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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1045). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *catena*-Poly[aqua(benzimidazole-*N*<sup>3</sup>)-copper(II)- $\mu$ -malonato-*O,O''*:*O'*]

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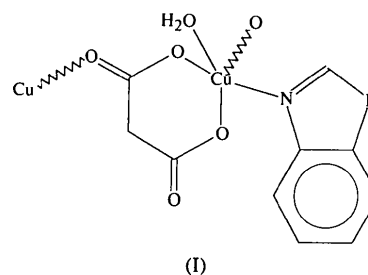
## Abstract

The title compound forms a polymeric chain,  $[\text{Cu}(\text{C}_3\text{H}_2\text{O}_4)(\text{C}_7\text{H}_6\text{N}_2)(\text{H}_2\text{O})]_\infty$ . A malonate ion adopts a bridging position between two Cu atoms, coordinating *via* three of its O atoms. Two form a six-membered chelate ring, the third coordinates to the neighbouring Cu atom.

The square-pyramidal  $\text{Cu}^{\text{II}}$  coordination comprises one N and three O atoms as the base, and an  $\text{H}_2\text{O}$  molecule in the apical position. The equalization of C—O bonds of both carboxylate groups and a differentiation of Cu—O bond lengths is observed.

## Comment

This work forms part of a wider study of the preparation, properties and structure of  $\text{Cu}^{\text{II}}$  complexes with dicarboxylic acids and benzimidazole (Tosik & Bukowska-Strzyżewska, 1992; Tosik, Sieroń & Bukowska-Strzyżewska, 1995). The structure of the title complex, (I), is polymeric with  $[\text{Cu}(\text{C}_3\text{H}_2\text{O}_4)(\text{C}_7\text{H}_6\text{N}_2)\text{H}_2\text{O}]_\infty$  chains along the *x* axis. Fig. 1 shows the independent fragment of the chain.



The malonate ion adopts a bridging position between two Cu atoms, forming with one, a six-membered chelate ring through two short Cu—O bonds [Cu—O2 = 1.954 (3) and Cu—O3 = 1.970 (3) Å], and with a second Cu atom, a short Cu—O1 bond of 1.990 (3) Å.

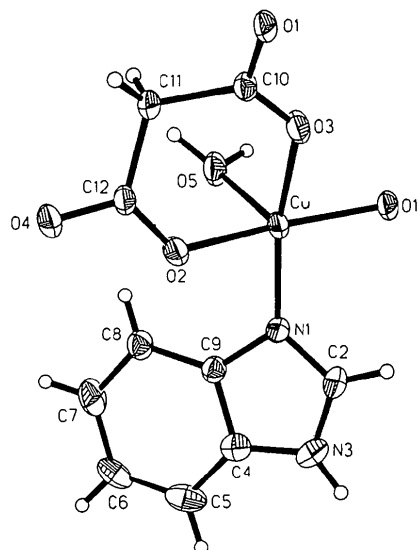


Fig. 1. An independent fragment of the  $[\text{Cu}(\text{C}_3\text{H}_2\text{O}_4)(\text{C}_7\text{H}_6\text{N}_2)\text{H}_2\text{O}]_\infty$  chain. Displacement ellipsoids are plotted at the 30% probability level.

The Cu—H<sub>2</sub>O apical bond of 2.253 (3) Å completes the elongated square-pyramidal (SQP) coordination. The observed SQP coordination is only slightly deformed in the direction of trigonal-bipyramidal (TBP) coordination, with the degree of trigonality  $\alpha = 0.08$  [ $\alpha$ , defined by Addison, Nagaswara Rao, Reedijk, van Rijn & Verschoor (1989), is 0 for SQP and 1 for TBP coordination]. The Cu atom is displaced more than usual from the average plane of the four base ligating atoms in the direction of the apical ligand — by 0.248 (2) Å. The observed Cu—O bonds in the chelate ring are shorter than the Cu—O bond of the bridging carboxylic group. All short Cu—O and Cu—N bonds are distinctly shorter than those observed in *catena*-poly[aquabis(benzimidazole-*N*<sup>3</sup>)copper(II)- $\mu$ -adipato-*O, O': O'', O'''*] (Tosik, Sieroń & Bukowska-Strzyżewska, 1995), and only Cu—N and Cu—O1 are longer than the average literature data for five-coordinate Cu<sup>II</sup> complexes [according to Orpen, Brammer, Allen, Kennard, Watson & Taylor (1989) for these kinds of ligands, average short Cu—N = 1.966, average short Cu—O = 1.962 and average long Cu—H<sub>2</sub>O = 2.331 Å].

The conformation of the chelated malonate ring is similar to an irregular boat conformation with all asymmetry parameters greater than 10° and the typical puckering of the rings being  $\varphi_{av} = 25.4^\circ$ . For five known Cu<sup>II</sup>-chelated malonate rings, the observed puckering ranges from 6.5 to 41.5°, with an average value of 25.9° (Chattopadhyay *et al.*, 1993; Pajunen & Nasakkala, 1977; Whei-Lu Kwik, Kok-Peng Ang, Sze-Oon Chan, Chebolu & Koch, 1986; Kansikas & Hamalainen, 1977). The large puckering differences indicate the conformational flexibility of this ring. The polymeric chains are connected by three different interchain hydrogen bonds: N3—H $\cdots$ O4, O5(H<sub>2</sub>O)—H $\cdots$ O4 and O5(H<sub>2</sub>O)—H $\cdots$ O3 (see Table 3).

## Experimental

CuCl<sub>2</sub>·2H<sub>2</sub>O (1 mmol, 0.17 g) and malonic acid (1 mmol, 0.104 g) dissolved in water (50 ml) were mixed with benzimidazole (2.5 mmol, 0.295 g) dissolved in water (50 ml). The resulting blue solution was heated to boiling, filtered and allowed to evaporate slowly. After several days blue needle-shaped crystals were obtained. Elemental analysis for C<sub>10</sub>H<sub>10</sub>N<sub>2</sub>O<sub>5</sub>Cu: calculated C 39.77, N 9.28; H 3.31%; found C 39.72, N 9.27, H 3.40%. The density  $D_m$  was measured by flotation in a mixture of iodomethane and benzene.

### Crystal data

[Cu(C<sub>3</sub>H<sub>2</sub>O<sub>4</sub>)(C<sub>7</sub>H<sub>6</sub>N<sub>2</sub>)-  
(H<sub>2</sub>O)]  
 $M_r = 301.74$   
Orthorhombic  
 $P2_12_12_1$   
 $a = 6.724$  (1) Å  
 $b = 8.721$  (1) Å  
 $c = 18.692$  (3) Å

Cu  $K\alpha$  radiation  
 $\lambda = 1.54184$  Å  
Cell parameters from 25  
reflections  
 $\theta = 15.0$ – $26.0^\circ$   
 $\mu = 3.007$  mm<sup>-1</sup>  
 $T = 293$  K  
Needle

$V = 1096.2$  (3) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.828$  Mg m<sup>-3</sup>  
 $D_m = 1.820$  Mg m<sup>-3</sup>

$0.20 \times 0.175 \times 0.15$  mm  
Blue

### Data collection

Enraf–Nonius CAD-4  
diffractometer  
 $\omega/2\theta$  scans  
Absorption correction:  
none  
1352 measured reflections  
1331 independent reflections  
1296 observed reflections  
[ $F > 4\sigma(F)$ ]

$\theta_{max} = 74.89^\circ$   
 $h = 0 \rightarrow 8$   
 $k = 0 \rightarrow 10$   
 $l = 0 \rightarrow 23$   
3 standard reflections  
frequency: 60 min  
intensity decay: 1.6%

### Refinement

Refinement on  $F^2$   
 $R = 0.0462$   
 $wR = 0.0470$   
 $S = 1.070$   
1296 reflections  
165 parameters  
 $w = 1/[\sigma^2(F_o^2) + (0.1077P)^2 + 0.49P]$   
where  $P = [\max(F_o^2, 0) + 2F_c^2]/3$

$(\Delta/\sigma)_{max} = 0.02$   
 $\Delta\rho_{max} = 0.45$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -1.11$  e Å<sup>-3</sup>  
Extinction correction: none  
Atomic scattering factors  
from SHELXL93  
(Sheldrick, 1993)  
Absolute configuration:  $\chi = 0.03$  (6) (Flack, 1983)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$
Cu	0.0751 (1)	0.3680 (1)	0.9141 (1)	0.024 (1)
O(1)	-0.3195 (5)	0.3449 (4)	1.0800 (2)	0.029 (1)
O(2)	-0.0833 (5)	0.5547 (4)	0.9046 (2)	0.032 (1)
O(3)	-0.0871 (6)	0.3092 (4)	0.9975 (2)	0.033 (1)
O(4)	-0.2227 (7)	0.7644 (4)	0.9445 (2)	0.040 (1)
O(5)	0.3333 (6)	0.4894 (4)	0.9660 (2)	0.040 (1)
N(1)	0.1750 (6)	0.3930 (5)	0.8153 (2)	0.026 (1)
N(3)	0.1946 (7)	0.3514 (6)	0.6993 (2)	0.035 (1)
C(2)	0.1558 (7)	0.2925 (6)	0.7631 (3)	0.030 (1)
C(4)	0.2433 (8)	0.5033 (7)	0.7080 (3)	0.031 (1)
C(5)	0.2947 (7)	0.6189 (8)	0.6599 (3)	0.043 (1)
C(6)	0.3367 (9)	0.7617 (7)	0.6877 (3)	0.043 (1)
C(7)	0.3275 (9)	0.7885 (7)	0.7629 (3)	0.042 (1)
C(8)	0.2766 (8)	0.6739 (6)	0.8094 (3)	0.033 (1)
C(9)	0.2313 (7)	0.5292 (6)	0.7818 (2)	0.026 (1)
C(10)	-0.1952 (7)	0.3931 (5)	1.0353 (2)	0.024 (1)
C(11)	-0.1768 (8)	0.5674 (5)	1.0299 (2)	0.028 (1)
C(12)	-0.1600 (6)	0.6310 (5)	0.9539 (2)	0.025 (1)

Table 2. Selected geometric parameters (Å, °)

Cu—O(1)	1.990 (3)	O(2)—C(12)	1.249 (5)
Cu—O(2)	1.954 (3)	O(3)—C(10)	1.250 (6)
Cu—O(3)	1.970 (3)	O(4)—C(12)	1.250 (5)
Cu—O(5)	2.253 (4)	C(10)—C(11)	1.528 (6)
Cu—N(1)	1.978 (4)	C(11)—C(12)	1.530 (6)
O(1)—C(10)	1.255 (6)		
O(1')—Cu—O(2)	167.6 (2)	O(5)—Cu—N(1)	95.1 (2)
O(1')—Cu—O(3)	84.9 (2)	O(1)—C(10)—O(3)	124.6 (4)
O(1')—Cu—O(5)	98.1 (1)	O(2)—C(12)—O(4)	122.2 (4)
O(2)—Cu—O(5)	93.9 (2)	O(1)—C(10)—C(11)	115.9 (4)
O(2)—Cu—O(3)	89.3 (2)	O(3)—C(10)—C(11)	119.9 (4)

O(3)—Cu—O(5)	102.3 (2)	O(2)—C(12)—C(11)	121.5 (4)
O(1')—Cu—N(1)	91.9 (2)	O(4)—C(12)—C(11)	116.3 (4)
O(2)—Cu—N(1)	90.4 (2)	C(10)—C(11)—C(12)	115.3 (4)
O(3)—Cu—N(1)	162.9 (2)		

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

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O(5)—H(51)...O(4')	0.93	1.84 (1)	2.748 (5)	167 (1)
O(5)—H(52)...O(3 <sup>ii</sup> )	0.93	2.16 (1)	2.742 (5)	121 (1)
N(3)—H(3)...O(4 <sup>iii</sup> )	0.86	1.98 (1)	2.797 (5)	159 (1)

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

The structure was solved by the conventional Patterson method and refined by full-matrix least-squares calculations. The H atoms of the H<sub>2</sub>O groups were located from a difference synthesis; all other H atoms were included in calculated positions (C—H and N—H of benzimidazole = 0.93 and 0.86 Å and C—H of malonate ion = 0.97 Å) and refined isotropically using a riding model, with fixed isotropic displacement parameters. All other non-H atoms were refined anisotropically.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: JZ1046). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 2-Ferrocenylquinoxaline

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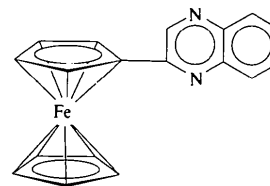
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## Abstract

In the title compound, [Fe(C<sub>13</sub>H<sub>9</sub>N<sub>2</sub>)(C<sub>5</sub>H<sub>5</sub>)], the heterocyclic ring is almost coplanar with the cyclopentadienyl ring to which it is bonded. The molecules are arranged in a herringbone pattern generated by a 2<sub>1</sub> axis and there are no significant intermolecular interactions.

## Comment

Ferrocenyl alcohols show a strong propensity both to self-assemble into oligomeric or polymeric aggregates (Sharma, Cervantes-Lee & Pannell, 1992; Ferguson, Gallagher, Glidewell & Zakaria, 1993a, 1994; Li, Ferguson, Glidewell & Zakaria, 1994) and to form host–guest complexes (Ferguson, Gallagher, Glidewell & Zakaria, 1993b; Glidewell, Ferguson, Lough & Zakaria, 1994; Ferguson, Glidewell, Lewis & Zakaria, 1995). Following the observation of a very wide range of hydrogen-bonding patterns in adducts of ferrocenyl alcohols with amines, where the ferrocene acts as a hydrogen-bond donor (Ferguson, Gallagher, Glidewell & Zakaria, 1993b; Glidewell, Ferguson, Lough & Zakaria, 1994; Ferguson, Glidewell, Lewis & Zakaria, 1995), we have now embarked on a study of functionalized ferrocenes, which are potentially strong hydrogen-bond acceptors. We report here the structure of one such ferrocene, 2-ferrocenylquinoxaline, (I), where the structural features of interest, aside from confirmation of the molecular constitution, include the relative orientation of the ferrocene and heterocyclic portions of the molecule, and the nature of the intermolecular interactions.



(I)