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trans-Bis(1,1,1,5,5,5-hexafluoropentane-2,4-dionato-κ^2O,O')bis(4-methyl-1,2,3-selenadiazole-κN^3)copper(II)

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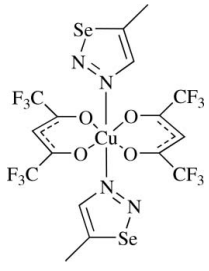
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; disorder in main residue; R factor = 0.055; wR factor = 0.126; data-to-parameter ratio = 14.0.

In the title compound, $[Cu(C_5HF_6O_2)_2(C_3H_4N_2Se)_2]$, the Cu^{II} atom (site symmetry $\overline{1}$) is coordinated by two O,O'-bidentate 1,1,1,5,5,5-hexafluoro-2,4-pentanedione (hp) ligands and two 4-methyl-1,2,3-selenadiazole molecules, resulting in a slightly distorted *trans*-CuN_2O_4 octahedral geometry in which the *cis* angles deviate by less than 3° from 90°. The selenadiazole plane is canted at 73.13 (17)° to the square plane defined by the pentanedionate O atoms. The F atoms of one of the hp ligands are disordered over two sets of sites in a 0.66 (3):0.34 (3) ratio. There are no significant intermolecular interactions in the crystal.

Related literature

Similar stuctures are exhibited by bis(hexafluoropentadionato) copper complexes of imidazole (Colacio *et al.*, 2000), pyrazole (Kogane *et al.*, 1990; Fokin *et al.*, 2002) and substituted pyridines (De Panthou *et al.*, 1996; Iwahori *et al.*, 2001; Sano *et al.*, 1997).



Experimental

Crystal data

$[Cu(C_5HF_6O_2)_2(C_3H_4N_2Se)_2]$
M_r = 771.74
Monoclinic, $P2_1/c$
a = 8.191 (2) Å
b = 14.390 (4) Å
c = 11.429 (4) Å
β = 104.86 (3)°
V = 1302.1 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.75 mm^{-1}
T = 293 K
0.25 × 0.25 × 0.25 mm

Data collection

Nicolet R3m/V diffractometer
Absorption correction: ψ scan (*SHELXTL*; Sheldrick, 2008)
T_{min} = 0.755, T_{max} = 0.793
3218 measured reflections
3014 independent reflections
1894 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
3 standard reflections every 97 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.126
S = 1.03
3014 reflections
215 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu—O1	1.967 (3)	Cu—N1	2.391 (4)
Cu—O2	1.981 (3)		

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXL97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5239).

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supplementary materials

Acta Cryst. (2010). E**66**, m241 [**doi:10.1107/S1600536810001297**]

trans-Bis(1,1,1,5,5,5-hexafluoropentane-2,4-dionato-κ^2O,O')bis(4-methyl-1,2,3-selenadiazole-κN^3)copper(II)

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Comment

The title molecule, $[Cu(C_3HF_6O_2)_2(C_3H_4N_2Se)_2]$, (I) was prepared as a potential precursor to $CuInSe_2$. The molecule (Fig. 1) is centrosymmetric resulting in pairs of equivalent ligands lying *trans* to each other in a slightly distorted octahedral coordination geometry in which the *cis* angles deviate less than 3° from right angles. The Cu is bound to N1 of the selenadiazole whose plane is canted at 73.13 (0.17) ° to the square plane defined by the pentanedionato oxygen atoms. The Cu—N bond is elongated. There are no intermolecular interactions.

Experimental

A solution of 4-methyl-1,2,3-selenadiazole (4.65 g, 0.032 mol) in dichloromethane was added dropwise to a solution of $Cu(hfac)_2.xH_2O$ (7.84 g, 0.016 mol) in dichloromethane/toluene (50 ml) at 273 K in the absence of light. The mixture was stirred for 12 h, the solvent removed *in vacuo* and the residue dissolved in warm toluene. Slow cooling afforded olive-green parallelepipeds of (I). Yield 8.13 g (65%), mpt. 354–356 K. Found: C, 24.86; H, 1.24; N, 7.31%. Calc.for $C_{16}H_{12}CuF_{12}N_4O_4Se_2$: C, 24.90; H, 1.31; N, 7.26%. μ 2.15 BM.

Refinement

All H atoms were placed in calculated positions and refined using a riding model. All other hydrogen atoms were located and fully refined.

Figures

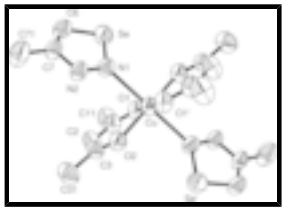

Fig. 1. The molecular structure of (I), with 50% probability displacement ellipsoids. Hydrogen atoms and the fluorine atoms have been excluded for clarity.

trans-Bis(1,1,1,5,5,5-hexafluoropentane-2,4-dionato- κ^2O,O')bis(4-methyl-1,2,3-selenadiazole- κN^3)copper(II)

Crystal data

$[Cu(C_5HF_6O_2)_2(C_3H_4N_2Se)_2]$	$D_x = 1.968$ Mg m^{-3}
$M_r = 771.74$	Melting point: 355 K

Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.191$ (2) Å	Cell parameters from 20 reflections
$b = 14.390$ (4) Å	$\theta = 7.9–14.5°$
$c = 11.429$ (4) Å	$\mu = 3.75$ mm^{-1}
$\beta = 104.86$ (3)°	$T = 293$ K
$V = 1302.1$ (7) Å^3	Parallelpiped, green
$Z = 2$	0.25 × 0.25 × 0.25 mm
$F(000) = 742$	

Data collection

Nicolet R3m/V diffractometer	$R_{int} = 0.048$
graphite	$\theta_{max} = 27.6°$, $\theta_{min} = 2.3°$
profile data from θ/2θ scans	$h = -2 \rightarrow 10$
Absorption correction: ψ scan (SHELXTL; Sheldrick, 2008)	$k = -6 \rightarrow 18$
$T_{min} = 0.755$, $T_{max} = 0.793$	$l = -14 \rightarrow 14$
3218 measured reflections	3 standard reflections every 97 reflections
3014 independent reflections	intensity decay: none
1894 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.055$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.126$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 1.964P]$ where $P = (F_o^2 + 2F_c^2)/3$
3014 reflections	$(\Delta/\sigma)_{max} < 0.001$
215 parameters	$\Delta\rho_{max} = 0.44$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.42$ e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cu	0.0000	0.0000	0.5000	0.0467 (2)	

Se	0.11516 (8)	0.14840 (4)	0.26686 (5)	0.0667 (2)	
N1	0.0700 (6)	0.1433 (3)	0.4188 (4)	0.0564 (11)	
N2	0.0391 (6)	0.2234 (3)	0.4541 (4)	0.0572 (11)	
C6	0.0853 (8)	0.2732 (4)	0.2739 (5)	0.0628 (15)	
C7	0.0470 (7)	0.2966 (4)	0.3777 (5)	0.0577 (13)	
C71	0.0087 (11)	0.3911 (4)	0.4164 (6)	0.093 (2)	
H71A	−0.0143	0.4322	0.3479	0.140*	
H71B	0.1040	0.4139	0.4772	0.140*	
H71C	−0.0881	0.3882	0.4489	0.140*	
O1	−0.1707 (5)	−0.0178 (2)	0.3457 (3)	0.0546 (9)	
O2	−0.1614 (5)	0.0692 (2)	0.5702 (3)	0.0520 (8)	
C1	−0.3071 (7)	0.0274 (4)	0.3148 (5)	0.0582 (14)	
C11	−0.4037 (9)	0.0109 (5)	0.1836 (6)	0.0771 (19)	
F11	−0.5552 (6)	0.0472 (5)	0.1545 (4)	0.157 (3)	
F12	−0.3218 (6)	0.0490 (3)	0.1084 (3)	0.1142 (15)	
F13	−0.4170 (6)	−0.0780 (3)	0.1545 (4)	0.1076 (14)	
C2	−0.3728 (8)	0.0879 (4)	0.3859 (5)	0.0674 (16)	
C3	−0.2960 (7)	0.1034 (4)	0.5075 (5)	0.0541 (13)	
C31	−0.3885 (10)	0.1722 (5)	0.5750 (7)	0.0782 (19)	
F311	−0.318 (2)	0.2515 (8)	0.581 (3)	0.145 (9)	0.66 (3)
F312	−0.5418 (12)	0.1700 (18)	0.5425 (19)	0.161 (10)	0.66 (3)
F313	−0.349 (2)	0.1452 (18)	0.6957 (10)	0.137 (6)	0.66 (3)
F321	−0.300 (2)	0.210 (2)	0.663 (3)	0.105 (10)	0.34 (3)
F322	−0.520 (6)	0.141 (2)	0.591 (5)	0.18 (2)	0.34 (3)
F323	−0.457 (6)	0.2452 (19)	0.4941 (19)	0.138 (14)	0.34 (3)
H2	−0.475 (7)	0.118 (4)	0.347 (5)	0.065 (17)*	
H6	0.085 (9)	0.314 (5)	0.207 (7)	0.12 (3)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu	0.0603 (5)	0.0401 (4)	0.0375 (4)	0.0048 (4)	0.0085 (4)	−0.0043 (4)
Se	0.0834 (5)	0.0652 (4)	0.0568 (4)	0.0044 (3)	0.0276 (3)	−0.0066 (3)
N1	0.072 (3)	0.051 (3)	0.051 (2)	0.001 (2)	0.023 (2)	0.004 (2)
N2	0.081 (3)	0.047 (3)	0.047 (2)	0.002 (2)	0.023 (2)	0.005 (2)
C6	0.080 (4)	0.063 (4)	0.049 (3)	−0.001 (3)	0.022 (3)	0.012 (3)
C7	0.080 (4)	0.047 (3)	0.046 (3)	0.002 (3)	0.014 (3)	0.009 (2)
C71	0.152 (7)	0.059 (4)	0.076 (4)	0.002 (4)	0.044 (5)	0.001 (3)
O1	0.065 (2)	0.051 (2)	0.0436 (18)	0.0042 (18)	0.0060 (17)	−0.0060 (16)
O2	0.064 (2)	0.047 (2)	0.0444 (18)	0.0014 (17)	0.0132 (17)	−0.0044 (15)
C1	0.067 (4)	0.058 (3)	0.045 (3)	0.003 (3)	0.007 (3)	−0.002 (2)
C11	0.077 (4)	0.092 (5)	0.052 (4)	0.012 (4)	−0.003 (3)	−0.016 (3)
F11	0.098 (3)	0.260 (7)	0.081 (3)	0.080 (4)	−0.033 (2)	−0.062 (4)
F12	0.149 (4)	0.133 (4)	0.052 (2)	−0.001 (3)	0.011 (2)	0.013 (2)
F13	0.130 (4)	0.102 (3)	0.072 (2)	−0.014 (3)	−0.008 (2)	−0.024 (2)
C2	0.061 (4)	0.074 (4)	0.060 (3)	0.013 (3)	0.003 (3)	−0.014 (3)
C3	0.056 (3)	0.048 (3)	0.061 (3)	−0.001 (3)	0.019 (3)	−0.010 (3)
C31	0.081 (5)	0.070 (5)	0.089 (5)	−0.002 (4)	0.032 (4)	−0.030 (4)

F311	0.164 (16)	0.064 (6)	0.23 (2)	0.004 (7)	0.095 (18)	−0.043 (9)
F312	0.045 (6)	0.27 (2)	0.158 (13)	0.041 (8)	−0.001 (6)	−0.123 (14)
F313	0.147 (12)	0.188 (15)	0.088 (6)	0.053 (10)	0.052 (7)	−0.030 (8)
F321	0.067 (11)	0.12 (2)	0.104 (17)	0.027 (14)	−0.015 (11)	−0.071 (14)
F322	0.23 (4)	0.132 (17)	0.27 (5)	−0.10 (2)	0.22 (4)	−0.10 (2)
F323	0.21 (3)	0.101 (14)	0.094 (12)	0.105 (18)	0.034 (16)	0.019 (10)

Geometric parameters (Å, °)

Cu—O1	1.967 (3)	C1—C2	1.390 (8)
Cu—O2	1.981 (3)	C1—C11	1.524 (8)
Cu—N1	2.391 (4)	C11—F11	1.308 (7)
Se—C6	1.817 (6)	C11—F13	1.318 (8)
Se—N1	1.867 (4)	C11—F12	1.336 (8)
N1—N2	1.268 (6)	C2—C3	1.389 (8)
N2—C7	1.380 (6)	C2—H2	0.95 (6)
C6—C7	1.345 (7)	C3—C31	1.565 (8)
C6—H6	0.97 (8)	C31—F312	1.214 (12)
C7—C71	1.488 (8)	C31—F311	1.273 (12)
C71—H71A	0.96	C31—F313	1.389 (15)
C71—H71B	0.96	C31—F321	1.204 (17)
C71—H71C	0.96	C31—F322	1.23 (2)
O1—C1	1.262 (6)	C31—F323	1.418 (17)
O2—C3	1.252 (6)		
O1—Cu—O2^{i}	88.06 (14)	O1—C1—C11	113.2 (5)
O1—Cu—O2	91.94 (14)	C2—C1—C11	119.5 (5)
O1—Cu—N1	87.15 (15)	F11—C11—F13	108.2 (6)
O1^{i}—Cu—N1	92.85 (15)	F11—C11—F12	105.9 (6)
O2^{i}—Cu—N1	91.44 (15)	F13—C11—F12	105.0 (6)
O2—Cu—N1	88.57 (15)	F11—C11—C1	114.0 (5)
C6—Se—N1	86.4 (2)	F13—C11—C1	112.8 (6)
N2—N1—Se	111.3 (3)	F12—C11—C1	110.4 (6)
N2—N1—Cu	125.0 (3)	C3—C2—C1	122.8 (6)
Se—N1—Cu	121.3 (2)	C3—C2—H2	121 (3)
N1—N2—C7	116.6 (4)	C1—C2—H2	116 (3)
C7—C6—Se	110.6 (4)	O2—C3—C2	128.1 (5)
C7—C6—H6	126 (4)	O2—C3—C31	115.7 (5)
Se—C6—H6	123 (4)	C2—C3—C31	116.3 (6)
C6—C7—N2	115.2 (5)	F312—C31—F311	117.2 (13)
C6—C7—C71	127.2 (5)	F312—C31—F313	104.9 (12)
N2—C7—C71	117.5 (5)	F311—C31—F313	102.0 (11)
C7—C71—H71A	109.5	F312—C31—C3	115.2 (8)
C7—C71—H71B	109.5	F311—C31—C3	109.0 (8)
H71A—C71—H71B	109.5	F313—C31—C3	107.1 (8)
C7—C71—H71C	109.5	F321—C31—F322	114 (2)
H71A—C71—H71C	109.5	F321—C31—F323	105.4 (16)
H71B—C71—H71C	109.5	F322—C31—F323	99 (2)
C1—O1—Cu	123.9 (3)	F321—C31—C3	115.3 (10)

C3—O2—Cu	123.2 (3)	F322—C31—C3	113.4 (14)
O1—C1—C2	127.4 (5)	F323—C31—C3	107.9 (9)
C6—Se—N1—N2	0.8 (4)	Cu—O1—C1—C11	171.1 (4)
C6—Se—N1—Cu	164.0 (3)	O1—C1—C11—F11	171.4 (6)
O1—Cu—N1—N2	109.9 (4)	C2—C1—C11—F11	−8.8 (10)
O1^{i}—Cu—N1—N2	−70.1 (4)	O1—C1—C11—F13	47.5 (8)
O2^{i}—Cu—N1—N2	−162.1 (4)	C2—C1—C11—F13	−132.7 (6)
O2—Cu—N1—N2	17.9 (4)	O1—C1—C11—F12	−69.6 (7)
O1—Cu—N1—Se	−50.9 (3)	C2—C1—C11—F12	110.2 (7)
O1^{i}—Cu—N1—Se	129.1 (3)	O1—C1—C2—C3	−3.2 (11)
O2^{i}—Cu—N1—Se	37.1 (3)	C11—C1—C2—C3	177.1 (6)
O2—Cu—N1—Se	−142.9 (3)	Cu—O2—C3—C2	11.9 (8)
Se—N1—N2—C7	−1.0 (6)	Cu—O2—C3—C31	−168.1 (4)
Cu—N1—N2—C7	−163.4 (4)	C1—C2—C3—O2	1.3 (10)
N1—Se—C6—C7	−0.5 (5)	C1—C2—C3—C31	−178.8 (6)
Se—C6—C7—N2	0.1 (7)	O2—C3—C31—F312	−145.3 (18)
Se—C6—C7—C71	−178.2 (6)	C2—C3—C31—F312	34.8 (19)
N1—N2—C7—C6	0.6 (8)	O2—C3—C31—F311	80.6 (17)
N1—N2—C7—C71	179.1 (6)	C2—C3—C31—F311	−99.4 (16)
O2^{i}—Cu—O1—C1	−164.5 (4)	O2—C3—C31—F313	−29.0 (14)
O2—Cu—O1—C1	15.5 (4)	C2—C3—C31—F313	151.0 (13)
N1^{i}—Cu—O1—C1	107.0 (4)	O2—C3—C31—F321	24 (3)
N1—Cu—O1—C1	−73.0 (4)	C2—C3—C31—F321	−156 (3)
O1—Cu—O2—C3	−16.9 (4)	O2—C3—C31—F322	−110 (3)
O1^{i}—Cu—O2—C3	163.1 (4)	C2—C3—C31—F322	71 (3)
N1^{i}—Cu—O2—C3	−109.8 (4)	O2—C3—C31—F323	142 (2)
N1—Cu—O2—C3	70.2 (4)	C2—C3—C31—F323	−38 (3)
Cu—O1—C1—C2	−8.6 (8)		

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

Fig. 1