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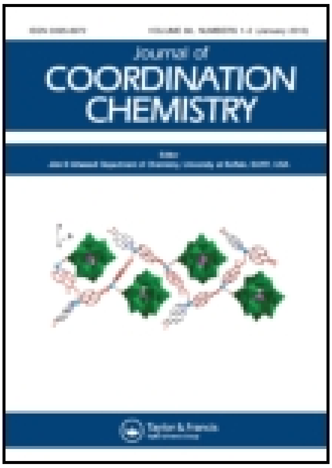

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Structure and magnetic properties of binuclear [Cu(amppz)(μ-NC)Fe$(CN)_4$NO] (amppz = 1,4-bis(3-aminopropyl)piperazine)

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Structure and magnetic properties of binuclear [Cu(amppz)(μ-NC)Fe(CN)$_4$NO] (amppz = 1,4-bis(3-aminopropyl) piperazine)

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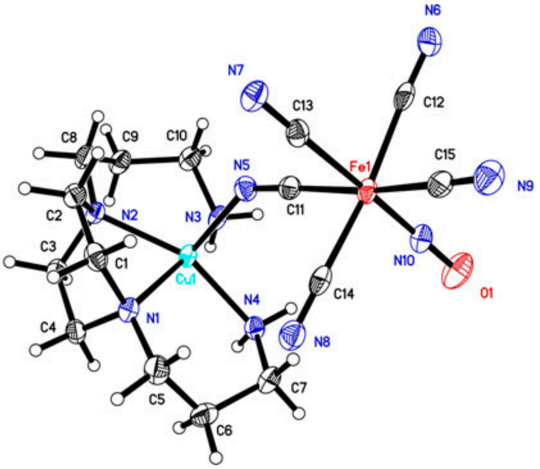

The heterobimetallic compound [Cu(amppz)(μ-NC)Fe(CN)$_4$NO] (amppz = 1,4-bis(3-aminopropyl) piperazine) has been prepared by the reaction of [Cu(amppz)(ClO$_4$)]ClO$_4$ and Na$_2$[Fe(CN)$_5$NO]·2H$_2$O in aqueous solution and was characterized by IR spectroscopy, magnetic measurement, and X-ray single-crystal diffraction. The neutral complex has a cyanide-bridged binuclear structure in which the iron(II) is six-coordinate by five carbons from cyano groups (one of them forms a bridge) and one nitrogen from nitrosyl in an octahedral arrangement, whereas the copper(II) is five-coordinate by four amppz-nitrogens and one cyanide-nitrogen in a distorted square-pyramidal geometry. Magnetic investigation revealed a weak antiferromagnetic intermolecular interaction between the copper(II) ions with $T_N = 6$ K.

Keywords: Copper(II) complexes; Nitroprusside; Cyano-bridged complexes; Heterobinuclear; X-ray structure analysis

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1. Introduction

Cyano-bridged homo- and heterometallic complexes attract attention in coordination chemistry, especially in the context of their fascinating structures with interesting magnetic, electrochemical, magneto-optical, and zeolitic properties [1–6]. For instance, copper(II) cyanide-bridged nitroprussides^{2-}, $[Fe(CN)_5NO]^{2-}$, are suitable model compounds for magneto-structural studies at low temperatures because the diamagnetic nitroprusside anion may bridge paramagnetic ions partially coordinated with amine ligands and thus form a rich variety of structures, such as discrete dinuclear to polynuclear species, 1-D chain, 2-D sheet, and 3-D structures [7–9]. In addition, a terminal cyanide can participate in hydrogen bonds that play an important role in forming supramolecular structures and can serve as a possible exchange path for magnetic interaction [5]. Copper(II) nitroprussides have also been tested on antileukemic activity [10] and as precursors for the preparation of the copper spinel ferrite $CuFe_2O_4$ nanocrystals that are interesting for their wide catalytic activity [7]. This type of coordination compound can be built by linking the appropriate building blocks by the brick and mortar method [11]. While $[Fe(CN)_5NO]^{2-}$ behaves as mortar, copper(II) coordinated by suitable ligands forms the brick.

In this article we report the synthesis, structural characterization, and magnetic properties of a cyanide-bridged Cu^{II}–Fe^{II} binuclear complex, $[Cu(amppz)(\mu\text{-}NC)Fe(CN)_4NO]$ (amppz = 1,4-bis(3-aminopropyl)piperazine).

2. Experimental

2.1. *Materials and instruments*

All chemicals used for the synthesis were analytical reagent grade and used as received. $[Cu(amppz)(ClO_4)]ClO_4$ was obtained by the direct reaction between $Cu(ClO_4)_2{\cdot}6H_2O$ and amppz in the mole ratio 1 : 1 in aqueous solution as previously described [12]. Elemental analysis (C, H, N) was performed on a Elementar EL III analyzer. IR spectrum (400–4000 cm^{-1}) was recorded on a FTIR spectrometer Nicolet 6700 using the ATR method. Magnetization measurement from 1.8 to 300 K was carried out on polycrystalline sample of complex, at magnetic field 0.5 T, using a Quantum Design SQUID magnetometer (type MPMS XL-5). A correction for diamagnetism of the constituting atoms was calculated from Pascal's constants [13]. The temperature-independent paramagnetism per copper(II) was assumed to be $60 \times 10^{-6}\ cm^3\ M^{-1}$.

2.2. *Preparation of $[Cu(amppz)(\mu\text{-}NC)Fe(CN)_4NO]$*

Solution of $[Cu(amppz)(ClO_4)]ClO_4$ (0.65 g, 1.4 mM) in water (25 mL) was mixed with solution of $Na_2[Fe(CN)_5NO]{\cdot}2H_2O$ (0.42 g, 1.4 mM) in water (25 mL). The reaction mixture was allowed to undergo slow evaporation whereupon dark purple crystalline product was obtained. The solid was filtered off, washed with a small amount of water, and dried in air. Yield: 0.50 g, 75%. Anal. Calcd for $C_{15}H_{24}CuFeN_{10}O$ (%): C, 37.55; H, 5.04; N, 29.19. Found: C, 37.05; H, 4.97; N, 29.23. IR (cm^{-1}): 3807w, 3315s, 3258s, 3151s, 2937sh, 2886m, 2139vs, 1980w, 1919vs, 1648m, 1468m, 1456w, 1433w, 1401w, 1348w, 1315w, 1288w, 1265w, 1224w, 1206m, 1174m, 1135w, 1103m, 1078m, 1063w, 1015m, 978w, 947w, 910w, 835w, 789m, 697w, 658m, 648w, 512w, 484w, and 430w.

2.3. *X-ray crystal structure determination*

Diffraction data for the studied compound were collected with Cu*Kα* radiation at 100 K using a Bruker Microstar diffractometer equipped with a Platinum 135 CCD Detector, a Helios optics, and a Kappa goniometer. APEX2 and SAINT [14] were used for data collection, and cell refinement and data reduction, respectively. Absorption correction was carried out with SADABS [15], and the structure was solved using SHELXS97 [16] and refined by full-matrix least squares on F^2 with SHELXL97 [16]. The resulting crystal data and details concerning data collection and refinement are quoted in table 1, while selected interatomic parameters are given in table 2.

Table 1. Crystal data and structure refinement for [Cu(amppz)(μ-NC)Fe(CN)$_4$NO].

Empirical formula	$C_{15}H_{24}CuFeN_{10}O$
Formula weight	479.83
Temperature (K)	100
Wavelength (Å)	1.54178
Crystal system, space group	Monoclinic, *P2₁/c*
Unit cell dimensions (Å, °)	
a	10.5603(5)
b	10.7805(5)
c	18.3122(8)
β	95.561(2)
Volume (Å^3)	2074.95(16)
Z	4
D_{Calcd} (g cm^{-3})	1.536
Absorption coefficient (mm^{-1})	7.107
F (0 0 0)	988
Crystal size (mm^3)	0.18 × 0.16 × 0.13
θ Range for data collection (°)	4.206–70.536
Index ranges	$-12 \le h \le 12$, $-13 \le k \le 13$, $-21 \le l \le 16$
Reflection collected/unique	22712/3899 [$R_{int} = 0.077$]
Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least squares on F^2
Data/restraints/parameters	3899/0/265
Goodness-of-fit on F^2	1.079
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0433$, $wR_2 = 0.1107$
R indices (all data)	$R_1 = 0.0488$, $wR_2 = 0.1144$
Largest difference between peak and hole (e Å^{-3})	0.747, −0.558

Table 2. Selected bond distances (Å) and angles (°) for [Cu(amppz)(μ-NC)Fe(CN)$_4$NO].

Cu(1)–N(4)	2.017(3)	Fe(1)–N(10)	1.651(2)
Cu(1)–N(3)	2.029(3)	Fe(1)–C(11)	1.934(3)
Cu(1)–N(1)	2.040(3)	Fe(1)–C(15)	1.939(3)
Cu(1)–N(2)	2.059(2)	Fe(1)–C(12)	1.942(3)
Cu(1)–N(5)	2.176(3)	Fe(1)–C(13)	1.950(3)
O(1)–N(10)	1.146(3)	Fe(1)–C(14)	1.953(3)
N(3)–Cu(1)–N(1)	159.95(11)	C(12)–Fe(1)-C(14)	170.42(12)
N(4)–Cu(1)–N(2)	156.51(11)	N(5)–C(11)–Fe(1)	176.9(3)
C(11)–Fe(1)–C(15)	169.48(12)	C(11)–N(5)–Cu(1)	142.1(2)
N(10)–Fe(1)–C(13)	178.76(13)	O(1)–N(10)–Fe(1)	178.6(3)

3. Results and discussion

3.1. *Crystal structure*

Single-crystal X-ray analysis of the complex revealed a cyanide-bridged binuclear structure in which iron(II) is six-coordinate by five cyanides (one of them is bridging) and one nitrosyl (chromophore FeC_5N), and copper(II) is five-coordinate by tetradentate amppz and one cyanide (figure 1). The copper is coordinated by four nitrogens from 1,4-bis(3-aminopropyl)piperazine and one nitrogen from a cyano bridge in a distorted square-pyramidal arrangement with four amppz-nitrogens bonded in the basal plane and one cyano-nitrogen as apical ligand. The in-plane Cu–N bond lengths range from 2.017(3) to 2.059(2) Å, and axial Cu–N bond distance of 2.176(3) Å is significantly longer than the in-plane Cu–N bond lengths. The angle N(1)–Cu(1)–N(2) (73.68(10)°) is smaller than the ideal value of 90° because this angle is part of a five-membered chelate. The value of the τ parameter (0.06) confirms the square-pyramidal geometry of the coordination polyhedron (ideal τ value for the square-pyramidal coordination sphere is 0 and for the trigonal-bipyramidal arrangement should be equal to 1) [17]. The mean bond lengths Fe–C (1.94 Å) and Fe–N (1.65 Å) are in accord with the reported values for similar compounds [7–9]. The Fe–C bonds in the nitroprusside anion for terminal cyanide vary from 1.939(3) to 1.953(3) Å, while the same distance associated with a bridging CN is 1.934(3) Å. The value for Fe–N–O is close to straight angle (178.6(3)°), and the bridging cyanide coordinates to copper in a bent fashion with the C–N–Cu 142.1(2)° bond angle. The intramolecular Fe···Cu distance is 4.954 Å, and the shortest intermolecular Cu···Cu distance is 7.309 Å.

The Cu(II)–Fe(II) units are joined through intermolecular H-bonding between secondary amine hydrogens of amppz and the terminal cyanide in $[Fe(CN)_5NO]^{2-}$, resulting in a

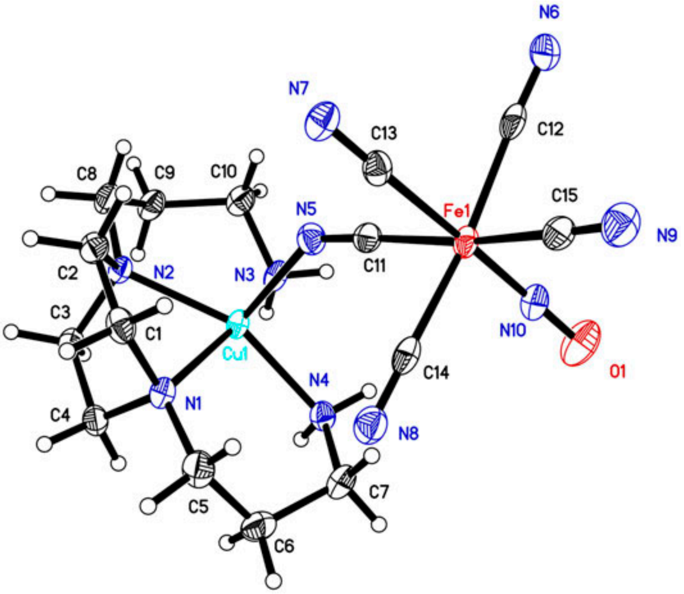

Figure 1. Molecular structure of [Cu(amppz)(μ-NC)Fe(CN)$_4$NO] with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

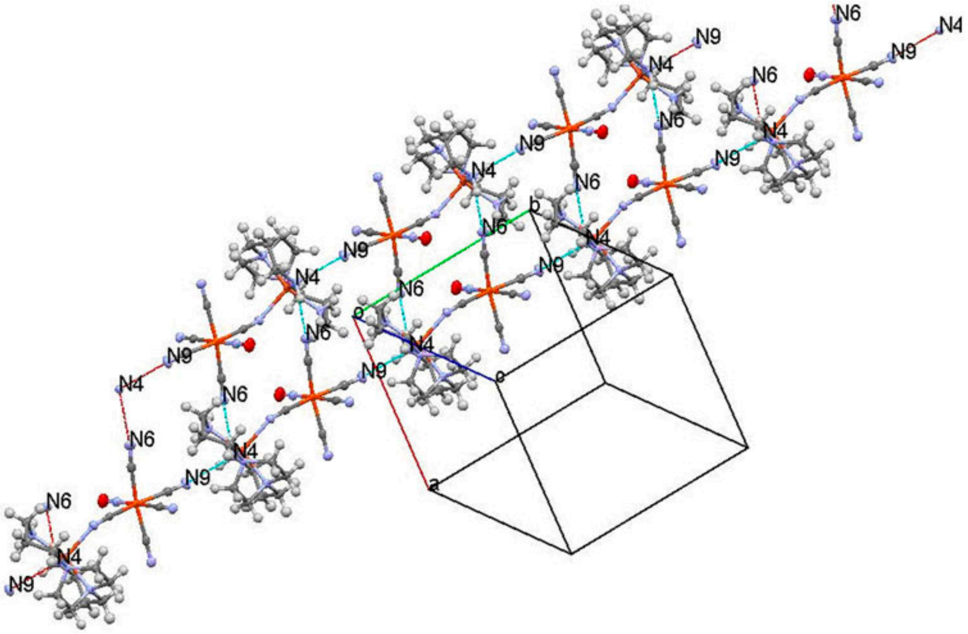

Figure 2. A view of the crystal structure of [Cu(amppz)(μ-NC)Fe(CN)$_4$NO]. Dashed lines indicate possible hydrogen bonds. Selected hydrogen bonding parameters (Å, °) are as follows: N(4)–H(4A)···N(6): D–H = 0.88(4), H–A = 2.12(4), D···A = 2.996(4), DH···A = 171(3); N(4)-H(4B)···N(9): D–H = 0.86(3), H–A = 2.22(3), D···A = 3.014(4), D–H···A = 153(3).

ladder supramolecular structure along the *b*-axis (figure 2). These H-bonds with distances and angles ranging from 3.00 to 3.01 Å and with 153°–171°, respectively, have values typical for hydrogen bonds of medium strength (D···A ≈ 2.5–3.2 Å and angle NHO ≈ 130°–180°) [18].

3.2. *Magnetic properties*

The temperature dependence of the magnetic susceptibility of [Cu(amppz)(μ-NC)Fe(CN)$_4$NO] was measured from 1.8 to 300 K. Thermal relation of the magnetic susceptibility is shown in figure 3 in the form of χ_m *versus.* T and χ_mT *versus.* T. The χ_mT value (0.389 cm^3 K M^{-1}) at room temperature is close to the spin-only value for an isolated copper(II) ion (0.375 cm^3 K M^{-1} for $S = 1/2$) [19]. Upon lowering of temperature, the χ_mT value decreases to 0.0204 cm^3 K M^{-1} at 1.8 K. This indicates the presence of a weak antiferromagnetic interaction ($T_N = 6$ K) observed at the lowest temperatures. Such antiferromagnetic intermolecular interactions between copper(II) ions might be explained by the presence of a variety of non-covalent interactions in the crystal structure of the compound, as shown in figure 2. There is no other pathway for antiferromagnetic exchange interactions than the system of hydrogen bonds. Moreover, the studied compound exhibits higher Neél temperature than the 1D chain polymeric complex [Cu(en)$_2$Fe(CN)$_5$NO] (en = ethylenediamine) [20] where superexchange pathway between the copper ions is mediated by the diamagnetic nitroprusside anion. Magnetic data were calculated and compared using two

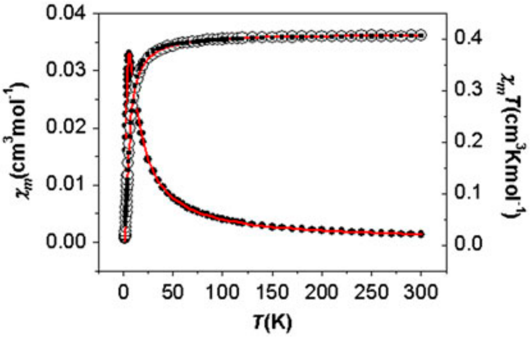

Figure 3. The temperature dependence of χ_m (●) and $\chi_\mathrm{m}T$ (○) for [Cu(amppz)(μ-NC)Fe(CN)$_4$NO]. Solid line (—) and squares (■) present calculated data using two models of interactions, BB model and chain Bonner–Fisher (BF) model, respectively.

models of interactions: Bleaney–Bowers (BB) expression of two interacting copper ions *A* and *B,* based on isotropic Heisenberg–Dirac–Van Vleck Hamiltonian $H = -JS_\mathrm{A}S_\mathrm{B}$ ($S_\mathrm{A} = S_\mathrm{B} = 1/2$) [13]:

$$\chi_\mathrm{m} = \frac{Ng^2\beta^2}{kT\left[3 + \exp\left(-\frac{J}{kT}\right)\right]}$$

and chain BF model of 1-D equally spaced copper(II) ions, with the spin Hamiltonian $H = -J\sum_{\mathrm{i=1}}^{\mathrm{n-1}} S_{\mathrm{A_i}}S_{\mathrm{A_{i+1}}}$, describing the isotropic interaction between nearest neighbor ions (S = 1/2) [13]:

$$\chi_\mathrm{m} = \frac{Ng^2\beta^2}{kT}\frac{0.25 + 0.074975x + 0.075235x^2}{1.0 + 0.9931x + 0.172135x^2 + 0.757825x^3}$$

with $x = |J|/kT$. In both cases, a molecular field model of magnetic interactions [13] was added to characterize any other magnetic interactions:

$$\chi_\mathrm{m}^\mathrm{corr} = \frac{\chi_\mathrm{m}}{1 - \frac{2zJ'\chi_\mathrm{m}}{Ng^2\beta^2}}$$

where χ_m is the molar magnetic susceptibility, zJ' is the molecular exchange parameter between z nearest neighbors, and other symbols have their usual meaning. Good extrapolation to experimental data has been obtained for both models (see figure 3 and table 3) with a good agreement factor *R* defined as follows:

Table 3. Spin Hamiltonian parameters resulting from fitting the magnetic data.

Model of interaction	g	J (cm^{-1})	$\lvert J\rvert$ (cm^{-1})$^\mathrm{a}$	zJ' (cm^{-1})	R
Bleaney–Bowers	2.05	−6.72	6.66	−0.28	2.1×10^{-5}
Bonner–Fisher	2.09	$\lvert J\rvert = 6.15$	6.50	1.72	1.6×10^{-3}

$^\mathrm{a}$From $T_\mathrm{max.}$

$$R = \sum_{\mathrm{i}=1}^{n} \frac{(\chi_{\mathrm{i}}^{\mathrm{exp}} T - \chi_{\mathrm{i}}^{\mathrm{calc}} T)^2}{(\chi_{\mathrm{i}}^{\mathrm{exp}} T)^2}$$

Careful analysis of hydrogen bonds parameters (figure 2) allows us to suggest that observed antiferromagnetic coupling is a result of interactions between two copper(II) ions [with the shortest intermolecular Cu···Cu distance (7.309 Å)] through nitroprusside anions and hydrogen bonds system. The supramolecular structure based on N–H···NC hydrogen bonds is the pathway (Cu–N–H···NC–Fe–CN–Cu, figure 2) that causes antiferromagnetic interaction. Binuclear $[\mathrm{Cu(hto)}(\mu\text{-NC})\mathrm{Fe(CN)_4NO}]\cdot 2\mathrm{H_2O}$ (hto = 1,3,6,9,11,14-hexaazatricyclo[12.2.1.1^{6,9}]octadecane) exhibits supramolecular two-dimensional structure based on N–H···NC, N–H···O, O–H···N, and O–H···O hydrogen bonds with significantly weaker intermolecular antiferromagnetic interaction ($zJ' = -2.37$ cm^{-1}, $T_\mathrm{N} < 2$ K) [7] than in our compound. This fact may be explained by the longer exchange pathway (Cu–N–H···O–H···NC–Fe–CN–Cu). Similar results were obtained for $[\mathrm{Cu(L)}(\mu\text{-NC})\mathrm{Fe(CN)_4NO}]\cdot 2\mathrm{H_2O}$ (L = 3,10-bis(2-hydroxymethyl)-1,3,5,8,10,12-hexaazacyclotetradecane) ($zJ' = -0.41$ cm^{-1}, $T_\mathrm{N} < 2$ K) [21]. Against it, cryomagnetic investigations revealed weak intermolecular ferromagnetic interaction with $J = 5.04$ cm^{-1} for binuclear $[\mathrm{Cu(bpy)_2}(\mu\text{-NC})\mathrm{Fe(CN)_4NO}]\cdot 3\mathrm{H_2O}$ (bpy = 2,2′-bipyridine) [22] where magnetic coupling between two copper(II) ions could be caused through water and bpy (Cu···O–H···C–C–N–Cu).

Exchange interactions through a priori weak supramolecular interactions, such as hydrogen-bond bridges linking metal centers, have been only rarely investigated [7, 23–32]. It is obvious that the distance between paramagnetic centers is not the only factor influencing the interaction, and the form of magnetic dimensionality could be different from its structural dimensionality [26–28]. A theoretical investigation of exchange coupling through hydrogen bonding indicates that hydrogen bonds are responsible for fixing molecules in position allowing overlap of magnetic orbitals [32].

3.3. *IR spectrum*

Characteristic vibrations of the nitroprusside anion $[\mathrm{Fe(CN)_5NO}]^{2-}$ were observed [33, 34]. The strong absorption peak at 2139 cm^{-1} is assigned to the cyanide; the compound exhibits only one peak corresponding to *ν*(CN) despite the presence of bridging and terminal cyanides in the structure. Bridging cyanide frequencies *ν*(CNb) in cyano-bridged complexes are generally observed at a higher frequency than those of terminal cyanides *ν*(CNt), but exceptions to this trend are observed too [35]. The strong broad absorption at 1919 cm^{-1} corresponds to *ν*(NO) and the bands at 3151–3315 cm^{-1} and 2886–2937 cm^{-1} belong to the N–H stretching vibration, and to the *ν*(C–H), respectively.

4. Conclusion

A bimetallic copper(II)–nitroprusside complex was prepared and characterized by physical techniques. X-ray analysis revealed the cyano-bridged binuclear structure, and magnetic susceptibility measurement confirmed weak antiferromagnetic intermolecular interaction between two copper(II) ions mainly due to hydrogen bonds. The careful analysis of the packing diagram is a very important step in understanding the magnetic behavior of such coordination compounds, and in identifying the possible intermolecular exchange pathways.

Supplementary material

The crystallographic data for the studied compound have also been deposited with the Cambridge Crystallographic Data Center as supplementary material with the deposition number: CCDC 980817. Copy of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223/336 033; E-mail: deposit@ccdc.cam.ac.uk).

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