

## Bis(2,2'-bipyridine)isocyanatocopper(II) Perchlorate

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**Abstract.**  $[\text{Cu}(\text{C}_{10}\text{H}_8\text{N}_2)_2(\text{NCO})]\text{ClO}_4$ ,  $M_r = 517.39$ , triclinic,  $P\bar{1}$ ,  $a = 10.20$  (2),  $b = 13.55$  (4),  $c = 7.98$  (1) Å,  $\alpha = 90.7$  (2),  $\beta = 99.7$  (2),  $\gamma = 105.1$  (2)°,  $V = 1047$  (4) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.640$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 1.22$  mm<sup>-1</sup>,  $F(000) = 526$ ,  $T = 293$  K,  $R = 0.056$ , 1970 unique reflexions [ $I \geq 3\sigma(I)$ ]. The Cu environment is a distorted trigonal bipyramid with each of the bipyridine ligands forming equatorial [2.095 (8), 2.111 (7) Å] and somewhat shorter axial [2.007 (9), 1.981 (8) Å] Cu—N bonds. However, at 1.961 (8) Å, the equatorial isocyanide ligand forms the shortest Cu—N bond.

**Experimental.** The compound was obtained as a side product during the synthesis of Cu complexes of 2(1*H*)-pyridone by use of an electrochemical method proposed by Tuck (Habeeb, Tuck & Walters, 1978). The electrochemical oxidation of a Cu anode in an acetonitrile solution (70 ml) containing 2,2'-bipyridine (0.12 g), 2(1*H*)-pyridone (0.15 g) and tetramethylammonium perchlorate (*ca* 10 mg) led to the formation of a green solid. On standing for several days at room temperature an additional green solid was recovered from the mother liquor. This was recrystallized from methanol to give crystals suitable for X-ray investigation.

Crystal dimensions  $0.3 \times 0.3 \times 0.2$  mm; Rigaku AFC-6S diffractometer, graphite-monochromated Mo  $K\alpha$  radiation; unit-cell dimensions from setting angles of 25 accurately centred reflexions ( $12.1 \leq 2\theta \leq 16.1^\circ$ );  $\omega-2\theta$  scan mode,  $\omega$ -scan width ( $0.84 + 0.30 \tan\theta$ )° and scan speed of  $4^\circ \text{ min}^{-1}$  with up to two additional scans of weak reflexions [ $I < 10\sigma(I)$ ];  $0 \leq h \leq 12$ ,  $-15 \leq k \leq 15$ ,  $-9 \leq l \leq 9$ ,  $0 \leq \theta \leq 25^\circ$ ; 4459 reflexions measured, 3698 unique, 1970 observed [ $I \geq 3\sigma(I)$ ]; intensity standards (241, 241, 250) measured every 150 reflexions showed no drift; Lp and absorption (maximum, minimum transmission 0.95, 1.0) corrections applied; MITHRIL (Gilmore, 1984) used to solve the phase problem; all non-H atoms found in Fourier map, H atoms from

Table 1. Positional parameters and equivalent isotropic vibrational parameters (Å<sup>2</sup>)

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$B_{\text{eq}}$
Cu(1)	0.1252 (1)	0.76484 (8)	0.0641 (1)	3.74 (4)
O(1)	0.1746 (7)	0.8143 (6)	0.5740 (9)	8.0 (4)
N(1)	0.2952 (6)	0.8817 (5)	0.1287 (8)	3.6 (3)
N(2)	0.0699 (6)	0.8771 (5)	-0.0893 (7)	3.6 (3)
N(3)	-0.0460 (6)	0.6566 (5)	-0.0238 (8)	3.9 (3)
N(4)	0.2002 (6)	0.6751 (5)	-0.0954 (8)	3.7 (3)
N(5)	0.1143 (7)	0.7331 (6)	0.3015 (9)	5.0 (3)
C(1)	0.1447 (8)	0.7722 (7)	0.4312 (13)	4.6 (4)
C(2)	0.4035 (8)	0.8759 (7)	0.2437 (10)	4.3 (4)
C(3)	0.5106 (9)	0.9615 (8)	0.3010 (12)	4.8 (4)
C(4)	0.5030 (9)	1.0540 (8)	0.2380 (12)	5.0 (4)
C(5)	0.3936 (9)	1.0597 (7)	0.1192 (11)	4.5 (4)
C(6)	0.2887 (7)	0.9712 (6)	0.0622 (10)	3.5 (3)
C(7)	0.1671 (7)	0.9678 (7)	-0.0673 (10)	3.7 (3)
C(8)	0.1500 (10)	1.0492 (8)	-0.1645 (12)	4.8 (4)
C(9)	0.0337 (11)	1.0400 (8)	-0.2794 (12)	4.9 (4)
C(10)	-0.0660 (10)	0.9489 (8)	-0.3025 (12)	4.8 (4)
C(11)	-0.0438 (9)	0.8697 (7)	-0.2052 (11)	4.2 (4)
C(12)	-0.1684 (8)	0.6520 (7)	0.0215 (13)	4.7 (4)
C(13)	-0.2855 (9)	0.5806 (8)	-0.0507 (14)	5.4 (5)
C(14)	-0.2797 (11)	0.5114 (9)	-0.1694 (15)	6.4 (5)
C(15)	-0.1539 (11)	0.5122 (7)	-0.2138 (12)	5.4 (4)
C(16)	-0.0396 (8)	0.5873 (6)	-0.1411 (10)	4.0 (3)
C(17)	0.1003 (8)	0.5965 (6)	-0.1806 (10)	3.8 (3)
C(18)	0.1326 (11)	0.5337 (7)	-0.2980 (11)	5.0 (4)
C(19)	0.2648 (12)	0.5513 (9)	-0.3265 (12)	5.6 (5)
C(20)	0.3652 (12)	0.6310 (9)	-0.2397 (13)	5.8 (5)
C(21)	0.3277 (9)	0.6885 (7)	-0.1215 (12)	4.7 (4)
Cl(1)	0.3534 (2)	1.2898 (2)	0.5808 (3)	5.1 (1)
O(2)	0.3748 (7)	1.2116 (5)	0.4797 (9)	8.6 (4)
O(3)	0.4303 (8)	1.3854 (5)	0.5415 (11)	9.5 (4)
O(4)	0.3985 (8)	1.2764 (6)	0.7579 (9)	9.0 (4)
O(5)	0.2131 (6)	1.2885 (6)	0.5563 (10)	9.8 (4)

$\Delta F$  synthesis; full-matrix least squares based on  $F$  using TEXSAN crystallographic software (Molecular Structure Corporation, 1985);  $S = 2.0$ , number of variables = 362, final  $R = 0.056$   $\{wR = 0.061$ ,  $w = 1/[\sigma^2(F_o) + (0.03F_o)^2]$ ; anisotropic thermal parameters for heavier atoms, isotropic for H atoms; maximum fluctuation in final  $\Delta F$  map in range  $-0.59-0.66 \text{ e } \text{Å}^{-3}$ ; maximum  $\Delta/\sigma = 0.004$ . Scattering factors were taken from Cromer & Waber (1974). Computation was carried out on a Digital VAX station 3520. A literature survey was performed via the Cambridge Structural Database using the Crystal Structure Search and Retrieval interactive system (CSSR, 1984). Fractional atomic coordi-

Table 2. *Intramolecular distances (Å) and angles (°) involving the non-H atoms*

Cu(1)—N(1)	2.007 (9)	Cu(1)—N(5)	1.961 (8)
Cu(1)—N(2)	2.095 (8)	O(1)—C(1)	1.22 (1)
Cu(1)—N(3)	1.981 (8)	N(5)—C(1)	1.11 (1)
Cu(1)—N(4)	2.111 (7)		
N(1)—Cu(1)—N(2)	79.5 (3)	N(2)—Cu(1)—N(4)	105.3 (3)
N(1)—Cu(1)—N(3)	173.7 (3)	N(2)—Cu(1)—N(5)	133.2 (3)
N(1)—Cu(1)—N(4)	99.3 (3)	N(3)—Cu(1)—N(4)	79.5 (3)
N(1)—Cu(1)—N(5)	93.5 (3)	N(3)—Cu(1)—N(5)	92.4 (3)
N(2)—Cu(1)—N(3)	94.8 (3)	N(4)—Cu(1)—N(5)	121.5 (3)

nates and vibrational parameters for non-H atoms are presented in Table 1 and selected bond lengths and angles in Table 2.\* The title molecule, including atomic labelling, is displayed in Fig. 1.

**Related literature.** The square-pyramidal distorted trigonal bipyramidal geometry seen here is very similar to that in bis(2,2'-bipyridyl)thiocyanatocopper(II) tetrafluoroborate (Tyagi & Hathaway, 1981). The relatively short equatorial thiocyanate Cu—N bond is interpreted as resulting from an angular distortion away from a trigonal bipyramid rather than to the potentially  $\pi$ -bonding role of the thiocyanate ligand.

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\* Lists of structure factors, H-atom parameters, bond lengths and angles and anisotropic vibrational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54933 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0480]

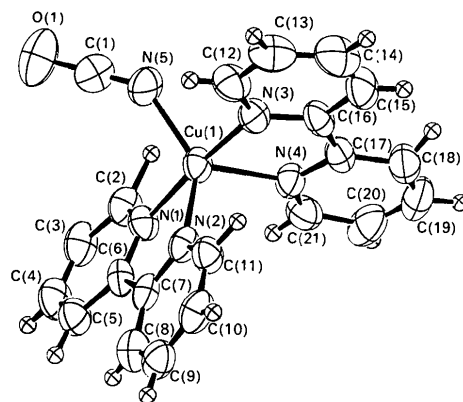


Fig. 1. The title molecule, drawn using ORTEPII (Johnson, 1976).

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## Structure of L-Phenylalanine L-Phenylalaninium Formate

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**Abstract.**  $C_9H_{11}NO_2 \cdot C_9H_{12}NO_2^+ \cdot CHO_2^-$ ,  $M_r = 376.41$ , monoclinic,  $P2_1$ ,  $a = 11.507$  (6),  $b = 5.638$  (3),  $c = 14.610$  (5) Å,  $\beta = 100.65$  (4)°,  $V = 932$  (1) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.342$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 0.94$  cm<sup>-1</sup>,  $F(000) = 400$ ,  $T = 295$  K, final  $R = 0.047$  for 2693 observed reflections. The phenylalanine zwitterion and the phenylalanine cation form a

Speakman-salt-type hydrogen bond [ $O \cdots O = 2.496$  (3) Å]. Aromatic side chains constitute a thick hydrophobic layer with edge-to-face interactions between the phenyl rings.

**Introduction.** Structural studies of peptides are often hampered by difficulties in obtaining crystals large enough for X-ray diffraction. Based on our success in controlling crystal morphology and size of small molecular crystals using cocrystallization techniques (Etter, 1988), we have now turned our attention to

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