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Poly[[tris[μ-2,2′-(butane-1,4-diyldithio)-bis(1,3,4-thiadiazole)-$\kappa^2N^4:N^{4'}$]-copper(II)] bis(perchlorate)]

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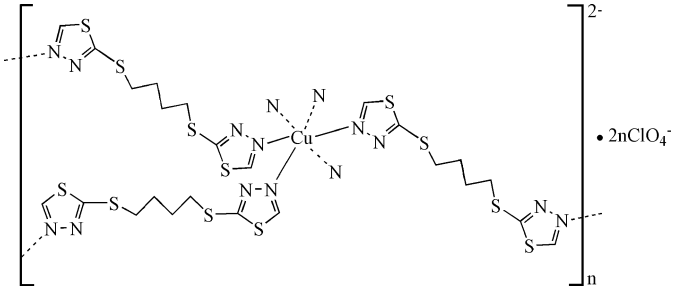
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.007 Å; R factor = 0.047; wR factor = 0.133; data-to-parameter ratio = 18.6.

In the title compound, $\{[Cu(C_8H_{10}N_4S_4)_3](ClO_4)_2\}_n$, the Cu^{II} atom is located on a threefold inversion axis coordinated by six N atoms of symmetry-equivalent 2,2′-(butane-1,4-diyldithio)bis(1,3,4-thiadiazole) ligands in a slightly distorted octahedral geometry. Adjacent Cu^{II} atoms are linked by the bridging bidentate thiadiazole ligands, which are situated about inversion centers. This leads to the formation of a three-dimensional network structure.

Related literature

For copper(II) complexes involving the same ligand, see: Huang *et al.* (2009); Wang *et al.* (2008).



Experimental

Crystal data

$[Cu(C_8H_{10}N_4S_4)_3](ClO_4)_2$
M_r = 1133.76
Trigonal, $R\overline{3}$
a = 10.5455 (6) Å
c = 33.728 (4) Å
V = 3248.3 (5) Å^3
Z = 3
Mo $K\alpha$ radiation
μ = 1.27 mm^{-1}
T = 291 K
0.28 × 0.21 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.717, T_{max} = 0.839
9432 measured reflections
1673 independent reflections
1320 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.133
S = 1.05
1673 reflections
90 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: SU2097).

References

Bruker (1997). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Huang, H.-M., Ju, F.-Y., Wang, J.-G. & Qin, J.-H. (2009). *Acta Cryst.* E**65**, m80–m81.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, J. G., Qin, J. H., Hu, P. Z. & Zhao, B. T. (2008). *Z. Kristallogr. New Cryst. Struct.* **223**, 225–227.

supporting information

Acta Cryst. (2009). E**65**, m307 [doi:10.1107/S1600536809005625]

Poly[[tris[μ-2,2′-(butane-1,4-diyldithio)bis(1,3,4-thiadiazole)-$\kappa^2N^4:N^{4'}$]copper(II)] bis(perchlorate)]

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S1. Comment

The asymmetric unit of the title compound consists of one sixth of a Cu^{II} atom, which is located on a three-fold inversion axis, half a 2,2′-(butane-1,4-diyldithio)bis(1,3,4-thiadiazole) ligand which possesses an inversion center, and one third of a perchlorate ion, which is situated on a three-fold rotation axis. As depicted in Fig. 1, the Cu^{II} atom is coordinated by six N atoms from six symmetry equivalent 2,2′-(butane-1,4-diyldithio)bis(1,3,4-thiadiazole) ligands, in a slightly distorted octahedral geometry of the central atom. The Cu—N bond distance is 2.149 (3) Å, within the range expected for such coordination bonds (Huang *et al.*, 2009; Wang *et al.*, 2008). The centrosymmetric 2,2′-(butane-1,4-diyldithio)bis(1,3,4-thiadiazole) ligand adopts a N,N′-bidentate bridging mode in a trans configuration and links the Cu^{II} atoms to form a three-dimensional network. The bridging Cu···Cu distance is 12.7854 (12) Å (Fig. 2).

S2. Experimental

The reaction of 2,2′-(butane-1,4-diyldithio)bis(1,3,4-thiadiazole) (0.3 mmol) with $Cu(ClO_4)_2$ (0.1 mmol) in MeOH(10 ml) for a few minutes gave a light blue solid, which was filtered off, washed with acetone, and dried in air. Single crystals, suitable for X-ray analysis, were obtained by slow diffusion of Et_2O into an acetonitrile solution of the solid.

S3. Refinement

The H-atoms were positioned geometrically and treated as riding: C—H = 0.93 - 0.97 Å and $U_{iso}(H) = 1.2U_{eq}$(parent C-atom).

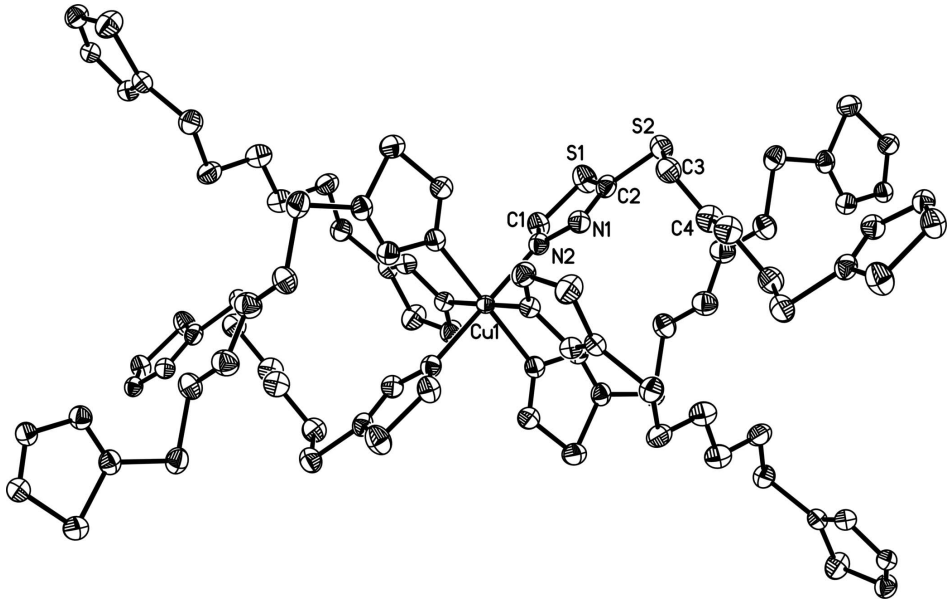

Figure 1

A view of the coordination around the Cu^{II} atom in the cation of the title compound. Displacement ellipsoids are drawn at the 30% probability level. The H atoms and perchlorate ion were omitted for clarity.

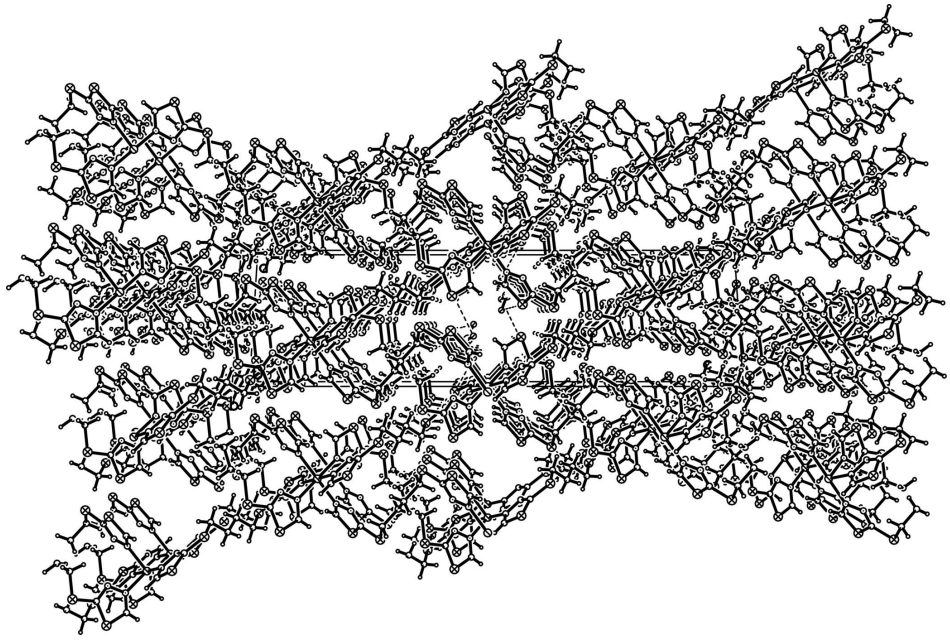

Figure 2

A view down the *b* axis of the crystal packing of the title compound.

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V = 3248.3 (5) Å^3
Z = 3
F(000) = 1731
D_x = 1.739 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2638 reflections

θ = 2.3–24.5°
μ = 1.27 mm^{-1}
T = 291 K
Block, blue
0.28 × 0.21 × 0.14 mm

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Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.717, T_{max} = 0.839

9432 measured reflections
1673 independent reflections
1320 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°, θ_{min} = 2.3°
h = −13→13
k = −13→13
l = −43→43

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.133
S = 1.05
1673 reflections
90 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2 + 10.9603P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
Cu1	1.0000	1.0000	0.5000	0.0338 (2)
Cl1	0.3333	0.6667	0.45984 (7)	0.0692 (5)
S1	0.94592 (14)	0.59055 (13)	0.43361 (3)	0.0702 (4)
S2	0.71904 (11)	0.56831 (12)	0.37580 (3)	0.0608 (3)
O1	0.3599 (4)	0.8063 (4)	0.47130 (14)	0.1084 (13)
O2	0.3333	0.6667	0.4165 (2)	0.124 (3)
N1	0.8519 (3)	0.7722 (3)	0.43379 (8)	0.0500 (7)
N2	0.9537 (3)	0.8133 (3)	0.46412 (8)	0.0461 (6)
C1	1.0099 (4)	0.7300 (4)	0.46704 (11)	0.0578 (9)
H1	1.0802	0.7448	0.4860	0.069*
C2	0.8366 (4)	0.6570 (4)	0.41560 (10)	0.0501 (8)
C3	0.6329 (4)	0.6782 (5)	0.36973 (12)	0.0615 (9)
H3A	0.5323	0.6158	0.3613	0.074*
H3B	0.6313	0.7208	0.3951	0.074*
C4	0.7119 (5)	0.8003 (5)	0.33947 (13)	0.0679 (11)
H4A	0.8032	0.8758	0.3508	0.082*
H4B	0.7351	0.7613	0.3163	0.082*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0332 (3)	0.0332 (3)	0.0350 (4)	0.01660 (15)	0.000	0.000
Cl1	0.0537 (6)	0.0537 (6)	0.1003 (14)	0.0269 (3)	0.000	0.000
S1	0.0854 (8)	0.0764 (7)	0.0679 (7)	0.0548 (6)	−0.0153 (5)	−0.0153 (5)
S2	0.0585 (6)	0.0671 (6)	0.0537 (6)	0.0292 (5)	−0.0048 (4)	−0.0096 (4)
O1	0.101 (3)	0.073 (2)	0.152 (4)	0.045 (2)	0.015 (3)	−0.023 (2)
O2	0.140 (4)	0.140 (4)	0.090 (5)	0.070 (2)	0.000	0.000
N1	0.0441 (15)	0.0547 (17)	0.0504 (16)	0.0242 (13)	−0.0014 (12)	−0.0007 (13)
N2	0.0424 (14)	0.0526 (16)	0.0430 (14)	0.0237 (12)	0.0009 (11)	0.0021 (12)
C1	0.063 (2)	0.067 (2)	0.053 (2)	0.040 (2)	−0.0060 (17)	−0.0057 (17)
C2	0.0449 (17)	0.059 (2)	0.0434 (17)	0.0241 (16)	0.0067 (13)	0.0052 (15)
C3	0.051 (2)	0.068 (2)	0.062 (2)	0.0271 (19)	−0.0115 (17)	−0.0019 (19)
C4	0.060 (2)	0.073 (3)	0.075 (3)	0.036 (2)	−0.004 (2)	−0.001 (2)

Geometric parameters (Å, º)

Cu1—N2^{i}	2.149 (3)	S2—C2	1.748 (4)
Cu1—N2	2.149 (3)	S2—C3	1.807 (4)
Cu1—N2^{ii}	2.149 (3)	N1—C2	1.298 (4)
Cu1—N2^{iii}	2.149 (3)	N1—N2	1.386 (4)
Cu1—N2^{iv}	2.149 (3)	N2—C1	1.286 (4)
Cu1—N2^{v}	2.149 (3)	C1—H1	0.9300
Cl1—O1	1.409 (4)	C3—C4	1.523 (6)
Cl1—O1^{vi}	1.409 (4)	C3—H3A	0.9700
Cl1—O1^{vii}	1.409 (4)	C3—H3B	0.9700
Cl1—O2	1.463 (8)	C4—C4^{viii}	1.494 (8)
S1—C1	1.702 (4)	C4—H4A	0.9700
S1—C2	1.731 (4)	C4—H4B	0.9700
N2^{i}—Cu1—N2	91.39 (10)	C2—N1—N2	110.8 (3)
N2^{i}—Cu1—N2^{ii}	91.40 (10)	C1—N2—N1	113.1 (3)
N2—Cu1—N2^{ii}	91.39 (10)	C1—N2—Cu1	127.7 (2)
N2^{i}—Cu1—N2^{iii}	88.61 (10)	N1—N2—Cu1	119.2 (2)
N2—Cu1—N2^{iii}	88.61 (10)	N2—C1—S1	114.9 (3)
N2^{ii}—Cu1—N2^{iii}	179.998 (1)	N2—C1—H1	122.6
N2^{i}—Cu1—N2^{iv}	88.61 (10)	S1—C1—H1	122.6
N2—Cu1—N2^{iv}	179.999 (2)	N1—C2—S1	114.7 (3)
N2^{ii}—Cu1—N2^{iv}	88.61 (10)	N1—C2—S2	125.9 (3)
N2^{iii}—Cu1—N2^{iv}	91.39 (10)	S1—C2—S2	119.4 (2)
N2^{i}—Cu1—N2^{v}	179.999 (1)	C4—C3—S2	112.3 (3)
N2—Cu1—N2^{v}	88.61 (10)	C4—C3—H3A	109.1
N2^{ii}—Cu1—N2^{v}	88.60 (10)	S2—C3—H3A	109.1
N2^{iii}—Cu1—N2^{v}	91.39 (10)	C4—C3—H3B	109.1
N2^{iv}—Cu1—N2^{v}	91.39 (10)	S2—C3—H3B	109.1
O1—Cl1—O1^{vi}	112.77 (18)	H3A—C3—H3B	107.9
O1—Cl1—O1^{vii}	112.77 (18)	C4^{viii}—C4—C3	111.9 (4)

O1^{vi}—Cl1—O1^{vii}	112.77 (18)	C4^{viii}—C4—H4A	109.2
O1—Cl1—O2	105.9 (2)	C3—C4—H4A	109.2
O1^{vi}—Cl1—O2	105.9 (2)	C4^{viii}—C4—H4B	109.2
O1^{vii}—Cl1—O2	105.9 (2)	C3—C4—H4B	109.2
C1—S1—C2	86.55 (18)	H4A—C4—H4B	107.9
C2—S2—C3	101.18 (18)		
C2—N1—N2—C1	0.6 (4)	Cu1—N2—C1—S1	179.51 (16)
C2—N1—N2—Cu1	−179.3 (2)	C2—S1—C1—N2	0.1 (3)
N2^{i}—Cu1—N2—C1	84.4 (4)	N2—N1—C2—S1	−0.6 (4)
N2^{ii}—Cu1—N2—C1	175.8 (3)	N2—N1—C2—S2	179.7 (2)
N2^{iii}—Cu1—N2—C1	−4.2 (3)	C1—S1—C2—N1	0.3 (3)
N2^{v}—Cu1—N2—C1	−95.6 (4)	C1—S1—C2—S2	−179.9 (2)
N2^{i}—Cu1—N2—N1	−95.76 (17)	C3—S2—C2—N1	−0.8 (4)
N2^{ii}—Cu1—N2—N1	−4.3 (2)	C3—S2—C2—S1	179.4 (2)
N2^{iii}—Cu1—N2—N1	175.7 (2)	C2—S2—C3—C4	93.0 (3)
N2^{v}—Cu1—N2—N1	84.24 (17)	S2—C3—C4—C4^{viii}	165.9 (4)
N1—N2—C1—S1	−0.4 (4)		

Symmetry codes: (i) −*y*+2, *x*−*y*+1, *z*; (ii) −*x*+*y*+1, −*x*+2, *z*; (iii) *x*−*y*+1, *x*, −*z*+1; (iv) −*x*+2, −*y*+2, −*z*+1; (v) *y*, −*x*+*y*+1, −*z*+1; (vi) −*x*+*y*, −*x*+1, *z*; (vii) −*y*+1, *x*−*y*+1, *z*; (viii) −*x*+4/3, −*y*+5/3, −*z*+2/3.