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Redetermination of the Structure of Triaqua(2,2′-bipyridine)nitratocopper(II) Nitrate

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Abstract. $[Cu(C_{10}H_8N_2)(H_2O)_3(NO_3)]NO_3$, $M_r = 397.8$, triclinic, $P\bar{1}$, $a = 7.702\,(1)$, $b = 9.343\,(1)$, $c = 11.275\,(1)$ Å, $\alpha = 99.24\,(1)$, $\beta = 103.36\,(1)$, $\gamma = 102.08\,(1)°$, $V = 753.1$ Å^3, $Z = 2$, $D_m = 1.75$, $D_x = 1.754$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 15.14$ cm^{-1}, $F(000) = 406$, $T = 294$ K, $R = 0.046$, $wR = 0.052$ for 2493 unique observed reflections [$F > 3\sigma(F)$]. The general features of the structure are the same as those reported earlier [Nakai, Ooi & Kuroya (1977). *Bull. Chem. Soc. Jpn*, **50**, 531–532]. The Cu—O bonds of 1.949 (2) and 1.963 (2) Å in the equatorial plane are not equal [1.99 (1) Å] as observed in the previous work. The structure is stabilized by a network of hydrogen bonds and van der Waals interactions. The bipyridine rings are involved in stacking interactions with centrosymmetrically related entities.

Experimental. Crystals of the title complex were isolated from a 1:1:1 molar mixture of $Cu(NO_3)_2.3H_2O$, bpy and salicylaldehyde, by slow evaporation. Density was determined by flotation in a mixture of $CHCl_3$ and $CHBr_3$. A blue rectangular-prismatic crystal of size 0.35 × 0.25 × 0.10 mm was used in the data collection on a CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. The unit-cell parameters were based on 20 centred reflections within the θ range 9.4–18.0°. Intensity data were collected for 2880 reflections with $\omega/2\theta$ scan, $-9 \le h \le 9$, $-11 \le k \le 11$, $0 \le l \le 13$, $\sin\theta/\lambda_{\max} = 0.59$ Å^{-1}. Variations in three standard reflections (411, 141 and 123) were less than 6%. The intensities were scaled by the use of these standards and averaged to 2493 unique reflections [$F > 3\sigma(F)$], $R_{\rm int} = 0.027$. They were corrected for Lp effects but not for absorption. The structure was solved by the heavy-atom method and refined on F by full-matrix least squares (Sheldrick, 1976). All the H-atom positions were located in difference maps and refined in the last few cycles. 285 parameters were refined. Final discrepancy indices are $R = 0.046$, $wR = 0.052$ and $S = 0.90$ with $w = 1.000/[\sigma^2(F) + 0.004972F^2]$, where the $\sigma(F)$ was derived from counting statistics. In the final cycle of refinement $(\Delta/\sigma)_{\max} = 0.04$ and the max. and min. heights in the final $\Delta\rho$ maps are 0.57 and −1.18 e Å^{-3}. Scattering factors used for H, C, N and O atoms were as available in *SHELX* and for Cu from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 99). The final atomic coordinates and equivalent isotropic temperature factors for non-H atoms are listed in Table 1 and selected bond lengths and angles in Table 2.* An *ORTEP* (Johnson, 1976) drawing of the molecule with the numbering scheme is shown in Fig. 1.

Related literature. The crystal structure of the compound has been determined previously (Nakai, Ooi & Kuroya, 1977), using multiple-film techniques ($R = 0.12$). In contrast to the earlier work, in the present study the coordinated waters and nitrate group show normal temperature factors. One of the O atoms of the uncoordinated nitrate group shows disorder. The coordinated nitrate and bipyridyl groups make dihedral angles of 72 and 7.8°, respectively, with the equatorial plane.

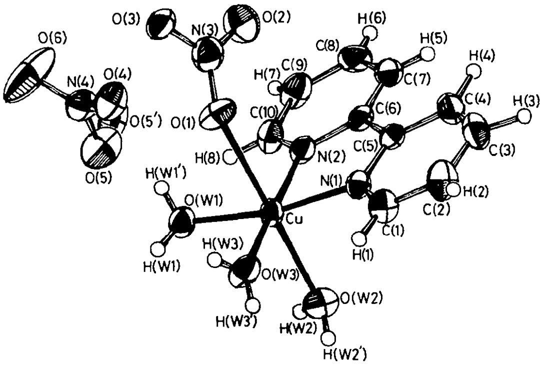

Fig. 1. *ORTEP* (Johnson, 1976) drawing of $[Cu(bpy)(H_2O)_3(NO_3)]NO_3$ with the labelling scheme used. Thermal ellipsoids scaled to 50% probability.

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* Lists of structure factors, bond lengths and angles, anisotropic thermal parameters, H-atom coordinates, distances and angles involving H atoms, least-squares planes, some torsion angles, tables of possible hydrogen bonds and intermolecular contacts (< 3.50 Å) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54103 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates* (× 10^5 *for* Cu *and* × 10^4 *for others*) *and equivalent isotropic temperature factors* (× 10^4), *with their e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}(Å^2)
Cu	11686 (4)	28136 (3)	30514 (2)	282 (2)
N(1)	955 (3)	3009 (3)	1288 (2)	299 (7)
C(1)	2018 (4)	4081 (3)	908 (3)	389 (10)
C(2)	1676 (5)	4165 (4)	−335 (3)	477 (12)
C(3)	230 (5)	3123 (4)	−1201 (3)	473 (12)
C(4)	−857 (4)	2015 (3)	−819 (3)	391 (10)
C(5)	−450 (3)	1988 (3)	433 (2)	298 (8)
C(6)	−1485 (3)	824 (3)	954 (2)	298 (9)
C(7)	−2916 (4)	−378 (3)	233 (3)	378 (9)
C(8)	−3794 (4)	−1370 (4)	822 (3)	452 (10)
C(9)	−3201 (5)	−1183 (4)	2092 (3)	461 (11)
C(10)	−1748 (4)	31 (3)	2769 (3)	367 (9)
N(2)	−907 (3)	1021 (3)	2205 (2)	301 (7)
O(*W*1)	904 (3)	2672 (3)	4722 (2)	383 (7)
O(*W*2)	3311 (3)	1398 (2)	3028 (2)	417 (8)
O(*W*3)	3161 (3)	4630 (3)	3804 (2)	447 (8)
N(3)	−2765 (3)	4341 (3)	2809 (2)	482 (9)
O(1)	−1068 (3)	4605 (3)	3066 (2)	479 (9)
O(2)	−3765 (4)	3189 (3)	2086 (3)	646 (10)
O(3)	−3477 (3)	5291 (3)	3301 (2)	472 (8)
N(4)	−2951 (3)	1825 (3)	5680 (2)	398 (8)
O(4)	−2576 (3)	2769 (2)	5039 (2)	478 (7)
O(5)*	−2070 (7)	841 (8)	5754 (6)	636 (23)
O(5′)*	−2814 (29)	584 (26)	5372 (20)	545 (76)
O(6)	−3934 (5)	1995 (4)	6350 (4)	1076 (18)

* Site occupancies 0·8 for O(5) and 0·2 for O(5′).

Table 2. *Selected bond lengths* (Å) *and angles* (°) *involving non-*H *atoms, with their e.s.d.'s in parentheses*

Cu—N(1)	1·998 (2)	Cu—N(2)	1·983 (3)
Cu—O(*W*1)	1·963 (2)	Cu—O(*W*2)	2·322 (2)
Cu—O(*W*3)	1·949 (2)	Cu—O(1)	2·642 (2)
N(1)—C(1)	1·347 (4)	N(3)—O(1)	1·233 (3)
N(1)—C(5)	1·342 (4)	N(3)—O(2)	1·224 (4)
C(6)—N(2)	1·346 (3)	N(3)—O(3)	1·258 (3)
C(10)—N(2)	1·345 (4)	N(4)—O(4)	1·256 (3)
N(4)—O(5)	1·255 (7)	N(4)—O(5′)	1·189 (24)
N(4)—O(6)	1·202 (5)	O(5)—O(5′)	0·606 (24)
N(1)—Cu—N(2)	81·6 (1)	N(1)—Cu—O(*W*1)	169·2 (1)
N(1)—Cu—O(*W*2)	92·9 (1)	N(1)—Cu—O(*W*3)	95·5 (1)
N(1)—Cu—O(1)	85·4 (1)	N(2)—Cu—O(*W*1)	93·4 (1)
N(2)—Cu—O(*W*2)	91·5 (1)	N(2)—Cu—O(*W*3)	176·7 (1)
N(2)—Cu—O(1)	92·4 (1)	Cu—N(2)—C(6)	114·9 (2)
O(*W*1)—Cu—O(*W*2)	96·8 (1)	Cu—N(2)—C(10)	125·9 (2)
O(*W*1)—Cu—O(*W*3)	89·2 (1)	O(*W*1)—Cu—O(1)	85·3 (1)
O(*W*2)—Cu—O(*W*3)	90·3 (1)	O(*W*2)—Cu—O(1)	175·5 (1)
O(*W*3)—Cu—O(1)	85·7 (1)	Cu—N(1)—C(1)	126·3 (2)
Cu—N(1)—C(5)	114·7 (2)	Cu—O(1)—N(3)	131·8 (2)
O(1)—N(3)—O(2)	121·8 (3)	O(4)—N(4)—O(6)	120·2 (3)
O(1)—N(3)—O(3)	118·7 (2)	O(4)—N(4)—O(5)	118·7 (4)
O(2)—N(3)—O(3)	119·5 (3)	O(5)—N(4)—O(6)	120·2 (4)

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2-(Chlorodimethylstannyl)-4-methoxy-2′,4′-dinitroazobenzene

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Abstract. $C_{15}H_{15}ClN_4O_5Sn$, M_r = 485·45, monoclinic, $P2_1/c$, a = 9·349 (2), b = 15·330 (3), c = 13·562 (3) Å, β = 106·14 (2)°, V = 1867·1 (7) Å^3, Z = 4, D_x = 1·727 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 1·55 mm^{-1}, F(000) = 960, T = 291 (1) K, final R = 0·031 for 3136 unique observed [$F \geq 4\cdot0\sigma(F)$] diffractometer data. Besides the three C atoms [Sn—C(mean) 2·127 (4) Å] and the Cl atom [Sn—Cl 2·433 (1) Å] bound to Sn there is a short intramolecular Sn···N contact [2·674 (2) Å] and a distorted trigonal bipyramid is formed around Sn with Cl and N in the apical [Cl—Sn—N 161·76 (6)°] and the C atoms in the equatorial [C—Sn—C 118·3 (2), 114·4 (1), 120·8 (1)°] positions. The structure consists of discrete molecular units.

Experimental. The crystals have been obtained from acetone/pentane 1:1, m.p. 434 K. A crystal of size 0·44 × 0·42 × 0·16 mm was used. Its quality was checked with optical polarizing microscopy. The crystal was mounted on a glass fibre. Intensity data were collected with $\omega/2\theta$ scans, variable scan speed 1·5–10·2° min^{-1} in θ, scan width 1·2° + dispersion, on a Nicolet *R*3*m*/*V* diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters determined from symmetry-constrained least-squares fit of 36 reflections with $2\theta_{max}$ = 38·2°. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (400, 040, 002, $\bar{4}00$, $0\bar{4}0$, $00\bar{2}$) recorded every 300 reflections, only random deviations

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