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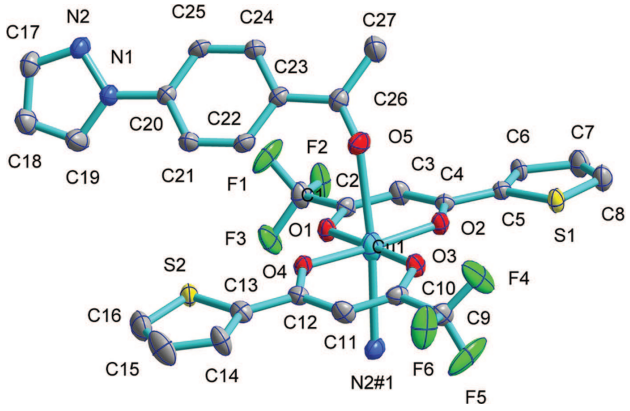
Crystal structure of *catena*-poly[(μ_2-1-(4-(1*H*-pyrazol-1-yl)phenyl)ethan-1-one-κ^2*N*:*O*)-bis(1,1,1-trifluoro-4-oxo-4-(thiophen-2-yl)but-2-en-2-olato-κ^2*O*,*O*′)copper(II)], $C_{27}H_{18}CuF_6N_2O_5S_2$



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Abstract

$C_{27}H_{18}CuF_6N_2O_5S_2$, triclinic, $P\bar{1}$ (no. 2), $a = 9.3162(5)$ Å, $b = 10.1290(6)$ Å, $c = 16.4352(9)$ Å, $\alpha = 90.430(4)°$, $\beta = 100.711(5)°$, $\gamma = 110.753(5)°$, $V = 1420.45(14)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0559$, $wR_{ref}(F^2) = 0.1617$, $T = 293$ K.

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The crystal structure is shown in the figure (#1: x, $y - 1$, z). Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

To a CH_2Cl_2 solution (20 mL) of $Cu(tta)_2$ (tta = theonyltrifluoroacetonate (systematic name: 1,1,1-trifluoro-4-oxo-4-(thiophen-2-yl)but-2-en-2-olate) 124.5 mg, 0.25 mmol), 1-(4-(1*H*-pyrazol-1-yl)phenyl)ethan-1-one (46.5 mg, 0.25 mmol) was added slowly with stirring. The mixture was stirred for 20 min and the resulting light green solution was kept at room temperature for several days. Green crystals formed upon evaporation of the solvent. The yield

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Table 1: Data collection and handling.

Crystal:	Block, green
Size:	0.28 × 0.22 × 0.19 mm
Wavelength:	Mo $K\alpha$ radiation (λ = 0.71073 Å)
μ:	0.996 mm^{-1}
Diffractometer, scan mode:	Eos Xcalibur, Φ and ω-scans
θ_{max}, completeness:	25.35°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10139, 5187, 0.0235
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3987
$N(param)_{refined}$:	389
Programs:	CrysAlis^{PRO} [1], SHELX [2], Diamond [3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Cu1	0.85687(5)	0.33336(4)	0.26454(3)	0.04122(19)
S1	1.28717(15)	0.25957(13)	0.43926(8)	0.0647(4)
S2	0.44177(16)	0.40753(13)	0.08438(9)	0.0675(4)
F1	1.1153(6)	0.7653(4)	0.1654(3)	0.1291(17)
F2	1.3025(4)	0.7073(4)	0.1458(2)	0.1046(13)
F3	1.0818(5)	0.6129(4)	0.07043(19)	0.1027(12)
F4	0.6254(7)	0.0066(5)	0.4360(2)	0.176(3)
F5	0.5812(8)	−0.1212(4)	0.3319(4)	0.176(3)
F6	0.4112(5)	−0.0669(5)	0.3641(3)	0.1426(19)
O1	0.9568(3)	0.4792(3)	0.19614(17)	0.0449(7)
O2	1.0552(3)	0.3319(3)	0.32513(16)	0.0437(6)
O3	0.7524(3)	0.1848(3)	0.33001(17)	0.0460(7)
O4	0.6589(3)	0.3430(3)	0.20816(16)	0.0423(6)
O5	0.8970(4)	0.5386(3)	0.36952(19)	0.0602(8)
N1	0.7829(4)	1.0064(3)	0.1351(2)	0.0451(8)
N2	0.8125(5)	1.1456(3)	0.1492(2)	0.0504(9)
C1	1.1517(5)	0.6590(5)	0.1481(3)	0.0526(11)
C2	1.1050(5)	0.5418(4)	0.2068(2)	0.0428(9)
C3	1.2198(5)	0.5177(4)	0.2614(3)	0.0483(10)
H3	1.3232	0.5750	0.2620	0.058
C4	1.1914(4)	0.4113(4)	0.3171(2)	0.0381(8)
C5	1.3224(5)	0.3852(4)	0.3691(2)	0.0395(9)
C6	1.4862(5)	0.4536(4)	0.3692(2)	0.0438(9)
H6	1.5299	0.5239	0.3355	0.053
C7	1.5712(6)	0.3933(5)	0.4316(3)	0.0597(12)
H7	1.6799	0.4221	0.4429	0.072
C8	1.4803(6)	0.2927(5)	0.4714(3)	0.0603(12)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H8	1.5198	0.2456	0.5130	0.072
C9	0.5594(6)	−0.0183(4)	0.3601(3)	0.0543(11)
C10	0.6070(5)	0.1101(4)	0.3105(2)	0.0432(9)
C11	0.4942(5)	0.1314(4)	0.2517(3)	0.0500(10)
H11	0.3928	0.0646	0.2428	0.060
C12	0.5240(5)	0.2490(4)	0.2043(2)	0.0389(8)
C13	0.3962(5)	0.2707(4)	0.1460(2)	0.0467(10)
C14	0.2309(5)	0.1929(6)	0.1314(3)	0.0614(12)
H14	0.1805	0.1162	0.1595	0.074
C15	0.1570(7)	0.2539(8)	0.0660(4)	0.0881(19)
H15	0.0495	0.2179	0.0451	0.106
C16	0.2539(7)	0.3661(7)	0.0373(3)	0.0765(16)
H16	0.2209	0.4163	−0.0046	0.092
C17	0.7614(6)	1.1848(5)	0.0763(3)	0.0545(11)
H17	0.7657	1.2766	0.0673	0.065
C18	0.7014(6)	1.0751(5)	0.0153(3)	0.0675(14)
H18	0.6595	1.0777	−0.0403	0.081
C19	0.7170(7)	0.9629(5)	0.0542(3)	0.0728(15)
H19	0.6878	0.8716	0.0298	0.087
C20	0.8253(5)	0.9241(4)	0.1985(2)	0.0412(9)
C21	0.7493(5)	0.7781(4)	0.1908(3)	0.0490(10)
H21	0.6689	0.7351	0.1453	0.059
C22	0.7922(5)	0.6972(4)	0.2499(3)	0.0484(10)
H22	0.7418	0.5993	0.2435	0.058
C23	0.9102(5)	0.7591(4)	0.3194(2)	0.0412(9)
C24	0.9832(5)	0.9066(4)	0.3271(3)	0.0500(10)
H24	1.0604	0.9504	0.3737	0.060
C25	0.9429(5)	0.9881(4)	0.2670(3)	0.0498(10)
H25	0.9946	1.0858	0.2724	0.060
C26	0.9548(5)	0.6674(4)	0.3815(3)	0.0466(10)
C27	1.0708(7)	0.7327(5)	0.4612(3)	0.0757(15)
H27A	1.0733	0.6599	0.4980	0.114
H27B	1.0398	0.8006	0.4872	0.114
H27C	1.1731	0.7790	0.4493	0.114

was 54%. Anal. Calcd. for $C_{30}H_{32}CuF_6N_4O_4S_2$: C, 46.86%; H, 2.62%; N, 4.05%. Found: C, 46.69%; H, 2.49%; N, 4.12%.

Experimental details

H atoms were situated into idealized positions with the carrier atom-H distances = 0.93 Å for aryl, 0.96 Å for the methyl H atoms. The U_{iso} values were constrained to be $1.5U_{eq}$(C) for the methyl H atoms and $1.2U_{eq}$ for others.

Discussion

Complexes built by N-heterocycle organic ligands have attracted much attention due to their applications as functional materials [4–6]. To get functional complexes, much effort has been devoted to modify the building blocks and to control them for required products *via* selecting different organic ligands [7–9]. In pursuit of our general research on the Cu-TTA chemistry, we have recently synthesized many Cu(II) complexes, which showed interesting structural features [10–12]. As known, the pyrazole derivatives such as hydrotris (3,5-dimethylpyrazolyl)borate are frequently chosen to fabricate various topological frameworks as for their rich coordination sites.

The Cu(II) ion is coordinated to one nitrogen and one oxygen atom from two symmetry related 1-(4-(1*H*-pyrazol-1-yl)phenyl)ethan-1-one and four oxygen atoms from four tta ligands to form a distorted octahedron. The bond lengths of Cu1—O1, Cu1—O2, Cu1—O3, Cu1—O4, Cu1—O5 are 1.937(3), 1.934(3), 1.935(3), 1.940(3) and 2.568(3) Å, respectively, while the bond length of Cu1—N1 is 2.545(3) Å. It is noted that the Cu1—O5 and Cu1—N2 are much longer than those reported in similar complexes [10–12], which may be due to its weak coordinating ability. The $Cu(TTA)_2$ units are linked by 1-(4-(1*H*-pyrazol-1-yl)phenyl)ethan-1-one ligands to a chain.

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