Redetermination of bis(2,2'-bipyridine-$\kappa^2 N,N'$)-iodocopper(II) iodide

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The crystal structure of the title complex, [CuI(C$_{20}$H$_{16}$N$_4$)]I, was first determined and reported by Barclay *et al.* [*J. Chem. Soc.* (1963), pp. 5691–5699]. We present here a redetermination, of greatly improved precision, in which the H-atom positions have been located.

Comment

The structure of the title compound, (I), consists of Cu^{II} complex cations and iodide anions. The Cu^{II} atom has a compressed trigonal–bipyramidal coordination geometry. The Cu—N bonds [1.980 (3) and 1.981 (3) Å] in the axial directions are significantly shorter than the Cu—N bonds [2.092 (3) and 2.089 (3) Å] in the equatorial plane. The C···I2 distance of 3.833 (5) Å and the C5—H5—I2 angle of 144° suggest weak C—H···I hydrogen bonding between the iodide ion and the pyridine ring. Separations of 3.308 (15) Å and 3.377 (5) Å between parallel bipyridine planes indicate significant π–π stacking. A rather short C···C contact of 3.190 (7) Å is observed between neighboring molecules in the crystal.

(I)
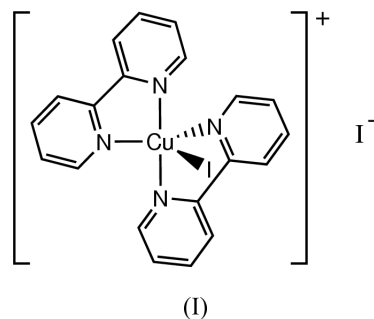
Experimental

A solution of dimethylformamide (10 ml) and methanol (5 ml) containing CuI (0.19 g, 1 mmol) and 2,2'-bipyridine (0.16 g, 1 mmol) was refluxed, under atmospheric pressure, for 2 h. After two weeks, dark-brown single crystals of the title compound, (I), were obtained from the green filtrate.

Crystal data

[CuI(C$_{20}$H$_{16}$N$_4$)]I	$Z = 2$
$M_r = 629.71$	$D_x = 2.015$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.5450$ (16) Å	Cell parameters from 18
$b = 14.299$ (3) Å	reflections
$c = 7.4229$ (6) Å	$\theta = 4.2$–11.0°
$\alpha = 95.445$ (10)°	$\mu = 4.04$ mm^{-1}
$\beta = 98.780$ (9)°	$T = 298$ (2) K
$\gamma = 108.300$ (14)°	Prism, dark brown
$V = 1038.0$ (3) Å^3	0.20 × 0.16 × 0.10 mm

metal-organic papers

Data collection

Rigaku AFC-7S diffractometer	$R_{int} = 0.013$
$\omega/2\theta$ scans	$\theta_{max} = 26.0°$
Absorption correction: ψ scan	$h = 0 \rightarrow 12$
(North *et al.*, 1968)	$k = -17 \rightarrow 16$
$T_{min} = 0.446$, $T_{max} = 0.668$	$l = -9 \rightarrow 9$
4307 measured reflections	3 standard reflections
4074 independent reflections	every 150 reflections
2818 reflections with $I > 2\sigma(I)$	intensity decay: 0.5%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0221P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.025$	$+ 0.5203P]$
$wR(F^2) = 0.066$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.001$
4074 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}
244 parameters	$\Delta\rho_{min} = -0.66$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

I1—Cu	2.6765 (6)	Cu—N4	2.089 (3)
Cu—N1	1.980 (3)	Cu—N2	2.092 (3)
Cu—N3	1.981 (3)		
N1—Cu—N3	174.53 (14)	N4—Cu—N2	113.89 (13)
N1—Cu—N4	99.80 (14)	N1—Cu—I1	90.53 (10)
N3—Cu—N4	80.29 (14)	N3—Cu—I1	94.00 (10)
N1—Cu—N2	80.06 (13)	N4—Cu—I1	123.04 (9)
N3—Cu—N2	94.87 (14)	N2—Cu—I1	123.07 (9)

H atoms were placed in calculated positions, with C—H = 0.93 Å, and included in the final cycles of refinement riding on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atoms.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1985); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Barclay, G. A., Hoskins, B. F. & Kennard, C. H. L. (1963). *J. Chem. Soc.* pp. 5691–5699.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Molecular Structure Corporation (1985). *TEXSAN*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1992). *MSC/AFC Diffractometer Control Software*. Version 5.32. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
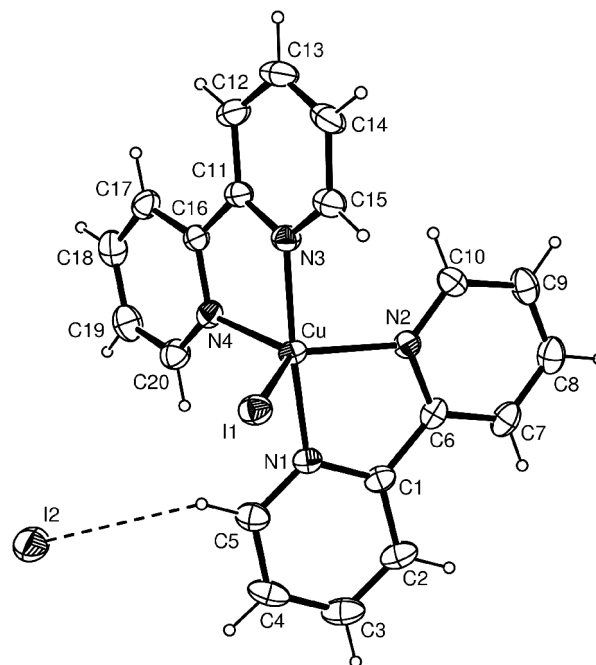North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Figure 1
A view of the structure of (I), shown with 30% probability displacement ellipsoids. The dashed line indicates the weak hydrogen bond.
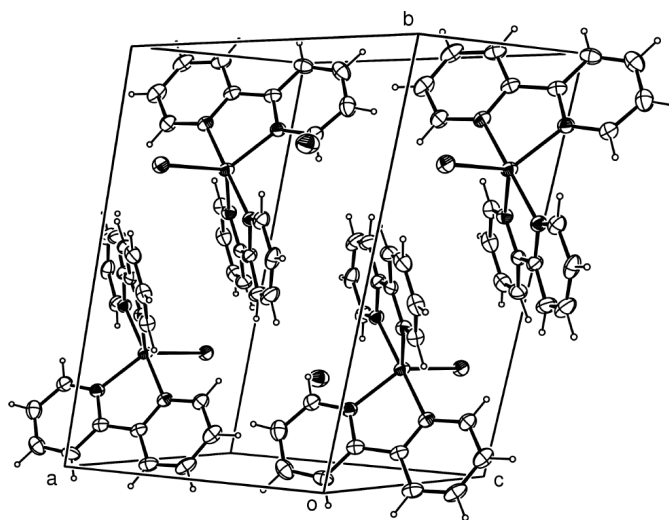

Figure 2
A packing diagram, showing π–π aromatic stacking between neighboring molecules.