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Isostructural Trigonal-Bipyramidal Cu^{II} and Nominally 1% Cu^{II}-Doped Zn^{II} Complexes with N$_5$ Ligation

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Abstract. [Cu(C$_6$H$_{18}$N$_4$)(C$_{10}$H$_{10}$N$_2$)](ClO$_4$)$_2$, (1-benzylimidazole)[N,N-bis(2-aminoethyl)-1,2-ethane-diamine]copper(II) diperchlorate (1), M_r = 566.88, monoclinic, $P2_1/c$, a = 10.549 (1), b = 9.2465 (9), c = 25.256 (2) Å, β = 101.42 (1)°, V = 2415 (1) Å^3, Z = 4, D_m = 1.54 (1), D_x = 1.559 Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 1.18 mm^{-1}, $F(000)$ = 1172, T = 296 (1) K, R = 0.048, wR = 0.069 for 3063 reflections. [Zn$_{0.99}$Cu$_{0.01}$(C$_6$H$_{18}$N$_4$)(C$_{10}$H$_{10}$N$_2$)](ClO$_4$)$_2$, 1% Cu^{II}-doped (1-benzylimidazole)[N,N-bis(2-amino-ethyl)-1,2-ethanediamine]zinc(II) diperchlorate (2), M_r = 568.72, monoclinic, $P2_1/c$, a = 10.708 (1), b = 9.229 (2), c = 25.205 (2) Å, β = 101.867 (8)°, V = 2438 (1) Å^3, Z = 4, D_m = 1.54 (1), D_x = 1.549, λ(Mo $K\alpha$) = 0.71073 Å, μ = 1.30 mm^{-1}, $F(000)$ = 1176, T = 295 (1) K, R = 0.041, wR = 0.055 for 3282 reflections. The isostructural distorted trigonal-bipyramidal MN$_5$ cations are separated by perchlorate anions. Equatorial M—N distances are similar in both cations [range 2.083 (3) to 2.105 (3) Å for (1); 2.073 (3) to 2.084 (3) Å for (2)]. In contrast, the axial M—N distances differ substantially [2.043 (3) and 1.971 (3) Å for (1); 2.255 (4) and 2.054 (3) Å for (2)]. Axial contraction in the copper complex (1) is consistent with a d_{z^2} ground state.

Introduction. As part of a long-term project designed to help understand the structural and spectroscopic properties of the active sites of selected metallo-proteins, we have prepared and characterized numerous low molecular-weight analogues (Bharadwaj, Schugar & Potenza, 1991). The goal in the present study was to prepare both a distorted trigonal-bipyramidal CuN$_5$ complex having apical imidazole

ligation and a magnetically dilute Cu^{II}-doped Zn^{II} analogue. We report here the preparation and crystal structure of Cu(3)(4).2ClO$_4$ (1), and its isostructural Cu^{II}-doped Zn^{II} analogue, Zn$_{0.99}$Cu$_{0.01}$(3)(4).2ClO$_4$ (2).

(3)   (4)   Cation in (1)
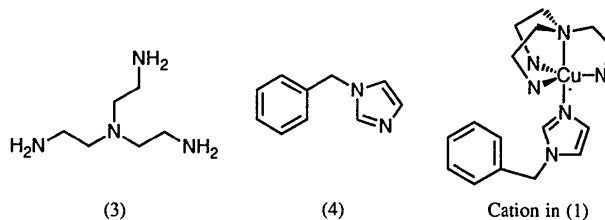
The tetradentate ligand tris(2-aminoethyl)amine (3) is well suited to impose trigonal-bipyramidal geometry, allowing molecules such as 1-benzylimi-dazole (4) to bind at the open site. Both amine oxidases and galactose oxidase are thought to be five-coordinate around the active Cu site, though the donors are mixed nitrogen and oxygen (Lontie, 1984).

Experimental. An acetonitrile solution of 230 mg (1.57 mmol) of (3), 580 mg (1.57 mmol) of [Cu(ClO$_4$)$_2$].6H$_2$O, and 250 mg (1.58 mmol) of (4) was filtered and placed in a dessicator containing tetrahydrofuran. Vapor diffusion afforded large bright-blue crystals of (1). Nominally 1% doped (2) was prepared in a similar way using a 1:99 mol % ratio of [Cu(ClO$_4$)$_2$].6H$_2$O and [Zn(ClO$_4$)$_2$].6H$_2$O.

Details of the structure solution have values for (2) in parentheses. D_m was measured by flotation using a hexane/carbon tetrachloride/1,2-dibromoethane mixture for (1) and (2); bright-blue prism, 0.19 × 0.32 ×

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0.34 mm for (1); pale-blue prism 0.32 × 0.58 × 0.22 mm for (2); Enraf–Nonius CAD-4 diffractometer; graphite-monochromated Mo $K\alpha$ radiation; θ–2θ scan. Reciprocal lattice symmetry and systematic absences consistent with the monoclinic space group $P2_1/c$. Cell constants from setting angles of 25 reflections with $16.25 \le \theta \le 20.28°$ ($15.15 \le \theta \le 20.13°$); data corrected for Lorentz, polarization and absorption [empirical correction, ψ scan; transmission coefficient range 0.98–1.00 (0.91–1.00)] effects. Variation in intensity of three standard reflections ±2.2% (±3.1%); 4531 (4311) unique reflections measured with $4 \le 2\theta \le 50°$; 3063 (3282) with $I \ge 3\sigma(I)$ used in refinement. Data collected: h 0 to 12 (0 to 12); k 0 to 10 (0 to 10); l −29 to 28 (−29 to 28). Structures solved by direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and difference Fourier techniques; H atoms from difference Fourier synthesis and at calculated positions; C—H and N—H distances 0.95 and 0.87 Å; H atoms not refined. Full-matrix refinement on F; all atoms except O atoms on one disordered perchlorate in (1) anisotropic; $w = 4(F_o)^2/[\sigma^2(F_o)^2 + 0.0016F_o^4]$. Final $R = 0.048$ (0.041), $wR = 0.069$ (0.055), $R_{int} = 0.013$ (0.013), $S = 2.28$ (1.85), $\Delta\rho_{min} = -0.38$ (−0.12), $\Delta\rho_{max} = 0.58$ (0.47) e Å^{-3}, $\Delta_{max}/\sigma = 0.12$ (0.21), number of parameters refined = 314 (334). Residuals larger than the largest negative peaks were all associated with the ClO_4 ions. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Programs from Enraf–Nonius (1983) *SDP*.

Discussion. Final positional parameters and their e.s.d.'s are given in Table 1.* A view of the cation in (2), showing the atom-numbering scheme for both cations, is given in Fig. 1. Selected bond distances and angles for both complexes are listed in Table 2.

The metal centers in both (1) and (2) are ligated by five N atoms from the ligands (3) and (4) to give approximately trigonal-bipyramidal coordination geometries as evidenced by the axial angle of 178.4 (2) [178.3 (1)°], the threefold angles, which range from 114.8 (1) to 123.8 (1) [115.3 (2) to 120.3 (1)°], and the fourfold angles, which range from 83.8 (1) to 96.9 (2) [80.5 (2) to 100.0 (2)°]. A major difference between (1) and (2) is the axial bond lengths and associated angle variations required to accommodate the M—N bond-length variation. The M—N(tertiary amine) distance in (1), Cu—N(4), is

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and interatomic distances and angles for (1) and (2) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71105 (83 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1040]

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters for* (1) *and* (2) *with values for* (2) *listed directly below those for* (1)

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq} (Å^2)
Cu	0.28354 (5)	0.18658 (5)	0.10754 (2)	3.80 (1)
Zn	0.27498 (4)	0.18625 (4)	0.10777 (2)	4.058 (8)
Cl(1)	0.3698 (1)	0.6993 (1)	0.24331 (5)	4.65 (3)
	0.36941 (9)	0.70012 (9)	0.24232 (3)	4.86 (2)
Cl(2)	0.2016 (1)	0.7150 (2)	0.01861 (6)	6.13 (3)
	0.1949 (1)	0.7173 (2)	0.02035 (4)	6.36 (3)
O(1)	0.3045 (5)	0.7896 (6)	0.2735 (2)	10.8 (1)
	0.3278 (4)	0.7326 (4)	0.1868 (2)	8.59 (9)
O(2)	0.5031 (5)	0.7186 (7)	0.2598 (2)	11.3 (2)
	0.3405 (5)	0.5566 (4)	0.2504 (2)	12.5 (1)
O(3)	0.3382 (6)	0.5571 (5)	0.2536 (2)	12.7 (2)
	0.3060 (4)	0.7879 (4)	0.2736 (1)	11.2 (1)
O(4)	0.3278 (5)	0.7266 (5)	0.1877 (2)	8.0 (1)
	0.5005 (3)	0.7209 (5)	0.2596 (2)	11.1 (1)
O(5)*	0.1246 (8)	0.8293 (9)	0.0040 (5)	17.2 (4)
	0.1138 (6)	0.8327 (8)	0.0023 (4)	14.0 (2)
O(6)	0.1579 (7)	0.5997 (9)	0.0472 (4)	11.9 (2)
	0.1389 (7)	0.6043 (8)	0.0312 (4)	19.3 (3)
O(7)	0.3227 (7)	0.754 (1)	0.0459 (3)	12.4 (3)
	0.3026 (7)	0.764 (1)	0.0515 (3)	18.8 (3)
O(8)	0.227 (1)	0.634 (1)	0.9733 (4)	15.1 (3)
	0.7783 (7)	0.3603 (8)	0.0257 (2)	13.5 (2)
O(5P)†	0.091 (3)	0.661 (3)	0.999 (1)	19.1 (9)
	0.146 (2)	0.770 (2)	0.9814 (6)	32.1 (5)
O(6P)	0.195 (2)	0.788 (2)	0.066 (1)	15.1 (6)
	0.167 (1)	0.623 (1)	0.0599 (4)	13.8 (4)
O(7P)	0.325 (2)	0.700 (2)	0.013 (1)	9.1 (4)
	0.3168 (7)	0.718 (1)	0.0199 (5)	10.7 (3)
O(8P)	0.171 (3)	0.805 (4)	0.975 (1)	21.6 (9)
	0.162 (1)	0.815 (2)	0.0608 (5)	12.8 (4)
N(1)	0.3143 (4)	0.4000 (4)	0.1333 (2)	6.1 (1)
	0.3124 (3)	0.4003 (3)	0.1304 (1)	5.75 (8)
N(2)	0.1684 (4)	0.1487 (5)	0.0307 (2)	6.3 (1)
	0.1726 (3)	0.1491 (4)	0.0294 (1)	6.16 (8)
N(3)	0.4047 (3)	0.0139 (4)	0.1391 (2)	4.84 (9)
	0.4036 (3)	0.0239 (3)	0.1400 (1)	5.08 (7)
N(4)	0.4243 (4)	0.2295 (4)	0.0646 (2)	4.59 (9)
	0.4289 (3)	0.2326 (3)	0.0611 (1)	4.76 (7)
N(5)	0.1442 (3)	0.1458 (4)	0.1471 (2)	4.40 (8)
	0.1312 (3)	0.1423 (3)	0.1482 (1)	4.54 (6)
N(6)	0.0240 (3)	0.0344 (4)	0.1955 (2)	4.34 (8)
	0.0145 (3)	0.0285 (3)	0.1966 (1)	4.47 (6)
C(1)	0.4363 (5)	0.4478 (5)	0.1205 (2)	5.4 (1)
	0.4352 (5)	0.4465 (4)	0.1188 (2)	5.64 (9)
C(2)	0.4475 (5)	0.3868 (6)	0.0660 (2)	5.8 (1)
	0.4501 (4)	0.3893 (4)	0.0650 (2)	5.80 (9)
C(3)	0.2396 (6)	0.2079 (7)	0.9901 (2)	7.1 (2)
	0.2391 (5)	0.2112 (5)	0.9893 (2)	7.3 (1)
C(4)	0.3788 (5)	0.1769 (7)	0.0085 (2)	6.4 (1)
	0.3786 (4)	0.1837 (5)	0.0056 (2)	6.7 (1)
C(5)	0.5093 (5)	0.0028 (6)	0.1086 (2)	5.7 (1)
	0.5026 (4)	0.0044 (5)	0.1082 (2)	6.1 (1)
C(6)	0.5433 (4)	0.1506 (6)	0.0916 (2)	5.9 (1)
	0.5414 (3)	0.1495 (4)	0.0890 (2)	5.9 (1)
C(7)	0.1401 (4)	0.0427 (5)	0.1823 (2)	4.5 (1)
	0.1288 (3)	0.0409 (4)	0.1845 (2)	4.88 (8)
C(8)	0.9483 (4)	0.1398 (6)	0.1673 (2)	5.6 (1)
	0.9381 (3)	0.1307 (4)	0.1680 (2)	5.68 (9)
C(9)	0.0239 (5)	0.2083 (5)	0.1383 (2)	5.7 (1)
	0.0105 (4)	0.2006 (4)	0.1381 (2)	5.7 (1)
C(10)	0.9831 (5)	0.9246 (6)	0.2301 (2)	5.5 (1)
	0.9758 (4)	0.9170 (4)	0.2315 (2)	5.67 (9)
C(11)	0.9145 (4)	0.7995 (5)	0.1976 (2)	4.5 (1)
	0.9103 (3)	0.7921 (4)	0.1991 (1)	4.61 (8)
C(12)	0.9732 (5)	0.7271 (5)	0.1617 (2)	5.3 (1)
	0.9677 (4)	0.7183 (4)	0.1633 (2)	5.50 (9)
C(13)	0.9080 (5)	0.6103 (6)	0.1312 (2)	5.8 (1)
	0.9079 (4)	0.6045 (4)	0.1339 (2)	6.5 (1)
C(14)	0.7918 (6)	0.5683 (6)	0.1415 (2)	6.6 (1)
	0.7890 (5)	0.5622 (4)	0.1406 (2)	7.5 (1)
C(15)	0.7352 (5)	0.6400 (7)	0.1775 (3)	7.0 (2)
	0.7321 (4)	0.6348 (5)	0.1762 (2)	7.7 (1)
C(16)	0.7941 (4)	0.7571 (6)	0.2057 (2)	5.2 (1)
	0.7908 (4)	0.7517 (5)	0.2043 (2)	5.82 (9)

* Multiplicities for atoms O(5)–O(8) are 0.67 and for atoms O(5P)–O(8P) are 0.33. These multiplicities were estimated from difference Fourier peak heights and were not refined.

† Atoms O(5P)–O(8P) for (1) were refined with isotropic displacement parameters.

Table 2. *Selected bond lengths (Å) and angles (°) with values for (2), where* Cu *becomes* Zn, *listed directly below those for* (1)

| Cu—N(1)	2.083 (3)	C(10)—C(11)	1.516 (5)
	2.073 (3)		1.501 (5)
Cu—N(2)	2.105 (3)	N(6)—C(10)	1.460 (4)
	2.081 (3)		1.468 (5)
Cu—N(3)	2.101 (3)	N(6)—C(7)	1.334 (4)
	2.084 (3)		1.326 (5)
Cu—N(4)	2.043 (3)	N(5)—C(7)	1.309 (4)
	2.255 (3)		1.313 (5)
Cu—N(5)	1.971 (3)	N(5)—C(9)	1.373 (4)
	2.054 (3)		1.374 (5)
N(1)—Cu—N(2)	118.1 (1)	N(3)—Cu—N(5)	96.9 (2)
	116.9 (1)		99.7 (1)
N(1)—Cu—N(3)	123.8 (1)	N(4)—Cu—N(5)	178.4 (2)
	120.3 (1)		178.3 (1)
N(1)—Cu—N(4)	84.1 (2)	N(6)—C(10)—C(11)	111.9 (3)
	80.9 (1)		111.8 (3)
N(1)—Cu—N(5)	96.3 (2)	C(10)—C(11)—C(16)	119.4 (3)
	100.0 (1)		120.1 (4)
N(2)—Cu—N(3)	114.8 (1)	C(10)—C(11)—C(12)	120.1 (4)
	115.3 (1)		121.0 (4)
N(2)—Cu—N(4)	83.8 (1)	C(8)—N(6)—C(10)	126.6 (3)
	80.5 (1)		126.4 (3)
N(2)—Cu—N(5)	94.8 (1)	C(7)—N(6)—C(10)	125.8 (3)
	97.8 (1)		125.9 (3)
N(3)—Cu—N(4)	84.1 (2)	C(7)—N(6)—C(8)	107.4 (3)
	81.0 (1)		107.6 (3)
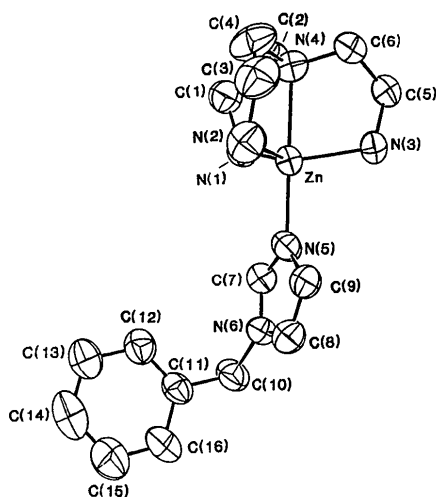
Fig. 1. View (*ORTEP*; Johnson, 1976) of the cation in (2) showing the atom-numbering scheme. Thermal ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. The cation in the copper complex (1) has the same numbering scheme for the C and N atoms.

2.043 (3) Å and the *M*—N(axial imidazole) distance, Cu—N(5), is 1.971 (3) Å. In (2), Zn—N(4) is 2.255 (3) and Zn—N(5) is 2.054 (3) Å. As expected for Cu^{II} having a d_{z^2} ground state, a Jahn–Teller compression along the axial bonds is observed relative to Zn^{II} with its filled d_{z^2} orbital. For the *M*—N(tertiary amine) bond, a shortening of 0.212 Å is observed while for the *M*—N(1-benzylimidazole) bond, the corresponding shortening is 0.083 Å. Axial compression of this sort has been observed previously in [Cu(3)(NCS)](SCN), where Cu—N(4) is

2.033 (4) and Cu—N(5) is 1.959 (5) Å (Jain & Lingafelter, 1967) relative to [Zn(3)(NCS)](SCN) where Zn—N(4) is 2.292 (4) and Zn—N(5) is 2.043 (5) Å (Andreetti, Jain & Lingafelter, 1969). The axial Zn—N distances in (2) may be shortened somewhat owing to Cu^{II} doping. While the level of doping suggests that this shortening is modest, the true distances will only be known from the structure of the neat Zn(3) complex.

A six-coordinate Co complex containing (4) as an axially-bound ligand (Gall & Schaefer, 1976) has been reported. Within 3σ, all angles are the same as those of ligand (4) in compounds (1) and (2). Similarly, within 5σ, all bond lengths except one are equivalent. The exception is C(10)—C(11), which is 1.516 (5) [1.501 (5) Å] for (1) [(2)] while in the Co complex, C(10)—C(11) is 1.469 (5) Å [a difference of 0.047 (0.032 Å)].

One of the perchlorate anions in each structure is disordered. A model for the disorder was developed for both (1) and (2) based on electron density surrounding Cl(2) determined from difference Fourier maps. In each case, partially occupied oxygen sites in a 2/3 to 1/3 ratio were used. The 1/3 occupied sites O(5*P*)–O(8*P*) in (1) were left isotropic and fixed.

The magnetically-dilute polycrystalline complex (2) exhibits a reversed electron paramagnetic resonance (EPR) spectrum expected for a Cu^{II} dopant having approximately trigonal-bipyramidal coordination geometry (Duggan, Ray, Hathaway, Tomlinson, Brint & Pelin, 1980). Simulation studies using the program *QPOWA* (Belford, 1987) show these spectra are consistent with a single Cu^{II} dopant site having the parameters $g_\perp = 2.145$, $A_\perp = 102 \times 10^{-4}\,cm^{-1}$ and $g_\| = 1.969$, $A_\| = 65 \times 10^{-4}\,cm^{-1}$. These results suggest that the Cu^{II} dopant structure approximates that found for the neat Cu^{II} complex.

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Redetermination of the Structure of Phenanthridine

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Abstract. $C_{13}H_9N$, $M_r = 179.22$, orthorhombic, $P2_12_12_1$, $a = 4.872$ (2), $b = 11.526$ (4), $c = 15.811$ (4) Å, $V = 887.7$ (5) Å^3, $Z = 4$, $D_x = 1.341$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.07$ mm^{-1}, $F(000) = 376$, $T = 110$ K, final $R = 0.045$ for 1713 observed reflections. Phenanthridine was found to be planar in the solid state with a mean deviation of 0.002 Å from the mean plane of the C and N atoms. The geometries of the benzene fragments in the molecule are rather similar and in the central pyridine moiety the C—N bond lengths are typical of C—N single and double bonds. In the crystal the molecules are stacked according to the screw axes. Comparison with a previous structure determination of phenanthridine reveals great differences in bond lengths and angles.

Introduction. Polycyclic compounds can be divided into four groups depending on the combination of the individual rings (Rademacher, 1987). The rings can be connected by a single bond as in bicyclopropyl, by a common C atom as in spiropentane, by bridging as in bicyclo[1.1.1]pentane or by a common bond (annelation) as in bicyclobutane. The annelation of aromatic and/or heteroaromatic rings leads to polycyclic aromatic hydrocarbons (PAHs) or polycyclic heteroaromatic hydrocarbons (PHHs). In the course of our studies on polycyclic heteroaromatic compounds we have reinvestigated the structure of phenanthridine by X-ray diffraction.

This compound can be regarded as the combination of two benzene molecules with a pyridine ring. The main questions in this structural investigation

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^4$) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	3640 (5)	10228 (2)	4918 (1)	250 (6)
C(2)	5062 (5)	9192 (2)	4925 (1)	263 (6)
C(3)	4537 (5)	8381 (2)	5567 (1)	257 (6)
C(4)	2625 (5)	8596 (2)	6186 (1)	234 (6)
C(5)	−1551 (5)	9233 (2)	7522 (1)	245 (6)
C(6)	−3569 (5)	9553 (2)	8085 (1)	276 (6)
C(7)	−4981 (5)	10603 (2)	7983 (1)	277 (6)
C(8)	−4321 (5)	11328 (2)	7325 (1)	262 (6)
N(9)	−1674 (4)	11816 (2)	6100 (1)	265 (5)
C(10)	183 (5)	11534 (2)	5550 (1)	243 (6)
C(11)	1680 (4)	10465 (2)	5548 (1)	203 (5)
C(12)	1158 (4)	9648 (2)	6197 (1)	193 (5)
C(13)	−870 (4)	9949 (2)	6828 (1)	185 (5)
C(14)	−2255 (4)	11025 (2)	6744 (1)	229 (6)

are how the geometries of the molecular benzene and pyridine subunits are affected by the annelation and how the geometry – relative to phenanthrene (Kay, Okaya & Cox, 1971) – is changed by the introduction of the heteroatom.

Experimental. Phenanthridine was obtained from Aldrich and crystals were grown from a solution in carbon tetrachloride. Crystal size: $0.2 \times 0.2 \times 0.3$ mm, Nicolet *R3m/V* four-circle diffractometer, Wyckoff scan, scan width 1.0°, lattice parameters determined with 49 reflections ($15 \le 2\theta \le 25°$), no absorption correction applied, maximum value of $(\sin\theta)/\lambda = 0.70$ Å^{-1}, *hkl* range $-6 \le h \le 24$, $0 \le k \le 5$, $0 \le l \le 22$, two standard reflections (*hkl* = 012 and 1̄12) with no significant variation during the experiment, 2220 measured intensities, $R_{int} = 0.048$, 2065

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