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Dipyridinebis[4,4,4-trifluoro-1-(4-nitrophenyl)butane-1,3-dionato]copper(II)

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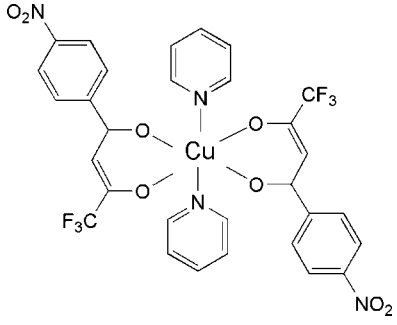
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.004 Å; R factor = 0.047; wR factor = 0.108; data-to-parameter ratio = 14.7.

In the centrosymmetric title compound, $[Cu(C_{10}H_5F_3NO_4)_2(C_5H_5N)_2]$, the Cu^{II} atom is hexacoordinate and lies in a square plane formed by four O atoms. Two pyridine molecules complete the coordination in *trans* positions. In the crystal structure there are intramolecular C—H···F and intermolecular C—H···O hydrogen bonds.

Related literature

For related literature, see: Shavaleev *et al.* (2003); Sloopa *et al.* (2002).



Experimental

Crystal data

$[Cu(C_{10}H_5F_3NO_4)_2(C_5H_5N)_2]$
M_r = 742.04
Monoclinic, $P2_1/c$
a = 12.5939 (9) Å
b = 8.7423 (6) Å
c = 14.1918 (10) Å
β = 106.245 (1)°
V = 1500.13 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.83 mm^{-1}
T = 292 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001*a*)
T_{min} = 0.852, T_{max} = 0.922
12289 measured reflections
3278 independent reflections
2298 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.108
S = 0.97
3278 reflections
223 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—N2	2.0197 (19)	Cu1—O3	2.2483 (18)
Cu1—O4	2.0443 (18)		
N2^i—Cu1—N2	180	N2—Cu1—O3	90.34 (7)
N2—Cu1—O4^i	88.70 (8)	O4^i—Cu1—O3	95.04 (7)
N2—Cu1—O4	91.30 (8)	O4—Cu1—O3	84.96 (7)
O4^i—Cu1—O4	180	O3—Cu1—O3^i	180
N2^i—Cu1—O3	89.66 (7)		

Symmetry code: (i) $-x+1, -y+2, -z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···F2	0.93	2.33	2.723 (3)	105
C5—H5···O1^{ii}	0.93	2.54	3.293 (4)	138

Symmetry code: (ii) $x, -y+\frac{5}{2}, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2036).

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supplementary materials

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Dipyridinebis[4,4,4-trifluoro-1-(4-nitrophenyl)butane-1,3-dionato]copper(II)

L. Fan, D.-J. Wang and J. Zheng

Comment

1,3-Diketones are invaluable chelating ligand for various lanthanide and transition metals in material chemistry (Shavaleev *et al.*, 2003). In this paper, we report the crystallography structure of the title compound, $Cu(C_{10}H_5O_4F_3N)_2(C_5H_5N)_2$. In the title molecular structure (I), Cu^{II} is coordinated by two 4,4,4-trifluoro-1-(4-nitrophenyl)butane-1,3-dione oxygen atoms and two nitrogen atoms of pyridines, forming a distorted octahedron coordination geometry (Fig. 1).

Experimental

The ligand 4,4,4-trifluoro-1-(4-nitrophenyl)butane-1,3-dione was synthesized from reported literature(Sloopa *et al.*, 2002). The coordination compounds were prepared according to the following procedure: The ligand (0.52 g, 2.0 mmol) and pyridine (0.16 g, 2.0 mmol) in 20 ml hot acetone was added slowly to the $CuCl_2{\cdot}2H_2O$ (0.17 g, 1.0 mmol) solution of 10 ml water. The mixture was stirred for 3 h. After filtration, the green solution was allowed to stand at room temperature. Green block-shaped crystals suitable for X-ray analysis were obtained after several days in 55% yield.

Refinement

All the H atoms were placed at their idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

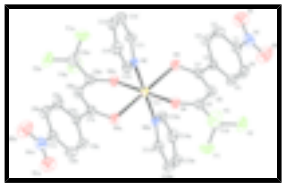


Fig. 1. View of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Dipyridinebis[4,4,4-trifluoro-1-(4-nitrophenyl)butane-1,3-dionato]copper(II)

Crystal data

$[Cu(C_{10}H_5F_3NO_4)_2(C_5H_5N)_2]$	$F_{000} = 750$
$M_r = 742.04$	$D_x = 1.643$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P2ybc	Cell parameters from 2698 reflections
$a = 12.5939$ (9) Å	$\theta = 2.8$–22.9°

b = 8.7423 (6) Å	μ = 0.83 mm^{-1}
c = 14.1918 (10) Å	T = 292 (2) K
β = 106.2450 (10)º	Block, green
V = 1500.13 (18) Å^3	0.20 × 0.10 × 0.10 mm
Z = 2	

Data collection

Bruker APEX CCD area-detector diffractometer	3278 independent reflections
Radiation source: fine-focus sealed tube	2298 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.080
T = 292(2) K	θ_{max} = 27.0º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001a)	h = −15→16
T_{min} = 0.852, T_{max} = 0.922	k = −11→11
12289 measured reflections	l = −16→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.047	H-atom parameters constrained
$wR(F^2)$ = 0.108	$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.97	$(\Delta/\sigma)_{max} < 0.001$
3278 reflections	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
223 parameters	$\Delta\rho_{min}$ = −0.61 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}

Cu1	0.5000	1.0000	0.0000	0.03413 (15)
C11	0.5305 (2)	0.7430 (3)	0.13701 (19)	0.0436 (6)
H11	0.4932	0.8076	0.1690	0.052*
C14	0.6375 (2)	0.5529 (3)	0.0444 (2)	0.0535 (8)
H14	0.6738	0.4893	0.0110	0.064*
C12	0.5706 (3)	0.6059 (4)	0.1804 (2)	0.0565 (8)
H12	0.5608	0.5784	0.2407	0.068*
C13	0.6253 (3)	0.5107 (3)	0.1328 (3)	0.0597 (8)
H13	0.6538	0.4179	0.1609	0.072*
C15	0.5954 (2)	0.6912 (3)	0.0050 (2)	0.0426 (6)
H15	0.6035	0.7195	−0.0558	0.051*
C9	0.3050 (2)	0.9483 (3)	−0.16549 (19)	0.0391 (6)
C10	0.2652 (3)	0.9380 (3)	−0.2776 (2)	0.0497 (7)
C7	0.2538 (2)	1.0169 (3)	−0.0145 (2)	0.0391 (6)
C4	0.1679 (2)	1.0904 (3)	0.02552 (18)	0.0367 (6)
C5	0.0934 (2)	1.1942 (3)	−0.0283 (2)	0.0511 (8)
H5	0.0922	1.2145	−0.0929	0.061*
C6	0.0200 (2)	1.2689 (4)	0.0128 (2)	0.0546 (8)
H6	−0.0301	1.3396	−0.0236	0.065*
C1	0.0226 (2)	1.2369 (3)	0.10764 (19)	0.0428 (7)
C2	0.0945 (2)	1.1325 (3)	0.1627 (2)	0.0503 (7)
H2	0.0946	1.1116	0.2270	0.060*
C3	0.1663 (2)	1.0594 (3)	0.1210 (2)	0.0469 (7)
H3	0.2151	0.9874	0.1576	0.056*
C8	0.2297 (2)	0.9966 (3)	−0.1179 (2)	0.0432 (6)
H8	0.1582	1.0175	−0.1560	0.052*
F1	0.31560 (18)	1.0415 (2)	−0.31809 (13)	0.0820 (6)
F2	0.15767 (15)	0.9595 (2)	−0.31617 (13)	0.0709 (5)
F3	0.28914 (16)	0.8027 (2)	−0.30905 (13)	0.0776 (6)
N2	0.54337 (16)	0.7864 (2)	0.05053 (15)	0.0355 (5)
N1	−0.0522 (2)	1.3223 (3)	0.1521 (2)	0.0583 (7)
O4	0.40646 (14)	0.9163 (2)	−0.13156 (13)	0.0436 (5)
O3	0.34342 (15)	0.9824 (2)	0.04590 (14)	0.0462 (5)
O1	−0.0459 (2)	1.2980 (3)	0.23779 (17)	0.0787 (7)
O2	−0.1149 (2)	1.4130 (4)	0.1026 (2)	0.1013 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0334 (3)	0.0330 (2)	0.0349 (3)	0.00422 (19)	0.0077 (2)	0.00201 (19)
C11	0.0448 (16)	0.0424 (15)	0.0437 (16)	0.0004 (12)	0.0124 (14)	0.0023 (13)
C14	0.0515 (19)	0.0434 (16)	0.060 (2)	0.0106 (14)	0.0055 (16)	−0.0050 (15)
C12	0.065 (2)	0.0530 (19)	0.0460 (18)	−0.0076 (16)	0.0066 (16)	0.0139 (15)
C13	0.062 (2)	0.0375 (16)	0.066 (2)	0.0076 (15)	−0.0041 (18)	0.0094 (16)
C15	0.0391 (15)	0.0410 (15)	0.0460 (17)	0.0018 (12)	0.0093 (13)	−0.0018 (13)
C9	0.0403 (16)	0.0327 (13)	0.0424 (17)	−0.0009 (11)	0.0084 (14)	0.0016 (12)
C10	0.0504 (19)	0.0483 (16)	0.0490 (19)	0.0025 (14)	0.0118 (16)	−0.0075 (15)
C7	0.0368 (15)	0.0344 (14)	0.0438 (16)	−0.0056 (11)	0.0076 (13)	0.0008 (12)

C4	0.0318 (14)	0.0381 (14)	0.0363 (15)	−0.0022 (11)	0.0033 (12)	−0.0016 (12)
C5	0.0516 (18)	0.068 (2)	0.0329 (16)	0.0151 (15)	0.0096 (14)	0.0078 (14)
C6	0.0487 (19)	0.065 (2)	0.0448 (18)	0.0191 (15)	0.0050 (15)	0.0042 (15)
C1	0.0384 (16)	0.0453 (16)	0.0439 (17)	−0.0041 (12)	0.0103 (14)	−0.0059 (13)
C2	0.062 (2)	0.0518 (17)	0.0394 (16)	0.0014 (15)	0.0190 (15)	0.0049 (14)
C3	0.0523 (18)	0.0455 (15)	0.0394 (16)	0.0096 (13)	0.0071 (14)	0.0091 (13)
C8	0.0361 (15)	0.0489 (16)	0.0414 (16)	0.0013 (12)	0.0053 (13)	−0.0034 (13)
F1	0.1064 (17)	0.0922 (15)	0.0497 (12)	−0.0276 (12)	0.0255 (12)	0.0104 (10)
F2	0.0570 (12)	0.1016 (15)	0.0449 (10)	0.0159 (10)	−0.0007 (9)	−0.0109 (10)
F3	0.0984 (15)	0.0669 (12)	0.0623 (12)	0.0208 (11)	0.0139 (11)	−0.0242 (10)
N2	0.0330 (12)	0.0333 (11)	0.0376 (12)	0.0026 (9)	0.0057 (10)	0.0032 (10)
N1	0.0515 (17)	0.0661 (18)	0.0602 (19)	0.0030 (14)	0.0202 (15)	−0.0093 (15)
O4	0.0390 (11)	0.0445 (11)	0.0465 (12)	0.0073 (8)	0.0106 (9)	0.0032 (9)
O3	0.0411 (11)	0.0515 (12)	0.0426 (11)	0.0077 (9)	0.0061 (10)	0.0037 (9)
O1	0.0898 (18)	0.0966 (19)	0.0572 (15)	0.0128 (14)	0.0331 (14)	−0.0094 (13)
O2	0.091 (2)	0.132 (3)	0.086 (2)	0.064 (2)	0.0332 (17)	0.0184 (19)

Geometric parameters (Å, °)

Cu1—N2^{i}	2.0197 (19)	C10—F2	1.324 (3)
Cu1—N2	2.0197 (19)	C10—F1	1.326 (3)
Cu1—O4^{i}	2.0443 (18)	C10—F3	1.328 (3)
Cu1—O4	2.0443 (18)	C7—O3	1.249 (3)
Cu1—O3	2.2483 (18)	C7—C8	1.424 (4)
Cu1—O3^{i}	2.2483 (18)	C7—C4	1.500 (3)
C11—N2	1.337 (3)	C4—C5	1.373 (4)
C11—C12	1.377 (4)	C4—C3	1.388 (3)
C11—H11	0.9300	C5—C6	1.387 (4)
C14—C13	1.357 (4)	C5—H5	0.9300
C14—C15	1.375 (4)	C6—C1	1.366 (4)
C14—H14	0.9300	C6—H6	0.9300
C12—C13	1.372 (4)	C1—C2	1.366 (4)
C12—H12	0.9300	C1—N1	1.475 (3)
C13—H13	0.9300	C2—C3	1.369 (4)
C15—N2	1.332 (3)	C2—H2	0.9300
C15—H15	0.9300	C3—H3	0.9300
C9—O4	1.264 (3)	C8—H8	0.9300
C9—C8	1.375 (4)	N1—O2	1.198 (3)
C9—C10	1.531 (4)	N1—O1	1.215 (3)
N2^{i}—Cu1—N2	180.0	F2—C10—C9	114.9 (2)
N2^{i}—Cu1—O4^{i}	91.30 (8)	F1—C10—C9	110.7 (2)
N2—Cu1—O4^{i}	88.70 (8)	F3—C10—C9	111.3 (2)
N2^{i}—Cu1—O4	88.70 (8)	O3—C7—C8	124.6 (2)
N2—Cu1—O4	91.30 (8)	O3—C7—C4	116.9 (2)
O4^{i}—Cu1—O4	180.0	C8—C7—C4	118.4 (2)
N2^{i}—Cu1—O3	89.66 (7)	C5—C4—C3	118.5 (2)
N2—Cu1—O3	90.34 (7)	C5—C4—C7	121.7 (2)

$O4^{i}$—Cu1—O3	95.04 (7)	C3—C4—C7	119.7 (2)
O4—Cu1—O3	84.96 (7)	C4—C5—C6	120.6 (3)
$N2^{i}$—Cu1—$O3^{i}$	90.34 (7)	C4—C5—H5	119.7
N2—Cu1—$O3^{i}$	89.66 (7)	C6—C5—H5	119.7
$O4^{i}$—Cu1—$O3^{i}$	84.96 (7)	C1—C6—C5	118.8 (3)
O4—Cu1—$O3^{i}$	95.04 (7)	C1—C6—H6	120.6
O3—Cu1—$O3^{i}$	180.0	C5—C6—H6	120.6
N2—C11—C12	122.3 (3)	C2—C1—C6	122.0 (3)
N2—C11—H11	118.9	C2—C1—N1	119.6 (2)
C12—C11—H11	118.9	C6—C1—N1	118.4 (3)
C13—C14—C15	119.0 (3)	C1—C2—C3	118.5 (3)
C13—C14—H14	120.5	C1—C2—H2	120.8
C15—C14—H14	120.5	C3—C2—H2	120.8
C13—C12—C11	118.7 (3)	C2—C3—C4	121.5 (3)
C13—C12—H12	120.6	C2—C3—H3	119.2
C11—C12—H12	120.6	C4—C3—H3	119.2
C14—C13—C12	119.4 (3)	C9—C8—C7	124.6 (3)
C14—C13—H13	120.3	C9—C8—H8	117.7
C12—C13—H13	120.3	C7—C8—H8	117.7
N2—C15—C14	122.6 (3)	C15—N2—C11	118.0 (2)
N2—C15—H15	118.7	C15—N2—Cu1	121.74 (17)
C14—C15—H15	118.7	C11—N2—Cu1	119.88 (17)
O4—C9—C8	130.0 (3)	O2—N1—O1	123.4 (3)
O4—C9—C10	112.7 (2)	O2—N1—C1	118.7 (3)
C8—C9—C10	117.2 (2)	O1—N1—C1	118.0 (3)
F2—C10—F1	106.4 (3)	C9—O4—Cu1	122.10 (16)
F2—C10—F3	106.9 (2)	C7—O3—Cu1	118.85 (16)
F1—C10—F3	106.3 (2)		
N2—C11—C12—C13	−0.2 (4)	C14—C15—N2—C11	−1.2 (4)
C15—C14—C13—C12	0.6 (5)	C14—C15—N2—Cu1	171.9 (2)
C11—C12—C13—C14	−0.6 (5)	C12—C11—N2—C15	1.2 (4)
C13—C14—C15—N2	0.4 (4)	C12—C11—N2—Cu1	−172.1 (2)
O4—C9—C10—F2	175.3 (2)	$O4^{i}$—Cu1—N2—C15	−121.37 (19)
C8—C9—C10—F2	−7.8 (4)	O4—Cu1—N2—C15	58.63 (19)
O4—C9—C10—F1	−64.2 (3)	O3—Cu1—N2—C15	143.59 (19)
C8—C9—C10—F1	112.7 (3)	$O3^{i}$—Cu1—N2—C15	−36.41 (19)
O4—C9—C10—F3	53.7 (3)	$O4^{i}$—Cu1—N2—C11	51.61 (19)
C8—C9—C10—F3	−129.4 (3)	O4—Cu1—N2—C11	−128.39 (19)
O3—C7—C4—C5	148.8 (3)	O3—Cu1—N2—C11	−43.43 (19)
C8—C7—C4—C5	−28.8 (4)	$O3^{i}$—Cu1—N2—C11	136.57 (19)
O3—C7—C4—C3	−27.9 (3)	C2—C1—N1—O2	−179.7 (3)
C8—C7—C4—C3	154.5 (2)	C6—C1—N1—O2	2.3 (4)
C3—C4—C5—C6	1.5 (4)	C2—C1—N1—O1	1.1 (4)
C7—C4—C5—C6	−175.3 (3)	C6—C1—N1—O1	−176.8 (3)
C4—C5—C6—C1	−0.4 (5)	C8—C9—O4—Cu1	−22.4 (4)
C5—C6—C1—C2	−0.7 (5)	C10—C9—O4—Cu1	154.05 (17)

C5—C6—C1—N1	177.2 (3)	$N2^{i}$—Cu1—O4—C9	−56.13 (19)
C6—C1—C2—C3	0.5 (4)	N2—Cu1—O4—C9	123.87 (19)
N1—C1—C2—C3	−177.3 (3)	O3—Cu1—O4—C9	33.64 (19)
C1—C2—C3—C4	0.6 (4)	$O3^{i}$—Cu1—O4—C9	−146.36 (19)
C5—C4—C3—C2	−1.6 (4)	C8—C7—O3—Cu1	31.7 (3)
C7—C4—C3—C2	175.2 (3)	C4—C7—O3—Cu1	−145.79 (17)
O4—C9—C8—C7	−1.8 (5)	$N2^{i}$—Cu1—O3—C7	49.69 (18)
C10—C9—C8—C7	−178.1 (2)	N2—Cu1—O3—C7	−130.31 (18)
O3—C7—C8—C9	−5.6 (4)	$O4^{i}$—Cu1—O3—C7	140.97 (18)
C4—C7—C8—C9	171.8 (2)	O4—Cu1—O3—C7	−39.03 (18)

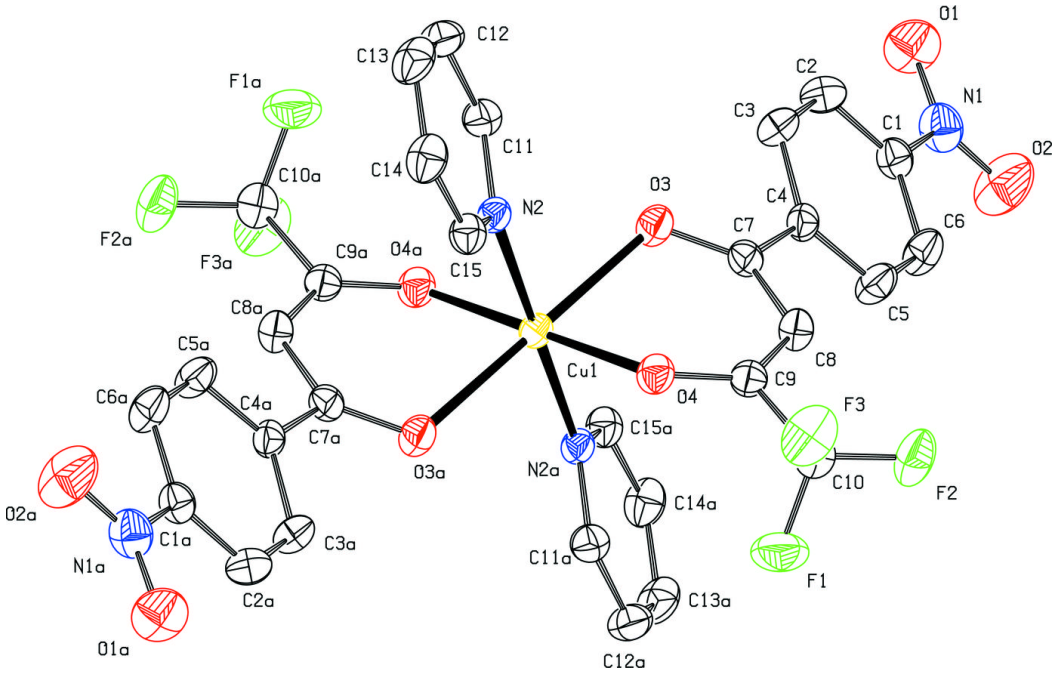
Symmetry codes: (i) −*x*+1, −*y*+2, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···F2	0.93	2.33	2.723 (3)	105
$C5—H5···O1^{ii}$	0.93	2.54	3.293 (4)	138

Symmetry codes: (ii) *x*, −*y*+5/2, *z*−1/2.

Fig. 1


O1
C12
C13
C2
F1a
N1
C3
C1
C11
O2
C14
C10a
O3
N2
C4
F2a
C7
C6
C9a
O4a
C15
F3a
C5
C8a
C8
C5a
Cu1
C9
O4
C6a
C4a
F3
C15a
C7a
O3a
N2a
C10
F2
C14a
O2a
C1a
C3a
C11a
F1
N1a
C2a
C13a
O1a
C12a