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Tris(N,N'-dibutylthiourea-κS)iodidocopper(I) 0.6-hydrate

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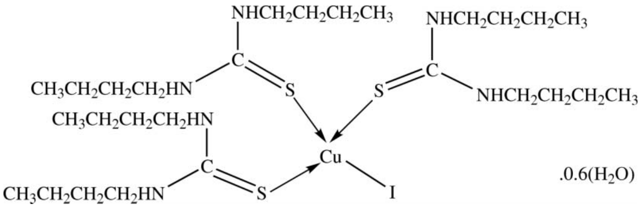
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.009 Å; H-atom completeness 99%; disorder in solvent or counterion; R factor = 0.062; wR factor = 0.173; data-to-parameter ratio = 30.3.

In the title complex, $[\mathrm{CuI}(\mathrm{C_9H_{20}N_2S})_3]\cdot0.6\mathrm{H_2O}$, the Cu$^{\mathrm{I}}$ and I atoms lie on a threefold rotation axis. The partially occupied water molecule is disordered across an inversion centre. Each Cu$^{\mathrm{I}}$ centre binds to the S atoms of three N,N'-dibutylthiourea ligands and I$^-$ in a distorted tetrahedral environment. N—H$\cdots$I and C—H$\cdots$S interactions are observed in the complex molecule. Intermolecular N—H$\cdots$S hydrogen bonds link the molecules into a two-dimensional network parallel to the ab plane.

Related literature

For related literature on values of bond lengths, see: Allen *et al.* (1987). For related structures, see: Bombicz *et al.* (2004); Lobana *et al.* (2006); Mufakkar *et al.* (2007). For related literature on the coordination chemistry of copper, see: Atkinson *et al.* (1985); Bombicz *et al.* (2004); Dubler & Bensch (1986); Eller *et al.* (1977); Girling & Amma (1971); Griffith *et al.* (1976); Hunt *et al.* (1979); Kaim & Schwederski (1994); Lobana *et al.* (2006); Stocker *et al.* (1996).



Experimental

Crystal data

$[\mathrm{CuI}(\mathrm{C_9H_{20}N_2S})_3]\cdot0.6(\mathrm{H_2O})$
M_r = 766.24
Trigonal, $P\bar{3}$
a = 13.5514 (3) Å
c = 11.4588 (6) Å
V = 1822.38 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.65 mm^{-1}
T = 100.0 (1) K
0.48 × 0.27 × 0.10 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{\min}$ = 0.507, $T_{\max}$ = 0.851
21130 measured reflections
3632 independent reflections
2454 reflections with $I > 2\sigma(I)$
R_{int} = 0.090

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.173
S = 1.05
3632 reflections
120 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 2.54 e Å^{-3}
$\Delta\rho_{\min}$ = −1.13 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

I1—Cu1	2.6251 (10)	Cu1—S1	2.3543 (12)
S1—Cu1—S1$^{\mathrm{i}}$	99.32 (4)	S1—Cu1—I1	118.34 (3)

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$S1$^{\mathrm{ii}}$	0.86	2.57	3.347 (5)	151
N2—H2$\cdots$I1	0.86	2.89	3.735 (4)	167
C2—H2$A\cdots$S1	0.97	2.65	3.114 (7)	110

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2443).

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Acta Cryst. (2007). E**63**, m2550–m2551 Khan *et al.* • [CuI$(C_9H_{20}N_2S)_3$]·0.6(H_2O) **m2551**

supplementary materials

Acta Cryst. (2007). E**63**, m2550-m2551 [**doi:10.1107/S1600536807044649**]

Tris(*N*,*N*'-dibutylthiourea-*κS*)iodidocopper(I) 0.6-hydrate

I. U. Khan, M. Mufakkar, S. Ahmad, H.-K. Fun and **S. Chantrapromma**

Comment

Crystal structures of several copper(I) complexes of thiourea and its derivatives have been reported (Eller *et al.*, 1977; Stocker *et al.*, 1996; Girling & Amma, 1971; Atkinson *et al.*, 1985; Dubler & Bensch, 1986). They show that a wide variety of stoichiometries and structural diversity exist for these complexes, with the copper to sulfur ratio ranging from 1:4 as in monomeric $[Cu(tu)_4(SiF_6)_{0.5}]$ (Hunt *et al.*, 1979) to 1:1.5 as in tetrameric $[Cu_4(tu)_6(NO_3)_4]·4H_2O$ (Griffith *et al.*, 1976). We are interested in understanding the correlation of the geometries of such complexes with the nature of substituents on thiourea and the strength and size of the other coordinating ligands. Previously, we have reported the crystal structure of tetrakis(*N*-methylthiourea-*κS*)copper(I) iodide (Mufakkar *et al.*, 2007). Now we report here the crystal structure of the title complex.

In the molecule of the title complex, atoms Cu1 and I1 lie on a threefold rotation axis and the asymmetric unit therefore contains one third of the complex molecule (Fig. 1). The asymmetric unit also contains a partially occupied water molecule, disordered across a inversion center. The coordination of Cu1 is a distorted tetrahedron, being coordinated by the S atoms of the three *N*,*N*'-dibutylthiourea ligands, with S—Cu—S angles of 99.32 (4)° (Table 1). These angles are smaller than those observed in related structures (Bombicz *et al.*, 2004; Lobana *et al.*, 2006; Mufakkar *et al.*, 2007). However, the Cu—S bond distances [2.3543 (12) Å] of the title complex lie within the range of those found in the Cu^I complexes with tetrahedral geometry (Bombicz *et al.*, 2004; Lobana *et al.*, 2006; Mufakkar *et al.*, 2007). The fourth coordination of Cu^I is occupied by the I1 atom with a Cu1—I1 distance of 2.6251 (10) Å and S—Cu—I angle of 118.34 (3)°. The dihedral angle between the mean planes of S1/N1/N2/C1/C2/C6/C7 and C2/C3/C4/C5 is 80.2 (6)°. All other bond lengths and angles are in normal ranges (Allen *et al.*, 1987).

The I atom is involved in an intramolecular N—H···I hydrogen bond and the S atoms form weak C—H···S intramolecular interactions (Fig. 1). Intermolecular N—H···S hydrogen bonds stabilize the crystal structure (Table 2). In the crystal packing (Fig. 2), the molecules are arranged into a two-dimensional network parallel to the *ab* plane.

Experimental

To a solution of copper(I) iodide (0.19 g, 1.0 mmol) in acetonitrile (15 ml) was added 2 molar equivalents of *N*,*N*'-dibutylthiourea in acetonitrile (10 ml). The mixture was stirred for half an hour. A clear solution was obtained. The solution was concentrated by slow evaporation at room temperature to yield colourless single crystals of the title compound suitable for *x*-ray stucture determination after several days.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H = 0.86 Å and C—H = 0.96 or 0.97 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The H atoms of the partially occupied disordered water molecule could not be located. The highest residual peak is located 1.03 Å from I1 and the deepest hole is located 0.80 Å from I1.

Figures

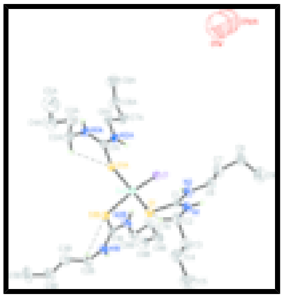

Fig. 1. The structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. For clarity, only N-bound H atoms and those involving in weak C—H···S interactions (dashed lines) are shown. Symmetry codes: (i) $-x+y+1$, $-x+1$, z; (ii) $-y+1$, $x-y$, z.

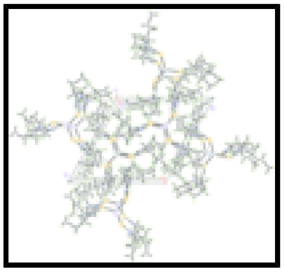

Fig. 2. The crystal packing of the title compound, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines.

Iodidotris(*N*,*N*'-dibutylthiourea-*κS*)copper(I) 0.6-hydrate

Crystal data

$[Cu(C_9H_{20}N_2S)_3I]\cdot 0.6(H_2O)$	$Z = 2$
$M_r = 766.24$	$F_{000} = 800$
Trigonal, $P\overline{3}$	$D_x = 1.396$ Mg m^{-3}
Hall symbol: -P 3	Mo *K*α radiation λ = 0.71073 Å
$a = 13.5514$ (3) Å	Cell parameters from 3632 reflections
$b = 13.5514$ (3) Å	θ = 1.7–30.4°
$c = 11.4588$ (6) Å	μ = 1.65 mm^{-1}
α = 90°	$T = 100.0$ (1) K
β = 90°	Plate, colourless
γ = 120°	0.48 × 0.27 × 0.10 mm
$V = 1822.38$ (11) Å^3	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	3632 independent reflections
Radiation source: fine-focus sealed tube	2454 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.090$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 30.4°$

$T = 100.0(1)$ K	$\theta_{\min} = 1.7°$
ω scans	$h = -19 \rightarrow 18$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -18 \rightarrow 18$
$T_{\min} = 0.508$, $T_{\max} = 0.851$	$l = -15 \rightarrow 15$
21130 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.062$	H-atom parameters constrained
$wR(F^2) = 0.173$	$w = 1/[\sigma^2(F_o^2) + (0.0894P)^2 + 1.4942P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\max} = 0.001$
3632 reflections	$\Delta\rho_{\max} = 2.54$ e Å^{-3}
120 parameters	$\Delta\rho_{\min} = -1.13$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The low-temparture data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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	$U_{\rm iso}$*/$U_{\rm eq}$	Occ. (<1)
I1	0.6667	0.3333	0.71844 (4)	0.02975 (18)	
Cu1	0.6667	0.3333	0.94753 (8)	0.0274 (2)	
S1	0.70138 (9)	0.50061 (9)	1.04506 (9)	0.0267 (3)	
N1	0.6251 (4)	0.6474 (3)	1.0253 (3)	0.0312 (9)	
H1	0.6035	0.6851	0.9824	0.037*	
N2	0.6362 (4)	0.5666 (3)	0.8559 (3)	0.0317 (9)	
H2	0.6547	0.5227	0.8197	0.038*	
C1	0.6508 (4)	0.5764 (4)	0.9706 (4)	0.0247 (9)	
C2	0.6302 (5)	0.6675 (5)	1.1508 (4)	0.0433 (13)	
H2A	0.6334	0.6061	1.1909	0.052*	
H2B	0.5608	0.6659	1.1751	0.052*	

C3	0.7295 (5)	0.7776 (5)	1.1870 (5)	0.0484 (14)	
H3A	0.7337	0.8383	1.1386	0.058*	
H3B	0.7990	0.7748	1.1755	0.058*	
C4	0.7200 (6)	0.8035 (6)	1.3162 (5)	0.0582 (17)	
H4A	0.6477	0.8005	1.3286	0.070*	
H4B	0.7211	0.7454	1.3647	0.070*	
C5	0.8158 (7)	0.9192 (7)	1.3530 (6)	0.077 (2)	
H5A	0.8021	0.9356	1.4309	0.115*	
H5B	0.8196	0.9763	1.3006	0.115*	
H5C	0.8866	0.9192	1.3509	0.115*	
C6	0.5907 (5)	0.6256 (4)	0.7868 (4)	0.0337 (11)	
H6A	0.6413	0.7072	0.7937	0.040*	
H6B	0.5170	0.6078	0.8179	0.040*	
C7	0.5777 (6)	0.5925 (5)	0.6594 (5)	0.0478 (14)	
H7A	0.5342	0.5101	0.6527	0.057*	
H7B	0.6524	0.6182	0.6258	0.057*	
C8	0.5178 (7)	0.6442 (5)	0.5911 (5)	0.0553 (16)	
H8A	0.5101	0.6196	0.5105	0.066*	
H8B	0.4417	0.6148	0.6226	0.066*	
C9	0.5787 (7)	0.7727 (6)	0.5941 (6)	0.071 (2)	
H9A	0.5432	0.7995	0.5401	0.106*	
H9B	0.6571	0.8026	0.5728	0.106*	
H9C	0.5746	0.7977	0.6714	0.106*	
O1W	0.0000	0.0000	0.042 (7)	0.31 (4)	0.60

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0312 (2)	0.0312 (2)	0.0267 (3)	0.01562 (11)	0.000	0.000
Cu1	0.0270 (3)	0.0270 (3)	0.0283 (5)	0.01350 (17)	0.000	0.000
S1	0.0267 (6)	0.0264 (6)	0.0275 (5)	0.0137 (5)	−0.0003 (4)	−0.0003 (4)
N1	0.041 (2)	0.033 (2)	0.0266 (19)	0.0237 (19)	−0.0042 (16)	−0.0031 (16)
N2	0.044 (2)	0.034 (2)	0.0259 (19)	0.026 (2)	0.0012 (17)	−0.0026 (16)
C1	0.020 (2)	0.024 (2)	0.028 (2)	0.0097 (17)	0.0004 (16)	0.0002 (17)
C2	0.060 (4)	0.044 (3)	0.035 (3)	0.032 (3)	−0.011 (2)	−0.010 (2)
C3	0.053 (4)	0.043 (3)	0.050 (3)	0.025 (3)	−0.004 (3)	0.003 (3)
C4	0.066 (4)	0.057 (4)	0.042 (3)	0.024 (3)	−0.006 (3)	−0.009 (3)
C5	0.085 (6)	0.075 (5)	0.049 (4)	0.024 (4)	−0.020 (4)	−0.022 (4)
C6	0.047 (3)	0.034 (3)	0.027 (2)	0.026 (2)	−0.001 (2)	0.0024 (19)
C7	0.073 (4)	0.051 (3)	0.035 (3)	0.042 (3)	−0.004 (3)	−0.004 (2)
C8	0.084 (5)	0.057 (4)	0.034 (3)	0.042 (4)	−0.016 (3)	−0.005 (3)
C9	0.095 (6)	0.067 (5)	0.058 (4)	0.047 (5)	−0.003 (4)	0.006 (3)
O1W	0.30 (3)	0.30 (3)	0.32 (13)	0.149 (15)	0.000	0.000

Geometric parameters (Å, °)

I1—Cu1	2.6251 (10)	C4—H4A	0.97
Cu1—S1	2.3543 (12)	C4—H4B	0.97

Cu1—S1^{i}	2.3543 (12)	C5—H5A	0.96
Cu1—S1^{ii}	2.3543 (12)	C5—H5B	0.96
S1—C1	1.720 (4)	C5—H5C	0.96
N1—C1	1.331 (5)	C6—C7	1.513 (7)
N1—C2	1.460 (6)	C6—H6A	0.97
N1—H1	0.86	C6—H6B	0.97
N2—C1	1.326 (6)	C7—C8	1.527 (8)
N2—C6	1.462 (6)	C7—H7A	0.97
N2—H2	0.86	C7—H7B	0.97
C2—C3	1.484 (8)	C8—C9	1.509 (9)
C2—H2A	0.97	C8—H8A	0.97
C2—H2B	0.97	C8—H8B	0.97
C3—C4	1.543 (8)	C9—H9A	0.96
C3—H3A	0.97	C9—H9B	0.96
C3—H3B	0.97	C9—H9C	0.96
C4—C5	1.512 (9)		

S1—Cu1—S1^{i}	99.32 (4)	C3—C4—H4B	109.2
S1—Cu1—S1^{ii}	99.32 (4)	H4A—C4—H4B	107.9
S1^{i}—Cu1—S1^{ii}	99.32 (4)	C4—C5—H5A	109.5
S1—Cu1—I1	118.34 (3)	C4—C5—H5B	109.5
S1^{i}—Cu1—I1	118.34 (3)	H5A—C5—H5B	109.5
S1^{ii}—Cu1—I1	118.34 (3)	C4—C5—H5C	109.5
C1—S1—Cu1	113.09 (15)	H5A—C5—H5C	109.5
C1—N1—C2	126.5 (4)	H5B—C5—H5C	109.5
C1—N1—H1	116.8	N2—C6—C7	112.2 (4)
C2—N1—H1	116.8	N2—C6—H6A	109.2
C1—N2—C6	124.4 (4)	C7—C6—H6A	109.2
C1—N2—H2	117.8	N2—C6—H6B	109.2
C6—N2—H2	117.8	C7—C6—H6B	109.2
N2—C1—N1	117.3 (4)	H6A—C6—H6B	107.9
N2—C1—S1	121.1 (3)	C6—C7—C8	111.6 (5)
N1—C1—S1	121.6 (3)	C6—C7—H7A	109.3
N1—C2—C3	113.4 (5)	C8—C7—H7A	109.3
N1—C2—H2A	108.9	C6—C7—H7B	109.3
C3—C2—H2A	108.9	C8—C7—H7B	109.3
N1—C2—H2B	108.9	H7A—C7—H7B	108.0
C3—C2—H2B	108.9	C9—C8—C7	114.1 (6)
H2A—C2—H2B	107.7	C9—C8—H8A	108.7
C2—C3—C4	111.1 (5)	C7—C8—H8A	108.7
C2—C3—H3A	109.4	C9—C8—H8B	108.7
C4—C3—H3A	109.4	C7—C8—H8B	108.7
C2—C3—H3B	109.4	H8A—C8—H8B	107.6
C4—C3—H3B	109.4	C8—C9—H9A	109.5
H3A—C3—H3B	108.0	C8—C9—H9B	109.5
C5—C4—C3	112.2 (6)	H9A—C9—H9B	109.5
C5—C4—H4A	109.2	C8—C9—H9C	109.5
C3—C4—H4A	109.2	H9A—C9—H9C	109.5

C5—C4—H4B	109.2	H9B—C9—H9C	109.5
S1^{i}—Cu1—S1—C1	162.01 (17)	Cu1—S1—C1—N1	155.8 (3)
S1^{ii}—Cu1—S1—C1	−96.85 (17)	C1—N1—C2—C3	104.8 (6)
I1—Cu1—S1—C1	32.58 (17)	N1—C2—C3—C4	170.6 (5)
C6—N2—C1—N1	−2.0 (7)	C2—C3—C4—C5	−175.9 (6)
C6—N2—C1—S1	178.3 (4)	C1—N2—C6—C7	−176.8 (5)
C2—N1—C1—N2	177.3 (5)	N2—C6—C7—C8	173.5 (5)
C2—N1—C1—S1	−3.0 (7)	C6—C7—C8—C9	59.6 (8)
Cu1—S1—C1—N2	−24.4 (4)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{iii}	0.86	2.57	3.347 (5)	151
N2—H2···I1	0.86	2.89	3.735 (4)	167
C2—H2A···S1	0.97	2.65	3.114 (7)	110

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

Fig. 1

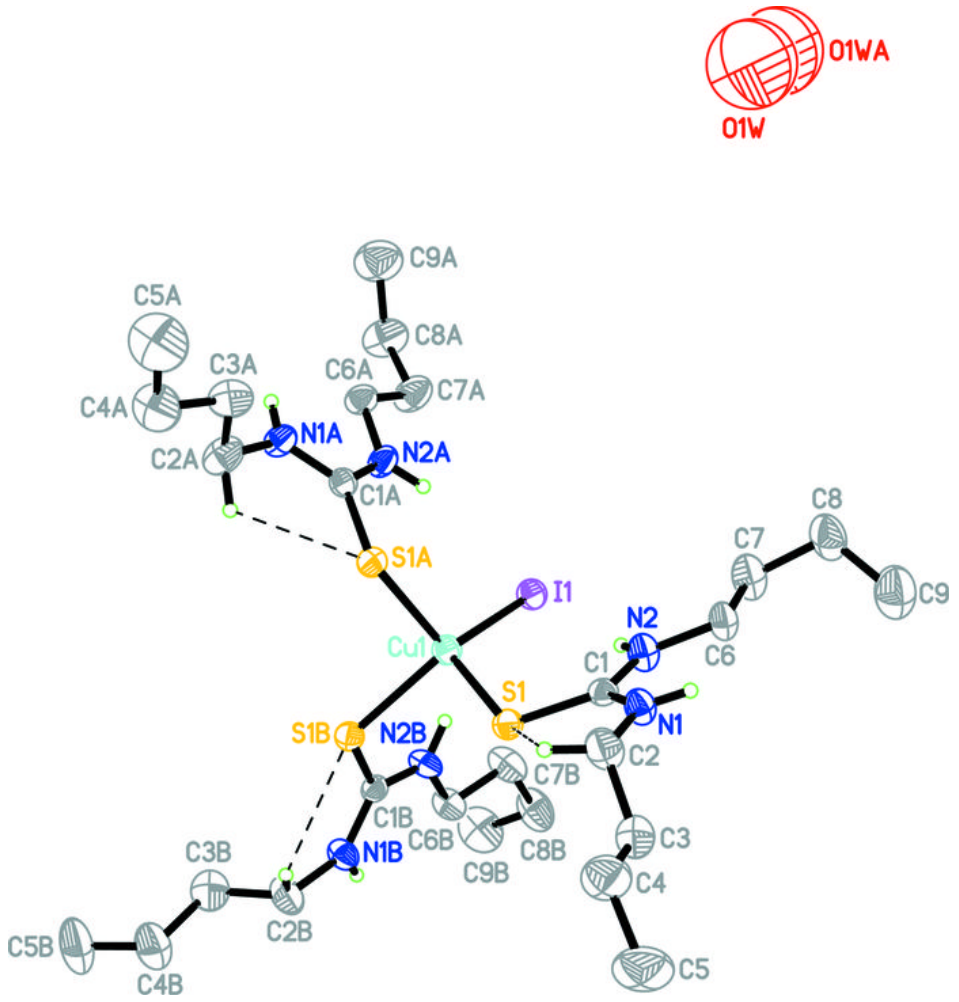
O1WA
O1W
C9A
C5A
C8A
C3A
C6A
C7A
C4A
N1A
N2A
C2A
C1A
C8
C7
S1A
I1
C9
N2
Cu1
C1
C6
S1
N1
S1B
N2B
C2
C7B
C1B
C6B
C8B
C3
C3B
N1B
C9B
C4
C5B
C2B
C4B
C5

Fig. 2