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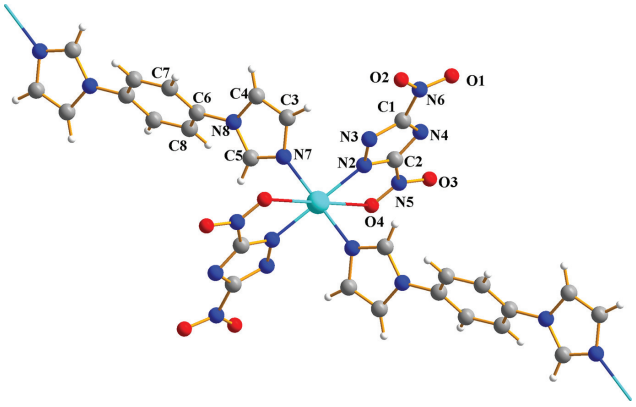
The crystal structure of *catena*-poly[bis-(3,5-dinitro-1,2,4-triazolato-κ²*N*:*O*)-(μ₂-1,4-bis(1-imidazolyl)benzene-κ²*N*:*N*′)copper(II)], $C_{16}H_{10}CuN_{14}O_8$



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Abstract

$C_{16}H_{10}CuN_{14}O_8$, monoclinic, $C2/c$ (no. 15), $a = 13.4851(13)$ Å, $b = 12.3808(12)$ Å, $c = 13.2151(13)$ Å, $\beta = 97.3040(10)°$, $V = 2188.4(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0310$, $wR_{ref}(F^2) = 0.1127$, $T = 296(2)$ K.

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A part of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

1,1′-(1,4-Phenylene)bis-1*H*-imidazole (21 mg; 0.1 mmol), sodium 3,5-dinitro-1,2,4-triazolate (50 mg; 0.25 mmol) and copper(II) nitrate trihydrate (24 mg; 0.1 mmol) were dissolved in 15 mL H_2O at 80 °C by adding 2 drops of concentrated nitric acid. The solution was transfered into a 25 mL sealed Teflon-lined stainless steel vessel. The system was heated to 130 °C for 2 days under autogenous pressure and allowed to naturally cool to room temperature. Blue block crystals were isolated by filtration, washed with water and dried in air. Yield: 78.4%. **Anal. Calc.:** C, 32.58; H, 1.71; N, 33.24. Found: C, 32.73; H, 1.92; N, 33.36%.

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	0.15 × 0.12 × 0.11 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.6°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6462, 2487, 0.019
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2170
$N(param)_{refined}$:	178
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.250000	0.250000	0.500000	0.02238(14)
O1	0.05587(16)	0.51234(17)	0.11316(13)	0.0683(6)
O2	0.12606(16)	0.60413(15)	0.24168(14)	0.0658(5)
O3	0.09686(17)	0.10323(17)	0.20575(15)	0.0709(6)
O4	0.18541(16)	0.09662(14)	0.35483(15)	0.0540(5)
N2	0.18328(10)	0.31349(12)	0.36947(11)	0.0265(3)
N3	0.17254(13)	0.41968(13)	0.34806(13)	0.0295(3)
N4	0.10724(12)	0.32474(15)	0.20877(12)	0.0340(4)
N5	0.14198(15)	0.14585(15)	0.28125(15)	0.0420(4)
N6	0.10206(13)	0.52001(15)	0.19826(13)	0.0413(4)
N7	0.38342(10)	0.28829(13)	0.45965(12)	0.0279(3)
N8	0.54559(11)	0.29616(13)	0.45581(12)	0.0290(3)
C1	0.12798(13)	0.41967(16)	0.25245(14)	0.0297(4)
C2	0.14390(16)	0.26183(14)	0.28522(16)	0.0281(4)
C3	0.40435(15)	0.37468(18)	0.40028(18)	0.0451(5)
H3	0.357089	0.421621	0.367129	0.054*
C4	0.50392(15)	0.38089(19)	0.39749(19)	0.0480(6)
H4	0.537596	0.432000	0.363152	0.058*
C5	0.47106(16)	0.24309(12)	0.49179(16)	0.0259(4)
H5	0.479504	0.182596	0.533720	0.031*
C6	0.64997(14)	0.27214(16)	0.47765(14)	0.0263(4)
C7	0.71734(16)	0.35625(15)	0.49875(16)	0.0339(4)
H7	0.694807	0.427291	0.497320	0.041*
C8	0.68184(14)	0.16594(16)	0.47806(15)	0.0318(4)
H8	0.636098	0.110223	0.462958	0.038*

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Experimental details

Indexing was performed using APEX2 (Difference Vectors method). Data integration and reduction were performed using SaintPlus. Absorption correction was performed by multiscan method implemented in SADABS [1]. The structure was refined against F^2 with anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms on carbon and nitrogen and oxygen were calculated in ideal positions with isotropic placement parameters set to $1.2U_{eq}$ of the attached atoms.

Comment

As a rigid ligand, 1,1′-(1,4-phenylene)bis-1*H*-imidazole, also named as 1,4-bis(1-imidazolyl)-benzene has been widely used in the synthesis of various mixed-ligand coordination polymers [5, 6]. A large number of these coordination polymers were constructed by mixed ligands including carboxylate ligands and other *N*-donor molecules, for example, nitrogen-rich heterocyclic compounds. For the title complex, 3,5-dinitro-1,2,4-triazolate anion is selected as the mixed ligand, which was mostly used as a component of energetic materials [7, 8].

The asymmetric unit of the title structure contains one half of a Cu(II) cation, one half of a 1,4-bis(1-imidazolyl) benzene (bib) and one 3,5-dinitro-1,2,4-triazolate (dnt^-) anion. Cu1 is six-coordinated by four N atoms and two O atoms from two bib ligands and two dnt^- anions leading to a distorted octahedral environment. The Cu(II) centres are bridged by bib ligands, giving a one-dimensional structure. The Cu–N bond length is 1.9975(14) Å and 2.0020(14) Å, while the distance between Cu and O atoms is 2.7605(2), which is close to the reported value 2.7312(5) Å [9].

References

1. Bruker. SMART, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: SHELXT – Integrated space-group and crystal-structure determination. Acta Crystallogr. **A71** (2015) 3–8.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
4. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Ver. 4.0. Crystal Impact, Bonn, Germany (2015).
5. Qi, Y.; Che, Y. X.; Zheng, J. M.: Self-penetrating and interpenetrating 3D metal-organic frameworks constructed from the rigid 1,4-bis (1-imidazolyl)-benzene ligand and aromatic carboxylate. Cryst. Growth Des. **8** (2008) 3602–3608.
6. Wang, L.; Ma, L.: Crystal structure of *catena*-poly [diaqua-bis (3-(4-trifluoromethyl-phenyl)-acrylato-κO^1)-(μ_2-1, 4-bis (1-imidazolyl) benzene-κ$^2N^3$: $N^{3\prime}$) cobalt (II)], $C_{32}H_{26}CoF_6N_4O_6$. Z. Kristallogr. NCS **235** (2020) 611–613.
7. Huynh, M. H. V.; Hiskey, M. A.; GILARDI, R.: Preparation and explosive properties of tetraamminebis (3,5-dinitro-1,2,4-triazolato-N^1) copper (II). J. Energ. Mater. **23** (2005) 27–32.
8. Xue, H.; Twamley, B.; Jean'ne, M. S.: Energetic salts of substituted 1,2,4-triazolium and tetrazolium 3,5-dinitro-1,2, 4-triazolates. J. Mater. Chem. **15** (2005) 3459–3465.
9. Degtyarik, M. M.; Lyakhov, A. S.; Ivashkevich, L. S.; Voitekhovich, S. V.; Sukhanov, G. T.; Grigoriev, Y. V.: 1-(2-Hydroxyethyl)-3-nitro-1,2,4-triazole and its complexes with copper(II) chloride and copper(II) perchlorate. Z. Anorg. Allg. Chem. **638** (2012) 1–8.