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Bis[(1,3-benzothiazol-2-ylsulfanyl)-acetato-κO]bis(imidazole-κN^3)copper(II)

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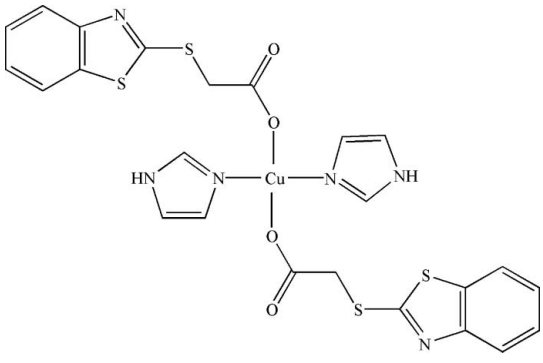
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.031; wR factor = 0.089; data-to-parameter ratio = 12.6.

In the title complex, $[Cu(C_9H_6NO_2S_2)_2(C_3H_4N_2)_2]$, the Cu^{II} atom, lying on an inversion centre, is coordinated by two O atoms and two N atoms in a geometry deviating slightly from square planar. Intermolecular hydrogen bonds link the complex molecules into a layer structure.

Related literature

For related literature, see: Tamura *et al.* (1987); Raptopoulu *et al.* (1998); Battaglia *et al.* (1983); Houser & Cheng (2005); Ying *et al.* (2004); Noro *et al.* (2005); Dobrzynska *et al.* (2002); Xu *et al.* (2005).



Experimental

Crystal data

$[Cu(C_9H_6NO_2S_2)_2(C_3H_4N_2)_2]$
M_r = 648.24
Monoclinic, $P2_1/c$
a = 10.086 (3) Å
b = 12.050 (3) Å
c = 10.521 (3) Å
β = 91.281 (3)°
V = 1278.4 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.23 mm^{-1}
T = 298 (2) K
0.31 × 0.23 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.702, T_{max} = 0.837
6476 measured reflections
2250 independent reflections
1768 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.089
S = 1.00
2250 reflections
178 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—N2	1.970 (2)	Cu1—O1	1.9961 (19)
N2^i—Cu1—N2	180	N2—Cu1—O1^i	89.70 (9)
N2^i—Cu1—O1^i	90.30 (9)	O1^i—Cu1—O1	180

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: CF2156).

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supplementary materials

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Bis[(1,3-benzothiazol-2-ylsulfanyl)acetato-κO]bis(imidazole-κN^3)copper(II)

H.-D. Bian, F.-P. Huang, Q. Yu and H. Liang

Comment

The recognition of strong antitumor activity of the *trans*-bis(acetato)bis(imidazole)copper(II) complex caused a growing interest in the synthesis and characterization of new compounds of this type (Tamura *et al.*, 1987; Raptopoulu *et al.*, 1998). We report here the synthesis and crystal stucture of a new copper(II) complex $[Cu(bttaa)_2(Him)_2]$ (where bttaa = 2-benzothiazolylthioacetate and Him = imidazole).

In the title complex, the Cu^{II} atom lying on an inversion centre, is coordinated by two imidazole nitrogen atoms and two carboxylate oxygen atoms. The Cu1···O2 separation of 2.767 Å indicates a weak interaction. Therefore, the title compound can be regarded as a pseudo-six-coordinate complex. This geometry around copper is typical of complexes of Cu^{II} with carboxylates and aromatic amines or imidazole (Battaglia *et al.*, 1983; Houser *et al.*, 2005; Ying *et al.*, 2004; Noro *et al.*, 2005). The length of Cu—O1 is in the normal range for a carboxylate group coordinated to copper in monodentate mode, and the Cu—N2 distance of 1.970 Å is similar to those observed for imidazole coordinated to copper(II) (Dobrzynska *et al.*, 2002; Xu *et al.*, 2005).

The supramolecular architecture is stabilized by an extensive 2-D network of intermolecular hydrogen bonds (N—H···O) involving imidazole N3 and bttaa O1 atoms.

Experimental

2-Benzothiazolylthioacetic acid (0.225 g, 1 mmol) and potassium hydroxide (0.06 g, 1 mmol) dissolved in water (10 ml) were added to a 1:1 methanol– water (10 ml) solution of $CuCl_2{\cdot}2H_2O$ (0.5 mmol). To this mixture was added a solution of imidazole (0.5 mmol) in methanol (4 ml). The blue solution was set aside for one week for the growth of blue block-shapped crystals.

Refinement

H atoms on C and N atoms were positoned geometrically and refined using a riding model (C—H = 0.93 Å for C—$H_{aromatic}$, C—H = 0.97 Å for C—$H_{aliphatic}$ and N—H = 0.86 Å) with $U_{iso}(H) = 1.2U_{eq}(C,N)$

Figures

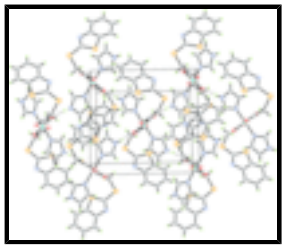


Fig. 1. The molecular structure of (I) with the atom-numbering scheme and 30% displacement ellipsoids (arbitrary spheres for the H atoms). Atoms with the suffix A are generated by the symmetry operation (−*x* + 1, −*y* + 2, −*z*).

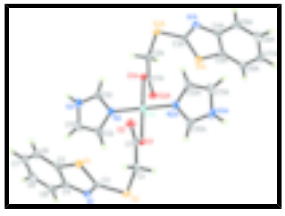


Fig. 2. The 2-D network structure of compound (I) (methylene H atoms are omitted for clarity). Hydrogen bonds are shown as dashed lines.

Bis[(1,3-benzothiazol-2-ylsulfanyl)acetato-κ*O*]bis(imidazole-κN^3)copper(II)

Crystal data

$[Cu(C_9H_6NO_2S_2)_2(C_3H_4N_2)_2]$	F_{000} = 662
M_r = 648.24	D_x = 1.684 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.086 (3) Å	Cell parameters from 2567 reflections
b = 12.050 (3) Å	θ = 2.6–27.6º
c = 10.521 (3) Å	μ = 1.23 mm^{-1}
β = 91.281 (3)º	T = 298 (2) K
V = 1278.4 (6) Å^3	Block, blue
Z = 2	0.31 × 0.23 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer	2250 independent reflections
Radiation source: fine-focus sealed tube	1768 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = −9→11
T_{min} = 0.702, T_{max} = 0.837	k = −12→14
6476 measured reflections	l = −11→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.089$

$S = 1.00$

2250 reflections

178 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.8447P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.43$ e Å^{-3}

$\Delta\rho_{min} = -0.26$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	0.02612 (16)
N1	0.1790 (2)	1.1037 (2)	0.4327 (2)	0.0350 (6)
N2	0.5031 (2)	0.9149 (2)	0.1598 (2)	0.0306 (6)
N3	0.5472 (3)	0.7866 (2)	0.3026 (3)	0.0429 (7)
H3	0.5779	0.7268	0.3370	0.052*
O1	0.35753 (19)	1.09765 (16)	0.06651 (17)	0.0297 (5)
O2	0.2287 (2)	0.97582 (18)	−0.0341 (2)	0.0438 (6)
S1	0.09591 (8)	0.95164 (7)	0.27425 (7)	0.0356 (2)
S2	0.12226 (8)	1.19910 (6)	0.21074 (7)	0.0355 (2)
C1	0.2436 (3)	1.0601 (2)	0.0281 (3)	0.0291 (6)
C2	0.1223 (3)	1.1293 (3)	0.0589 (3)	0.0340 (7)
H2A	0.0453	1.0811	0.0541	0.041*
H2B	0.1110	1.1850	−0.0071	0.041*
C3	0.1361 (3)	1.0878 (2)	0.3167 (3)	0.0312 (7)
C4	0.1860 (3)	1.0024 (3)	0.4970 (3)	0.0325 (7)
C5	0.1485 (3)	0.9090 (3)	0.4248 (3)	0.0310 (7)
C6	0.1636 (3)	0.8019 (3)	0.4724 (3)	0.0380 (8)
H6	0.1410	0.7404	0.4232	0.046*
C7	0.2133 (3)	0.7898 (3)	0.5949 (3)	0.0445 (8)
H7	0.2260	0.7190	0.6282	0.053*
C8	0.2446 (3)	0.8817 (3)	0.6688 (3)	0.0468 (9)
H8	0.2750	0.8714	0.7520	0.056*

C9	0.2317 (3)	0.9881 (3)	0.6217 (3)	0.0415 (8)
H9	0.2532	1.0491	0.6723	0.050*
C10	0.5633 (3)	0.8185 (3)	0.1828 (3)	0.0369 (7)
H10	0.6104	0.7787	0.1230	0.044*
C11	0.4737 (3)	0.8653 (3)	0.3607 (3)	0.0485 (9)
H11	0.4470	0.8649	0.4447	0.058*
C12	0.4469 (3)	0.9442 (3)	0.2732 (3)	0.0423 (8)
H12	0.3981	1.0084	0.2871	0.051*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0296 (3)	0.0238 (3)	0.0250 (3)	0.0048 (2)	0.0015 (2)	0.0009 (2)
N1	0.0394 (15)	0.0286 (14)	0.0373 (14)	0.0021 (11)	0.0031 (12)	−0.0042 (12)
N2	0.0298 (13)	0.0320 (14)	0.0301 (13)	0.0031 (11)	0.0013 (10)	0.0070 (11)
N3	0.0392 (16)	0.0440 (17)	0.0456 (16)	0.0033 (13)	0.0004 (13)	0.0229 (13)
O1	0.0316 (11)	0.0277 (11)	0.0298 (10)	0.0058 (9)	0.0004 (8)	−0.0015 (9)
O2	0.0503 (14)	0.0364 (13)	0.0447 (13)	−0.0035 (10)	0.0046 (11)	−0.0134 (10)
S1	0.0399 (5)	0.0299 (4)	0.0368 (4)	−0.0038 (3)	−0.0006 (3)	−0.0031 (3)
S2	0.0383 (4)	0.0258 (4)	0.0425 (5)	0.0060 (3)	0.0045 (3)	−0.0019 (3)
C1	0.0355 (17)	0.0263 (16)	0.0255 (14)	0.0018 (13)	0.0007 (13)	0.0054 (13)
C2	0.0333 (17)	0.0340 (18)	0.0347 (16)	0.0013 (13)	−0.0022 (13)	0.0009 (14)
C3	0.0278 (15)	0.0257 (16)	0.0404 (17)	0.0051 (12)	0.0096 (13)	−0.0008 (13)
C4	0.0288 (16)	0.0344 (17)	0.0346 (16)	0.0014 (13)	0.0070 (13)	−0.0023 (14)
C5	0.0253 (15)	0.0356 (17)	0.0324 (16)	−0.0024 (12)	0.0059 (12)	−0.0019 (13)
C6	0.0373 (18)	0.0326 (18)	0.0445 (19)	−0.0076 (14)	0.0066 (15)	0.0010 (15)
C7	0.044 (2)	0.040 (2)	0.049 (2)	−0.0029 (15)	0.0113 (16)	0.0151 (16)
C8	0.049 (2)	0.059 (2)	0.0330 (17)	−0.0030 (17)	0.0013 (15)	0.0056 (17)
C9	0.044 (2)	0.045 (2)	0.0355 (18)	−0.0025 (15)	0.0017 (14)	−0.0065 (15)
C10	0.0378 (18)	0.0353 (18)	0.0375 (17)	0.0039 (14)	0.0007 (14)	0.0069 (14)
C11	0.0365 (18)	0.071 (3)	0.0383 (18)	0.0105 (17)	0.0098 (15)	0.0213 (18)
C12	0.0363 (18)	0.051 (2)	0.0395 (18)	0.0130 (15)	0.0093 (14)	0.0111 (16)

Geometric parameters (Å, °)

Cu1—N2^{i}	1.970 (2)	C1—C2	1.521 (4)
Cu1—N2	1.970 (2)	C2—H2A	0.970
Cu1—O1^{i}	1.9961 (19)	C2—H2B	0.970
Cu1—O1	1.9961 (19)	C4—C9	1.391 (4)
N1—C3	1.300 (4)	C4—C5	1.404 (4)
N1—C4	1.397 (4)	C5—C6	1.392 (4)
N2—C10	1.330 (4)	C6—C7	1.380 (4)
N2—C12	1.378 (4)	C6—H6	0.930
N3—C10	1.332 (4)	C7—C8	1.385 (5)
N3—C11	1.358 (4)	C7—H7	0.930
N3—H3	0.860	C8—C9	1.380 (5)
O1—C1	1.291 (3)	C8—H8	0.930
O2—C1	1.216 (3)	C9—H9	0.930

S1—C5	1.737 (3)	C10—H10	0.930
S1—C3	1.746 (3)	C11—C12	1.346 (4)
S2—C3	1.748 (3)	C11—H11	0.930
S2—C2	1.806 (3)	C12—H12	0.930
$N2^{i}$—Cu1—N2	180.0	C9—C4—N1	125.2 (3)
$N2^{i}$—Cu1—$O1^{i}$	90.30 (9)	C9—C4—C5	119.4 (3)
N2—Cu1—$O1^{i}$	89.70 (9)	N1—C4—C5	115.3 (3)
$N2^{i}$—Cu1—O1	89.70 (9)	C6—C5—C4	121.5 (3)
N2—Cu1—O1	90.30 (9)	C6—C5—S1	129.1 (2)
$O1^{i}$—Cu1—O1	180.0	C4—C5—S1	109.3 (2)
C3—N1—C4	109.8 (2)	C7—C6—C5	117.9 (3)
C10—N2—C12	105.2 (2)	C7—C6—H6	121.0
C10—N2—Cu1	127.4 (2)	C5—C6—H6	121.0
C12—N2—Cu1	127.4 (2)	C6—C7—C8	120.9 (3)
C10—N3—C11	107.7 (3)	C6—C7—H7	119.6
C10—N3—H3	126.2	C8—C7—H7	119.6
C11—N3—H3	126.2	C9—C8—C7	121.5 (3)
C1—O1—Cu1	109.05 (17)	C9—C8—H8	119.2
C5—S1—C3	88.84 (14)	C7—C8—H8	119.2
C3—S2—C2	101.87 (14)	C8—C9—C4	118.7 (3)
O2—C1—O1	124.0 (3)	C8—C9—H9	120.7
O2—C1—C2	118.8 (3)	C4—C9—H9	120.7
O1—C1—C2	117.1 (3)	N2—C10—N3	111.0 (3)
C1—C2—S2	117.4 (2)	N2—C10—H10	124.5
C1—C2—H2A	108.0	N3—C10—H10	124.5
S2—C2—H2A	108.0	C12—C11—N3	106.8 (3)
C1—C2—H2B	108.0	C12—C11—H11	126.6
S2—C2—H2B	108.0	N3—C11—H11	126.6
H2A—C2—H2B	107.2	C11—C12—N2	109.3 (3)
N1—C3—S1	116.7 (2)	C11—C12—H12	125.3
N1—C3—S2	120.4 (2)	N2—C12—H12	125.3
S1—C3—S2	122.89 (18)		
$N2^{i}$—Cu1—N2—C10	35 (100)	C3—N1—C4—C5	0.8 (4)
$O1^{i}$—Cu1—N2—C10	−11.8 (3)	C9—C4—C5—C6	−4.0 (4)
O1—Cu1—N2—C10	168.2 (3)	N1—C4—C5—C6	173.3 (3)
$N2^{i}$—Cu1—N2—C12	−148 (100)	C9—C4—C5—S1	179.9 (2)
$O1^{i}$—Cu1—N2—C12	165.9 (3)	N1—C4—C5—S1	−2.8 (3)
O1—Cu1—N2—C12	−14.1 (3)	C3—S1—C5—C6	−172.7 (3)
$N2^{i}$—Cu1—O1—C1	85.13 (18)	C3—S1—C5—C4	2.9 (2)
N2—Cu1—O1—C1	−94.87 (18)	C4—C5—C6—C7	1.9 (4)
$O1^{i}$—Cu1—O1—C1	−55 (100)	S1—C5—C6—C7	177.0 (2)
Cu1—O1—C1—O2	2.9 (3)	C5—C6—C7—C8	1.3 (5)
Cu1—O1—C1—C2	−174.60 (19)	C6—C7—C8—C9	−2.3 (5)
O2—C1—C2—S2	145.5 (2)	C7—C8—C9—C4	0.1 (5)
O1—C1—C2—S2	−36.9 (3)	N1—C4—C9—C8	−174.0 (3)
C3—S2—C2—C1	−60.2 (3)	C5—C4—C9—C8	3.0 (5)

C4—N1—C3—S1	1.6 (3)	C12—N2—C10—N3	0.3 (4)
C4—N1—C3—S2	−177.7 (2)	Cu1—N2—C10—N3	178.4 (2)
C5—S1—C3—N1	−2.8 (2)	C11—N3—C10—N2	−0.1 (4)
C5—S1—C3—S2	176.57 (19)	C10—N3—C11—C12	−0.1 (4)
C2—S2—C3—N1	159.1 (2)	N3—C11—C12—N2	0.3 (4)
C2—S2—C3—S1	−20.2 (2)	C10—N2—C12—C11	−0.3 (4)
C3—N1—C4—C9	177.9 (3)	Cu1—N2—C12—C11	−178.4 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O1^{ii}	0.86	1.96	2.819	174

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

Fig. 1

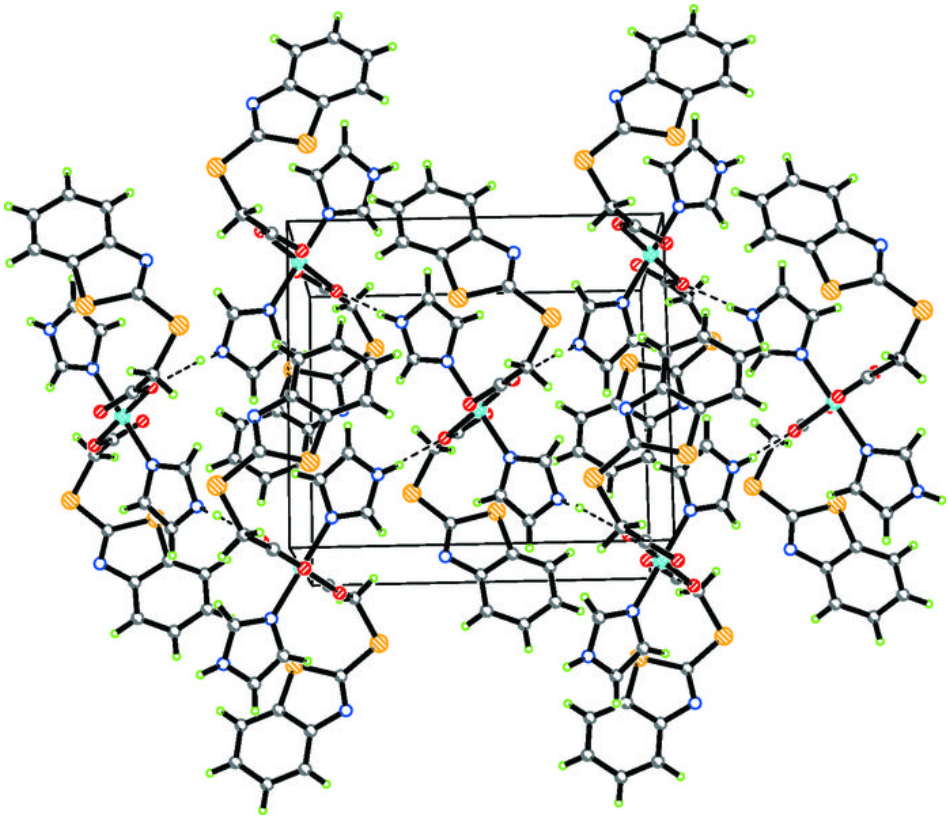
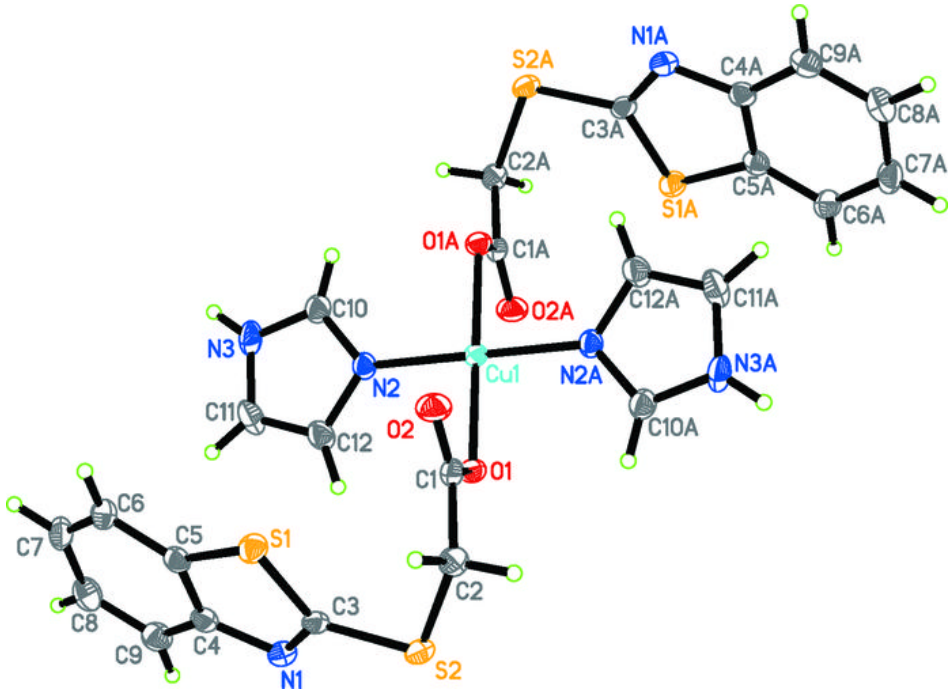


Fig. 2


N1A
S2A
C4A
C9A
C3A
C8A
C2A
C7A
C5A
S1A
C6A
O1A
C1A
C12A
C11A
C10
O2A
N3
N2
Cu1
N2A
N3A
C11
C12
O2
C10A
C1
O1
C6
C7
C5
S1
C2
C3
C8
C9
C4
N1
S2