groups (C3—C5 and C8—C10) are disordered and were therefore refined isotropically.

Data collection: COLLECT (Nonius, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997); data reduction: DENZO; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP (Siemens, 1990); software used to prepare material for publication: SHELXL97.

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