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## $\mu$ -Terephthalato-bis[bis(1,10-phenanthroline)copper(II)] diperchlorate

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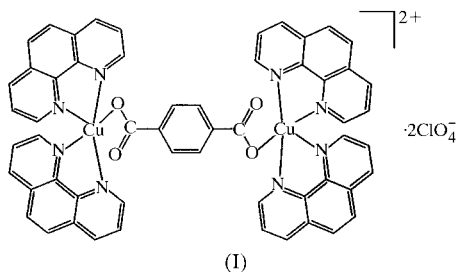
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The title compound,  $[\text{Cu}_2(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{12}\text{H}_8\text{N}_2)_4](\text{ClO}_4)_2$ , was prepared from the hydrothermal reaction of  $\text{CuCl}_2$ , 1,4-dicyanobenzene, 1,10-phenanthroline and water at 443 K. The compound is a dimer in which the cation lies about an inversion center. The terephthalate moiety acts as a bridging ligand and the phenanthrolines as terminal ligands. The unique Cu atom is coordinated by two O and four N atoms in a distorted octahedral geometry, with Cu—O distances of 1.955 (2) and 2.815 (2) Å, and Cu—N distances of 2.008 (2) to 2.216 (2) Å.

### Comment

Owing to their important role in the development of magnetochemistry, dinuclear copper(II) complexes have attracted much interests in recent years (Kahn, 1993). With the aim of



investigating the dependence of magnetic exchange on metal-metal distances, a large number of dinuclear copper(II) complexes have been prepared using different extended bridging ligands (Hatfield, 1985; Coffman & Buettner, 1979). For this family, many reports have focused on extended bridging ligands containing O- or N-donor atoms, such as oxalate (Julve *et al.*, 1984) and 4,4'-bipyridine (Julve *et al.*, 1984), especially terephthalate (Bakalbassis *et al.*, 1991; Burger *et al.*, 1995; Cano *et al.*, 1997; Chaudhuri *et al.*, 1988; Xanthopoulos *et al.*, 1993), and most of the complexes were synthesized by conventional solution reactions. We report

here the crystal structure of a dinuclear copper(II) complex, (I), containing a terephthalate bridging ligand prepared *via* hydrothermal reaction.

The title compound,  $[\{\text{Cu}(\text{C}_{12}\text{H}_8\text{N}_2)_2\}_2(\text{C}_8\text{H}_4\text{O}_4)](\text{ClO}_4)_2$ ,  $[\text{C}_8\text{H}_4\text{O}_4 = \text{terephthalate (ta), C}_{12}\text{H}_8\text{N}_2 = 1,10\text{-phenanthroline (phen)}]$ , is a dimer in which the ta ligand bridges two  $\text{Cu}^{\text{II}}$  atoms *via* O atoms from the two carboxylate groups, and each  $\text{Cu}^{\text{II}}$  is further chelated by two terminal phenanthroline ligands with chelating angles in the range 79.25 (9)–81.26 (9)°. Thus, the unique  $\text{Cu}^{\text{II}}$  atom is in a distorted octahedral geometry in which N2, N3, N4 and carboxyl O5 form an approximate plane, with a Cu—O1 distance of 1.955 (2) Å and Cu—N distances of 2.008 (2), 2.022 (2) and 2.056 Å. Atoms N1 and O6 form the apices with geometry Cu—O6 2.815 (2), Cu—N1 2.216 (2) Å and O6—Cu—N1 143.73 (8)°.

### Experimental

A mixture of  $\text{Cu}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$  (64 mg, 0.5 mmol), 1,4-dicyanobenzene (185 mg, 0.5 mmol), 1,10-phenanthroline (200 mg, 1 mmol) and water (10 ml) was sealed in a 25 ml teflon-lined stainless steel reactor and heated to 443 K for 72 h. Crystals of  $[\text{Cu}_2(\text{ta})(\text{phen})_4](\text{ClO}_4)_2$  suitable for X-ray analysis were obtained. In the reaction, hydrolysis of 1,4-dicyanobenzene resulted in the formation of terephthalate.

#### Crystal data

$[\text{Cu}_2(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{12}\text{H}_8\text{N}_2)_4](\text{ClO}_4)_2$   
 $M_r = 1210.91$   
 Monoclinic,  $C2/c$   
 $a = 21.0780$  (4) Å  
 $b = 15.0708$  (4) Å  
 $c = 15.9811$  (3) Å  
 $\beta = 102.8230$  (10)°  
 $V = 4949.98$  (19) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.625$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 6459 reflections  
 $\theta = 1.68\text{--}25.01^\circ$   
 $\mu = 1.045$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Rectangle, blue  
 $0.30 \times 0.25 \times 0.20$  mm

#### Data collection

Siemens Smart CCD diffractometer  
 $\omega$  scans  
 Absorption correction: empirical from equivalent reflections (*XEMP* in *SHELXTL*; Sheldrick, 1994)  
 $T_{\text{min}} = 0.745$ ,  $T_{\text{max}} = 0.818$   
 9568 measured reflections

4337 independent reflections  
 3590 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$   
 $\theta_{\text{max}} = 25.01^\circ$   
 $h = -25 \rightarrow 22$   
 $k = -11 \rightarrow 17$   
 $l = -19 \rightarrow 11$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.106$   
 $S = 1.042$   
 4337 reflections  
 361 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 6.9792P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.54$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.43$  e Å<sup>-3</sup>

The title compound crystallized in the monoclinic system, space group  $C2/c$  or  $Cc$  from the systematic absences.  $C2/c$  was assumed and confirmed by the analysis.

Data collection: *SMART* (Siemens, 1994); cell refinement: *SMART*; data reduction: *SMART*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

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## References

- Bakalbassis, E. G., Tsipis, C., Bozopoulos, A., Dreisig, W., Hartl, H. & Mrozinski, J. (1991). *Inorg. Chim. Acta*, **186**, 113–118.
- Burger, K. S., Chaudhuri, P., Wieghardt, K. & Nuber, B. (1995). *Chem. Eur. J.* **1**, 583–589.
- Cano, J., Munno, G. D., Sanz, J. L., Ruiz, R., Faus, J., Lloret, F., Julve, M. & Caneschi, A. (1997). *J. Chem. Soc. Dalton Trans.* pp. 1915–1923.
- Chaudhuri, P., Oder, K., Wieghardt, K., Gehring, S., Haase, W., Nuber, B. & Weiss, J. (1988). *J. Am. Chem. Soc.* **110**, 3657–3658.
- Coffman, R. E. & Buettner, G. R. (1979). *J. Phys. Chem.* **18**, 2387–2392.
- Hatfield, W. E. (1985). *Magneto-Structural Correlations in Exchange Coupled Systems*, edited by R. D. Willet, D. Gatteschi & O. Kahn, p. 555. Dordrecht: Reidel.
- Julve, M., Verdaguier, M., Gleizes, A., Philoche-Levisalles, M. & Kahn, O. (1984). *Inorg. Chem.* **23**, 3808–3818.
- Kahn, O. (1993). In *Molecular Magnetism*. Weinheim: VCH.
- Sheldrick, G. M. (1997). *SHELXTL*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1994). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Xanthopoulos, C. E., Sigalas, M. P., Katsoulos, G. A., Tsipis, C. A., Terzis, A., Mentzafos, M. & Hountas, A. (1993). *Inorg. Chem.* **32**, 5433–5436.