Crystal structure of bis[4,4,4]-trifluoro-1-(2-thienyl)butanedione-1,3] copper(II) with two 1-butylimidazole ligands, $C_{30}H_{32}CuF_6N_4O_4S_2$

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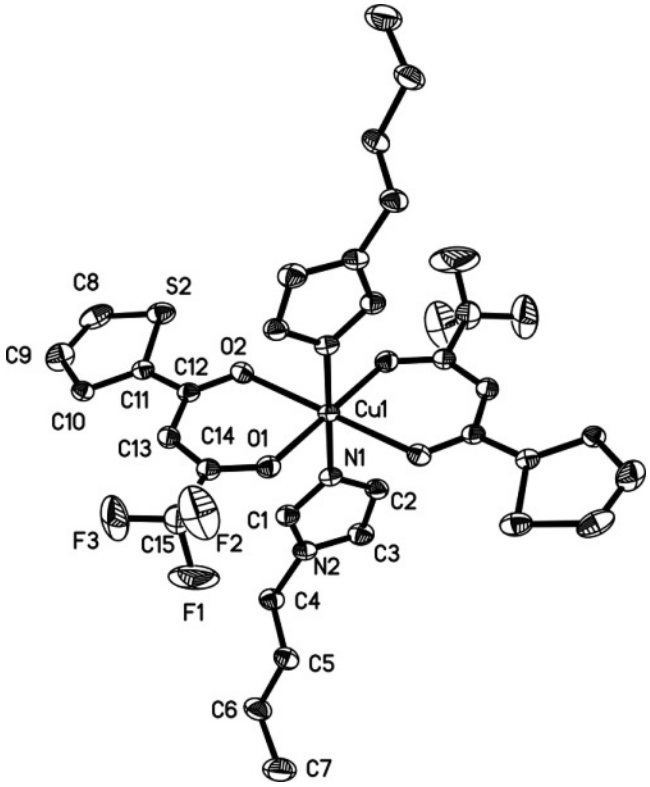

Abstract

$C_{30}H_{32}CuF_6N_4O_4S_2$, monoclinic, $P2_1/n$ (no. 14), a = 13.209(5) Å, b = 8.715(3) Å, c = 14.599(5) Å, β = 92.217(5)°, V = 1679.3 Å^3, Z = 2, $R_{gt}(F)$ = 0.0509, $wR_{ref}(F^2)$ = 0.1683, T = 296 K.

Table 1. Data collection and handling.

Crystal:	green blocks, size 0.21×0.24×0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.49 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12545, 3280
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2901
$N(param)_{refined}$:	216
Programs:	SHELX [9]

Source of material

To a CH_2Cl_2 solution (20 mL) of copper(II)thenoyltrifluoroacetonate (Cu(*tta*)$_2$, 124.5 mg, 0.25 mmol), 1-butylimidazole (93 mg, 0.75 mmol) was added slowly. The mixture was stirred for 20 min and the resulting light green solution filtrated was kept at room temperature for several days. Green crystals, suitable for X-ray crystallography, formed upon evaporation of the solvent. The yield was 86%. **Elemental Analysis** calcd. for $C_{30}H_{32}CuF_6N_4O_4S_2$: C, 47.77; H, 4.28; N, 7.43. Found: C, 47.72; H, 4.21; N, 7.49.

Experimental details

All H atoms were constrained to ideal geometries, with C–H = 0.93–0.97 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

Discussion

In the past decades, the fascinating applications of diketonate complexes have prompted studies on the architectures of many such complexes [1–3]. Among various ligands, imidazole derivatives are of special interest because of their high thermal stability [4–6] and their complex-forming ability [4–6]. The structure of these complexes may be influenced by such factors as the typical coordination of the metal ions, the structure characteristics of organic ligands, the metal-ligand ratio, the counter ions and many other contributions. Alteration of these factors can lead to the formation of new structures or extended frameworks, which allowed for the construction of a great variety of supramolecular architecture. In this paper, we report the synthesis and characterization of a copper(II) thenoyltri-fluoroacetonate complexes with two 1-butylimidazole ligands. The molecular structure of the compound is shown in the figure. The asymmetric unit of the title structure consists of one half of the title complex. In the crystal structure of the title complex, the O_{tta} atoms lie in the equatorial plane of a pulled tetragonally distorted octahedron (Cu–O_{tta} distances are 2.018(2) and 2.294(2) Å; the N atoms of 1-butylimidazole are in the axial positions forming Cu–N distances of 2.004(2) Å. The *tta* ligands, which are centrosymmetrically *trans* to each other, are nearly planar, the angle between the thenoyl heterocycle and the respective metallocycle being up to 14.7°. All the bond lengths in the molecule are within normal values [8].

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.6245	0.2893	0.4382	0.048
H(2)	4*e*	0.4844	0.2404	0.6677	0.051
H(3)	4*e*	0.5680	0.4904	0.6726	0.059
H(4A)	4*e*	0.6556	0.6597	0.5414	0.055
H(4B)	4*e*	0.6842	0.5711	0.4525	0.055
H(5A)	4*e*	0.8197	0.4552	0.5294	0.055
H(5B)	4*e*	0.7879	0.5262	0.6226	0.055
H(6A)	4*e*	0.8671	0.6914	0.4755	0.075
H(6B)	4*e*	0.8207	0.7750	0.5591	0.075
H(7A)	4*e*	0.9470	0.6819	0.6562	0.128
H(7B)	4*e*	0.9980	0.7611	0.5733	0.128
H(7C)	4*e*	0.9901	0.5818	0.5775	0.128

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(8)	4*e*	0.2281	0.4190	0.1195	0.077
H(9)	4*e*	0.3839	0.4424	0.0503	0.074

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(10)	4*e*	0.5236	0.3052	0.1307	0.043
H(13)	4*e*	0.6009	0.1201	0.2258	0.046

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.5997(2)	0.3080(3)	0.4959(2)	0.040(2)	0.036(1)	0.044(2)	–0.003(1)	–0.000(1)	–0.006(1)
C(2)	4*e*	0.5231(2)	0.2813(4)	0.6218(2)	0.038(2)	0.046(2)	0.044(2)	–0.007(1)	0.004(1)	–0.010(1)
C(3)	4*e*	0.5690(3)	0.4199(4)	0.6248(2)	0.049(2)	0.042(2)	0.055(2)	–0.006(1)	0.003(1)	–0.016(1)
C(4)	4*e*	0.6831(2)	0.5644(3)	0.5188(2)	0.048(2)	0.032(2)	0.057(2)	–0.007(1)	–0.002(1)	0.002(1)
C(5)	4*e*	0.7898(2)	0.5447(4)	0.5572(2)	0.044(2)	0.035(1)	0.057(2)	–0.003(1)	0.005(1)	0.003(1)
C(6)	4*e*	0.8561(3)	0.6832(5)	0.5407(3)	0.054(2)	0.059(2)	0.076(2)	–0.019(2)	–0.001(2)	0.017(2)
C(7)	4*e*	0.9569(3)	0.6764(6)	0.5915(4)	0.059(3)	0.092(3)	0.103(4)	–0.026(2)	–0.012(2)	0.015(3)
C(8)	4*e*	0.2871(3)	0.3720(4)	0.1422(3)	0.053(2)	0.054(2)	0.082(3)	0.019(2)	–0.029(2)	–0.017(2)
C(9)	4*e*	0.3757(3)	0.3855(4)	0.1033(3)	0.060(2)	0.057(2)	0.067(2)	0.016(2)	–0.015(2)	0.014(2)
C(10)	4*e*	0.4568(2)	0.3068(3)	0.1487(2)	0.027(1)	0.036(1)	0.043(1)	0.003(1)	–0.001(1)	0.004(1)
C(11)	4*e*	0.4187(2)	0.2292(3)	0.2277(2)	0.030(1)	0.037(1)	0.040(1)	0.001(1)	–0.002(1)	–0.004(1)
C(12)	4*e*	0.4733(2)	0.1369(3)	0.2986(2)	0.031(1)	0.035(1)	0.039(1)	–0.003(1)	0.001(1)	–0.007(1)
C(13)	4*e*	0.5757(2)	0.0960(4)	0.2827(2)	0.031(1)	0.046(2)	0.039(1)	0.003(1)	0.002(1)	0.001(1)
C(14)	4*e*	0.6397(2)	0.0238(3)	0.3452(2)	0.033(2)	0.035(1)	0.041(2)	0.001(1)	0.002(1)	–0.003(1)
C(15)	4*e*	0.7469(3)	–0.0099(5)	0.3155(3)	0.041(2)	0.080(3)	0.056(2)	0.018(2)	0.005(2)	0.008(2)
Cu(1)	2*c*	½	0	½	0.0253(3)	0.0306(3)	0.0352(3)	–0.0025(2)	–0.0006(2)	–0.0043(2)
F(1)	4*e*	0.8133(2)	0.0733(7)	0.3605(3)	0.034(1)	0.267(6)	0.173(4)	–0.034(2)	0.015(2)	–0.080(4)
F(2)	4*e*	0.7719(3)	–0.1535(5)	0.3344(3)	0.110(3)	0.133(3)	0.150(3)	0.084(2)	0.058(2)	0.052(2)
F(3)	4*e*	0.7626(3)	0.0083(4)	0.2296(2)	0.068(2)	0.201(4)	0.076(2)	0.061(2)	0.037(2)	0.038(2)
N(1)	4*e*	0.5428(2)	0.2107(3)	0.5406(2)	0.033(1)	0.035(1)	0.041(1)	–0.0032(9)	–0.0036(9)	–0.005(1)
N(2)	4*e*	0.6173(2)	0.4369(3)	0.5442(2)	0.036(1)	0.032(1)	0.049(1)	–0.004(1)	–0.003(1)	–0.004(1)
O(1)	4*e*	0.6257(2)	–0.0187(2)	0.4265(2)	0.035(1)	0.043(1)	0.044(1)	0.0015(8)	–0.0029(9)	0.0009(9)
O(2)	4*e*	0.4268(2)	0.1021(3)	0.3686(1)	0.032(1)	0.053(1)	0.045(1)	–0.0022(9)	0.0045(8)	0.0008(9)
S(2)	4*e*	0.29212(6)	0.2619(1)	0.23593(7)	0.0346(5)	0.0634(6)	0.0664(6)	0.0029(3)	–0.0015(4)	–0.0146(4)

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