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Bis(3,5-dimethyl-1-phenylpyrazole)dinitratocopper(II)

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Abstract. $Cu(C_{11}H_{12}N_2)_2(NO_3)_2$, $C_{22}H_{24}CuN_6O_6$, orthorhombic, $P2_12_12_1$, a = 10·613 (2), b = 13·188 (3), c = 17·715 (3) Å, V = 2479 Å^3, Z = 4, D_m = 1·43 (flotation), D_x = 1·42 Mg m^{-3}. The final R was 0·064 for 1750 observations. The Cu^{2+} is coordinated to the nitrate ions and the organic ligands in a near square-planar *cis* arrangement.

Introduction. Blue prismatic crystals of the title compound were prepared by Dr Cristo B. Melios *et al.* (Institute of Chemistry of Araraquara, UNESP). Chemical analysis showed a composition ratio of 1:2:2 for the Cu^{II} ions, nitrate and organic ligands (Melios, 1978). The structure determination was undertaken to study the coordination between the Cu^{II} and NO_3^- ions. Cell dimensions were refined by a least-squares fit to the setting angles of 25 reflexions on a CAD-4 automatic diffractometer. Intensity measurements were carried out up to $\theta = 30°$, using the ω–2θ scan mode, with graphite-monochromated Mo $K\alpha$ radiation and a rectilinear crystal, 0·4 × 0·3 × 0·6 mm. Lorentz and polarization corrections were applied, but no absorption correction was made [μ(Mo $K\alpha$) = 0·97 mm^{-1}]. Of the 2478 measured unique reflexions, 1750 were considered observed [$F^2 \geq 2\sigma(F^2)$] and retained for use in the structure analysis* and refinement.

The structure was solved by the heavy-atom method. It was refined by full-matrix least-squares calculations by minimization of $\sum \omega(k|F_o| - |F_c|)^2$, where $\omega = [\sigma(F_o)^2 + (0{\cdot}05F_o)^2]^{-1}$ for observed and $\omega = 0$ for unobserved reflexions, until all the atomic parameter shifts were smaller than each standard deviation. The final unweighted R factor, omitting unobserved reflexions, was 0·064 and including them 0·070. Aniso-

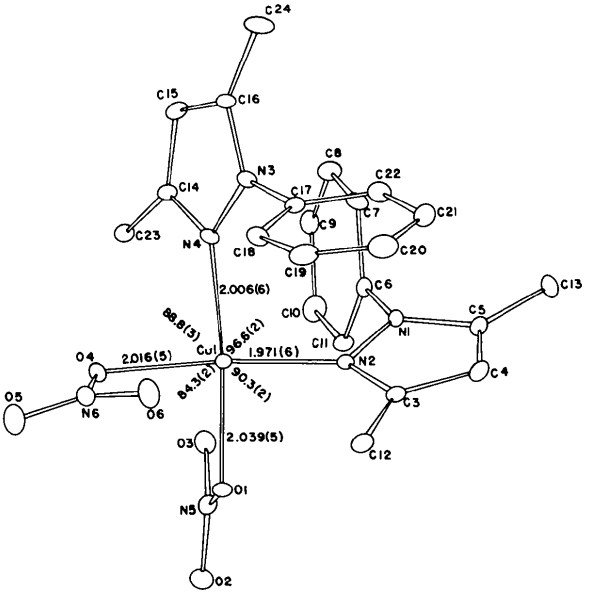

Fig. 1. Diagram of the molecule showing bond lengths (Å) and angles (°) around the copper ion and atom numbering.

* Lists of structure factors, anisotropic thermal parameters, hydrogen atom parameters and bond distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34236 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates* ($\times 10^5$) *for the copper atom and* ($\times 10^4$) *for non-hydrogen atoms with e.s.d.'s in parentheses*

	x	*y*	*z*
Cu(1)	64326 (9)	1199 (8)	23288 (6)
O(1)	5487 (6)	746 (5)	1442 (3)
O(2)	3535 (7)	812 (6)	1124 (4)
O(3)	4094 (6)	119 (6)	2190 (3)
O(4)	6320 (6)	−1170 (5)	1722 (3)
O(5)	7165 (8)	−1670 (7)	688 (5)
O(6)	7894 (7)	−316 (6)	1291 (5)
N(1)	6148 (7)	1684 (5)	3540 (4)
N(2)	6571 (6)	1463 (5)	2810 (4)
N(3)	8414 (7)	−474 (6)	3501 (4)
N(4)	7252 (6)	−671 (6)	3163 (4)
N(5)	4325 (7)	546 (6)	1582 (5)
N(6)	7147 (8)	−1048 (7)	1213 (4)
C(3)	7193 (8)	2267 (7)	2559 (6)
C(4)	7182 (10)	3011 (7)	3131 (7)
C(5)	6530 (10)	2664 (7)	3723 (6)
C(6)	5287 (9)	1061 (7)	3939 (5)
C(7)	5689 (10)	525 (9)	4566 (6)
C(8)	4847 (9)	−50 (10)	4970 (6)
C(9)	3658 (10)	−108 (9)	4764 (5)
C(10)	3218 (10)	401 (10)	4123 (7)
C(11)	4026 (9)	1013 (8)	3710 (5)
C(12)	7806 (11)	2275 (8)	1792 (6)
C(13)	6148 (12)	3121 (8)	4488 (6)
C(14)	6736 (9)	−1452 (7)	3527 (5)
C(15)	7552 (10)	−1743 (8)	4070 (6)
C(16)	8625 (10)	−1135 (8)	4057 (5)
C(17)	9262 (8)	303 (7)	3236 (5)
C(18)	9935 (9)	158 (9)	2590 (6)
C(19)	10793 (10)	877 (10)	2379 (7)
C(20)	10965 (10)	1701 (11)	2751 (7)
C(21)	10295 (11)	1884 (9)	3399 (8)
C(22)	9443 (9)	1161 (9)	3664 (6)
C(23)	5455 (10)	−1817 (8)	3337 (6)
C(24)	9785 (10)	−1095 (12)	4555 (7)

tropic temperature factors were assigned to all non-hydrogen atoms. Complex neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1974). Final positional parameters for the hydrogen atoms were calculated assuming a C—H bond length and trigonal angle, with the isotropic temperature factors assumed to be 5·0 Å^2. Table 1 shows the heavy-atom coordinates.

Discussion. The molecule has a pseudo-twofold axis through the copper ion which is parallel to the [324] axis.

The pyrazolic rings are *cis* coordinated to the copper ion through atoms N(2) and N(4).

The nitrate groups are planar within the standard deviations (Addison, Logan & Wallwork, 1971). They are coordinated to the copper ion through O(1) and O(4). Each nitrate has another oxygen close to the copper ion [Cu(1)—O(3) 2·494 (5) and Cu(1)—O(6) 2·473 (6) Å with O(3)—Cu(1)—O(6) 123·4 (2)°] completing a very distorted octahedron as previously reported for the two structural forms of bis(antipyrine)dinitratocopper(II) (Brassy, Renaud, Delettré & Mornon, 1974; Brassy, Mornon, Delettré & Lepicard, 1974).

The coordination around the copper ion is shown in Fig. 1. The deviations of the atoms Cu(1), N(2), N(4), O(1), O(4) from the average plane of equation $0{\cdot}8834x + 0{\cdot}1725y - 0{\cdot}4357z - 4{\cdot}2644 = 0$, referred to the conventional orthogonalized coordinates, are less than 0·1 Å.

The two rings of each organic ligand are planar within the standard deviation. All other distances and angles agree within 3σ with the values found in tris-(3,5-dimethyl-1-phenylpyrazole)silver(I) nitrate (Francisco, Mascarenhas & Lechat, 1979).

All computer calculations were performed using the *Enraf–Nonius Structure Determination Package*. The figure was drawn with the *ORTEP* program (Johnson, 1965).

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