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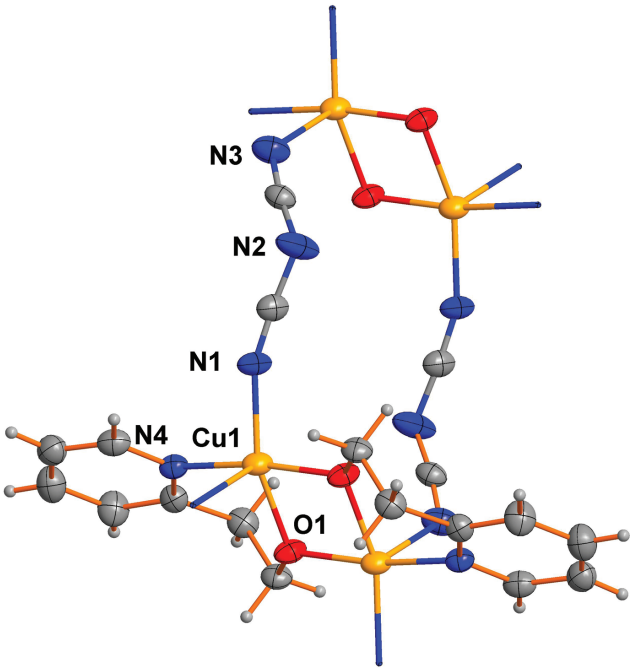
Crystal structure of *catena*-poly[(μ_2-dicyanamido-$\kappa^2N{:}N'$)-(μ_2-2-(pyridin-2-yl)ethan-1-olato-$\kappa^3N,O{:}O'$)copper(II)], $C_9H_8CuN_4O$



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Abstract

$C_9H_8CuN_4O$, triclinic, $P\bar{1}$ (no. 2), $a = 7.6740(6)$ Å, $b = 8.6401(8)$ Å, $c = 8.8869(9)$ Å, $\alpha = 109.201(2)°$, $\beta = 106.629(1)°$, $\gamma = 105.410(1)°$, $V = 489.21(8)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0406$, $wR_{ref}(F^2) = 0.0993$, $T = 298(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.20 × 0.14 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	2.21 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	2520, 1710, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1351
$N(param)_{refined}$:	136
Programs:	Bruker programs [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
Cu1	0.51395(7)	0.44079(6)	0.32654(7)	0.0417(2)
O1	0.3834(4)	0.3509(3)	0.4552(4)	0.0432(7)
N1	0.7298(5)	0.5400(5)	0.2647(5)	0.0492(9)
C1	0.3281(6)	0.1747(5)	0.4360(6)	0.0445(10)
H1A	0.1846	0.1155	0.3835	0.053*
H1B	0.3760	0.1764	0.5503	0.053*
N2	1.0708(6)	0.6740(5)	0.3067(6)	0.0778(14)
C2	0.4104(7)	0.0707(6)	0.3236(6)	0.0473(11)
H2A	0.5539	0.1297	0.3776	0.057*
H2B	0.3749	−0.0476	0.3193	0.057*
N3	1.2734(6)	0.5396(5)	0.1896(6)	0.0601(10)
C3	0.3371(6)	0.0525(5)	0.1400(5)	0.0408(9)
N4	0.3865(5)	0.2047(4)	0.1219(4)	0.0387(8)
C4	0.2245(7)	−0.1099(6)	−0.0036(6)	0.0568(12)
H4	0.1889	−0.2141	0.0106	0.068*
C5	0.1641(7)	−0.1177(7)	−0.1701(6)	0.0628(13)
H5	0.0869	−0.2266	−0.2682	0.075*
C6	0.2197(7)	0.0368(6)	−0.1874(6)	0.0532(12)
H6	0.1837	0.0350	−0.2973	0.064*
C7	0.3298(6)	0.1950(6)	−0.0393(6)	0.0464(11)
H7	0.3668	0.3003	−0.0513	0.056*
C8	0.8893(6)	0.5934(5)	0.2791(6)	0.0449(10)
C9	1.1695(6)	0.5913(6)	0.2379(6)	0.0450(10)

A part of the polymeric title crystal structure is shown in the figure. Table 1 and Table 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized by the reaction of $CuCl_2$ (1 mmol), 2-(2-pyridyl)ethanol (2 mmol) and $Na(N(CN)_2)$ (2 mmol) in methanol (10 mL). The mixture was stirred for 6 h, then a colourless solution formed. The resulting solution was filtered. The filtrate was allowed to stand for a few days at room temperature until light-pink crystals were obtained. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a dichloromethane/*n*-hexane solution (30%, m.p. 290−291 K).

Experimental details

All H atoms were placed geometrically and treated as riding on their parent atoms, with C—H 0.96, with $U_{iso}(H) = 1.5U_{eq}(C)$.

Discussion

The rational design, synthesis and characterization of supermolecular frameworks are of great interest [4, 5]. One of the greatest challenges in this area is the construction of porous materials from metal ions and organic ligands as building blocks [6, 7]. As part of our search for new porous metal-organic frameworks, we are studying complexes of transition metals with thiocyanato ligands.

As shown in the figure, the complex is a polymer. Every unit possesses binuclear structures consisting of two Cu atoms, two dicyanamido ligands and two 2-(pyridin-2-yl)ethan-1-olato ligands. The asymmetric unit contains one half of such a dinuclear unit. The Cu atom is coordianted by two nitrogen atoms from two different dicyanamide ligands and by two O atoms and one N atoms from organic ligand. Adjacent units are linked to one-dimensional chain polymer by bridging bonds of dicyanamido ligands. Bond lengths and angels are in the expected ranges [8].

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References

1. Bruker. *APEX2*, *SAINT* and *SADABS*. Brucker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: *SHELXT*-Integrated space-group and crystal-structure determination. Acta Crystallogr. **C71** (2015) 3–8.
3. Sheldrick, G. M.: A short history of *SHELX*. Acta Crystallogr. **A64** (2008) 112–122.
4. Liu, Q. Y.; Yang, Y. T.; Lv, X. T.; Ding, Y. N.; Zhang, Y. Z.; Jing, J. J.; Xu, C. X.: One-step synthesis of uniform nanoparticles of porphyrin functionalized ceria with promising peroxidase mimetics for H_2O_2 and glucose colorimetric detection. Sens. Actuator B Chem. **240** (2017) 726–734.
5. Liu, Q. Y.; Zhao, Q. R.; Li, Y.; Wang, X. Y.: $CdCl_2{\cdot}H_2O$ Nanorods oriented parallel on the langmuir film of (phthalocyaninato) [tetrakis(4-pyridyl)porphyrinato] cerium complex. CrystEngComm. **14** (2012) 1105–1110.
6. Yaghi, O. M.; Davis, C. E.; Li, G.; Li, H. L.: Selective guest binding by tailored channels in a 3-D porous zinc(II)-benzenetricarboxylate network. J. Am. Chem. Soc. **119** (1997) 2861–2868.
7. Siaw-Lattey, C.; Zhang, H. M.; Son, M. Y.: Synthesis and characterization of a versatile bis(pyridylether) ligand and its complexes with Ag(I), Cu(II) and Co(II) Polyhedron **24** (2005) 785–790.
8. Zhu, X.; Yong-feng Wang, Y.-F.: Crystal structure of bis(dicyanamido-k^1*N*)-tetrakis[1-benzyl-1*H*-1,2,4-triazole-k^1*N*]cobalt(II),$CoC_{40}H_{36}N_{18}$. Z. Kristallogr. NCS **231** (2016) 1175–1176.