Each ribose residue chelates the cation through three hydroxyl groups; the irregular coordination sphere is completed by seven water molecules, one of them is disordered [the O(5*WA*)···O(5*WB*) distance is 1·01 (1) Å].

The Ba^{2+} ions are linked by the O(4*W*) molecule (lying on the twofold axis) [Ba···Ba; $1-x$, y, $-z$] and bonded along the 2_1 axis by O(*W*1) and O(*W*3) water molecules [Ba···Ba; $1{\cdot}5-x$, $y+0{\cdot}5$, $-z$]; the Ba···Ba separations are 5·38 (1) and 4·96 (1) Å, respectively. The result is a two-dimensional net of Ba cations, parallel to the *xy* plane, with Ba coordination polyhedra sharing vertices or edges.

The hydroxyl and water H atoms are involved in hydrogen bonds. The hydrogen-bonding data are summarized in Table 3. Because some H-atom positions of the disordered water molecules are missing, only the O···O distances may suggest a possibility of hydrogen bonds. The disordered O(*W*6) water molecule is situated about 3·0 Å apart from the other O atoms, hence no strong hydrogen bonding involving the water of crystallization would be expected. This explains the easy loss of part of water from the crystal (Furberg & Mostad, 1962).

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Structure of Hydridotris[3-(*p*-tolyl)pyrazolyl]boratothallium(I)

By George Ferguson* and Michael C. Jennings

*Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N*1*G* 2*W*1

and Fergus J. Lalor* and Claire Shanahan

Department of Chemistry, University College, Cork, Republic of Ireland

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Abstract. $C_{30}H_{28}BN_6Tl$, $M_r = 687{\cdot}8$, trigonal, $R3c$, $a = 17{\cdot}551\,(4)$, $c = 31{\cdot}414\,(6)$ Å, $V = 8380\,(5)$ Å^3, $Z = 12$, $D_x = 1{\cdot}64$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 58{\cdot}7$ cm^{-1}, $F(000) = 4032$, $T = 294$ K, $R = 0{\cdot}017$ for 1034 unique reflections with $I > 2\sigma(I)$. There are two independent molecules of the complex in the asymmetric unit, both lying on crystallographic threefold axes. The two independent Tl—N bond lengths are 2·568 (4) and 2·587 (5) Å. In one molecule the *p*-tolyl ring is almost coplanar with the pyrazolyl ring to which it is bonded (interplanar angle 1·8°); in the other molecule the corresponding angle is 29·6°. The shortest Tl···Tl intermolecular distance is 3·8636 (4) Å along the threefold axis.

Introduction. Recent work from these and other laboratories has demonstrated amply that the chemical and structural properties of poly(1-pyrazolyl)borato complexes are remarkably sensitive to changes in the nature of the substituent at the 3-position of the pyrazolyl groups (Desmond, Lalor, Ferguson, Ruhl & Parvez, 1983; Trofimenko, Calabrese, Domaille & Thompson, 1989; Trofimenko, Calabrese & Thompson, 1987). In connection with a study of Group VI transition metal complexes containing sterically crowded poly(1-pyrazolyl)borate ligands, we had occasion

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* E-mail addresses: GF CHMFERG@VM.UOGUELPH.CA
FJL STCH8002@IRUCCVAX

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to prepare hydridotris[3-(*p*-tolyl)pyrazolyl]boratothallium(I), $HB(p\text{-}MeC_6H_4\text{-}C_3H_2N_2)_3Tl$ (1). Since there is little structural information available on main-group-element poly(1-pyrazolyl)borates as a class (Nicholson, 1984; Lee & Nicholson, 1986; Han, Looney & Perkin, 1989) and only a single crystallographically characterized thallium(I) derivative {the *tert*-butyl analogue of (1), hydridotris[3-(*tert*-butyl)pyrazol-1-yl]thallium(I)} (Cowley, Geerts, Nunn & Trofimenko, 1989), we have carried out an X-ray crystal structure analysis on (1).

Experimental. Complex (1) was prepared essentially as described by Trofimenko *et al.* (1987) for the 3-phenyl analogue.* Colourless prisms of (1) were grown from dichloromethane/petrol by slow diffusion. A small prism was chosen for data collection. Accurate cell dimensions and crystal orientation matrix were determined on a CAD-4 diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range $5 < \theta < 20°$. Crystal dimensions $0{\cdot}45 \times 0{\cdot}46 \times 0{\cdot}59$ mm; intensities of reflections from three equivalent sections of reciprocal space were measured with indices h −22 to 22, k 0 to 22, l −40 to 0, with $2 < 2\theta < 54°$; ω–2θ scans, ω-scan width $(0{\cdot}60 + 0{\cdot}35\tan\theta)°$; graphite-monochromatized Mo $K\alpha$ radiation; intensities of three reflections ($\bar{7}4\bar{2}$, $\bar{6}5\bar{8}$, $\bar{7}8\bar{6}$) measured every 3 h showed no significant variation. Data corrected for Lorentz, polarization and absorption effects (transmission factors 0·099 to 0·173). 6510 reflections were measured (three equivalent data sets), 2081 unique ($R_{\rm int}$ 0·018) and 1034 with $I > 2\sigma(I)$ were labelled observed and used in structure solution and refinement. The hexagonal cell used in data collection and refinement can be converted to its rhombohedral equivalent cell [$a = 14{\cdot}572\ (5)$ Å, $\alpha = 74{\cdot}06\ (2)°$, $V = 2793\ (4)$ Å^3] using the matrix: [$\frac{2}{3}$, $\frac{1}{3}$, $\frac{1}{3}$; $-\frac{1}{3}$, $\frac{1}{3}$, $\frac{1}{3}$; $-\frac{1}{3}$, $-\frac{2}{3}$, $\frac{1}{3}$]. Laue symmetry $\bar{3}m$ and conditions limiting possible reflections (hkl present if $-h + k + l = 3n$; $h\text{–}hl$ present if $l = 2n$) were consistent with either space group $R3c$ (No. 161) or $R\bar{3}c$ (No. 167). In $R\bar{3}c$ with $Z = 12$, the asymmetric unit would consist of $\frac{1}{3}$ of a molecule of the complex with the B and Tl atoms lying on a crystallographic threefold axis; in $R3c$ there would be two independent molecules each lying on threefold axes. The structure was solved using the Patterson heavy-atom method; attempts were made initially to solve the structure in the centrosymmetric space group but it soon became obvious that this was an incorrect choice; the structure was then solved and refined in space group $R3c$. Refinement was by full-matrix least-squares calculations on F, initially with isotropic and then with anisotropic thermal parameters for the non-H atoms. Difference electron-density contour plots through the planes of the anticipated locations of the methyl H atoms of the *p*-methyl groups yielded in each case a torus of electron density indicating that these H atoms were disordered over several conformers; they were allowed for by placing six H atoms with 0·5 occupancy equally spaced around the torus. In the final rounds of calculations the other H atoms were also positioned on geometrical grounds (C—H, 0·95; B—H, 1·08 Å) and included (as riding atoms) in the structure-factor calculations. A secondary-extinction

Table 1. *Positional and thermal parameters and their e.s.d.'s*

$$B_{\rm eq} = \tfrac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + (ab\cos\gamma)\beta_{12} + (ac\cos\beta)\beta_{13} + (bc\cos\alpha)\beta_{23}].$$

	x	y	z	$B_{\rm eq}$ (Å^2)
Tl*A*	0·0	0·0	0·0*	4·483 (6)
N1*A*	0·0951 (4)	0·1164 (3)	−0·0564 (2)	4·6 (1)
N2*A*	0·0833 (4)	0·0858 (4)	−0·0966 (2)	4·8 (1)
C3*A*	0·1508 (5)	0·1407 (5)	−0·1209 (2)	6·5 (2)
C4*A*	0·2087 (4)	0·2074 (5)	−0·0966 (2)	6·2 (2)
C5*A*	0·1714 (4)	0·1914 (4)	−0·0558 (2)	4·4 (2)
C11*A*	0·2068 (4)	0·2481 (4)	−0·0180 (2)	4·7 (2)
C12*A*	0·1623 (6)	0·2280 (5)	0·0187 (3)	8·1 (3)
C13*A*	0·1987 (6)	0·2818 (6)	0·0548 (2)	9·6 (3)
C14*A*	0·2765 (4)	0·3571 (4)	0·0543 (2)	6·0 (2)
C15*A*	0·3188 (5)	0·3786 (5)	0·0163 (2)	7·3 (3)
C16*A*	0·2848 (5)	0·3247 (5)	−0·0191 (2)	6·3 (2)
C17*A*	0·3159 (6)	0·4138 (5)	0·0929 (2)	8·1 (3)
B*A*	0·0	0·0	−0·1102 (4)	9·5 (4)
Tl*B*	0·0	0·0	0·12299 (1)	4·876 (7)
N1*B*	0·1187 (3)	0·0521 (3)	0·1810 (1)	4·1 (1)
N2*B*	0·0958 (3)	0·0504 (3)	0·2225 (2)	4·3 (1)
C3*B*	0·1677 (4)	0·1014 (4)	0·2455 (2)	5·3 (2)
C4*B*	0·2391 (4)	0·1367 (4)	0·2190 (2)	4·9 (2)
C5*B*	0·2052 (4)	0·1043 (4)	0·1784 (2)	4·5 (2)
C11*B*	0·2516 (4)	0·1221 (4)	0·1378 (2)	4·6 (2)
C12*B*	0·2212 (4)	0·0616 (4)	0·1045 (2)	5·1 (2)
C13*B*	0·2680 (4)	0·0847 (4)	0·0663 (2)	5·2 (2)
C14*B*	0·3395 (4)	0·1631 (4)	0·0589 (2)	5·6 (2)
C15*B*	0·3719 (5)	0·2215 (5)	0·0926 (2)	6·5 (2)
C16*B*	0·3276 (5)	0·2037 (4)	0·1314 (2)	5·7 (2)
C17*B*	0·3878 (5)	0·1839 (5)	0·0168 (2)	6·7 (2)
B*B*	0·0	0·0	0·2383 (3)	4·0 (2)

The two independent molecules lie on a crystallographic threefold axis and the remaining atoms' coordinates are given by application of the symmetry transformations ($-y$, $x - y$, z) and ($y - x$, $-x$, z).

* Fixed to define the origin in the space group $R3c$.

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* Anal. Calcd for $C_{30}H_{28}BN_6Tl$: C, 52·39; H, 4·10; N, 12·22. Found: C, 52·46; H, 4·14; N, 12·20%. IR (CH_2Cl_2) ν(BH) 2473 (*m*) cm^{-1}. ^{1}H NMR (Jeol GSX270, acetone-d_6, Me_4Si. Note that here and in the ^{13}C NMR data which follow, the atoms are identified according to the crystallographic numbering scheme shown in Fig. 1, and all three ligand *p*-tolylpyrazolyl groups are equivalent) δ 7·84 [*d*, 3H, H(3) of pyrazolyl groups, $^3J_{\rm HH}$ *ca* 2·15 Hz], 7·59, 7·22 [*dd*, 12H, C_6H_4, $^3J_{\rm HH}$ *ca* 8·2 Hz], 6·51 [*d*, 3H, H(4) of pyrazolyl groups, $^3J_{\rm HH}$ *ca* 2·15 Hz], 2·32 [*s*, 9H, Me-*p*]. ^{13}C NMR (Jeol GSX270. Acetone-d_6, Me_4Si) δ 154·49, [C(5) of pyrazolyl groups], 138·28 [C(14) of *p*-tolyl groups], 138·06 [C(3) of pyrazolyl groups], 132·00 [C(11) of *p*-tolyl groups], 130·33 [C(13), C(15) of *p*-tolyl groups], 127·61 [C(12), C(16) of *p*-tolyl groups], 104·02 [C(14) of *p*-tolyl groups], 21·15 [CH_3 of *p*-tolyl groups]. Significant broadening of the resonance due to the C(13), C(15) carbons of the *p*-tolyl groups (line-width at half-height ≈ 28·4 Hz) is ascribed to unresolved coupling (Cowley *et al.*, 1989) with ^{205}Tl.

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

Tl*A*	N1*A*	2·587 (5)	Tl*B*	N1*B*	2·568 (4)
N1*A*	N2*A*	1·348 (7)	N1*B*	N2*B*	1·359 (6)
N1*A*	C5*A*	1·327 (6)	N1*B*	C5*B*	1·325 (7)
N2*A*	C3*A*	1·331 (8)	N2*B*	C3*B*	1·337 (7)
N2*A*	B*A*	1·544 (6)	N2*B*	B*B*	1·539 (6)
C3*A*	C4*A*	1·341 (9)	C3*B*	C4*B*	1·370 (9)
C4*A*	C5*A*	1·403 (9)	C4*B*	C5*B*	1·403 (8)
C5*A*	C11*A*	1·472 (9)	C5*B*	C11*B*	1·459 (8)
C11*A*	C12*A*	1·337 (11)	C11*B*	C12*B*	1·392 (9)
C11*A*	C16*A*	1·358 (8)	C11*B*	C16*B*	1·399 (7)
C12*A*	C13*A*	1·410 (11)	C12*B*	C13*B*	1·397 (9)
C13*A*	C14*A*	1·343 (9)	C13*B*	C14*B*	1·340 (7)
C14*A*	C15*A*	1·358 (10)	C14*B*	C15*B*	1·381 (10)
C14*A*	C17*A*	1·498 (10)	C14*B*	C17*B*	1·514 (10)
C15*A*	C16*A*	1·387 (10)	C15*B*	C16*B*	1·396 (10)
Tl*A*···Tl*B*		3·8636 (4)			

N1*A*	Tl*A*	N1*A*^{i}	78·2 (2)	N1*B*	Tl*B*	N1*B*^{i}	75·2 (2)
Tl*A*	N1*A*	N2*A*	114·7 (3)	Tl*B*	N1*B*	N2*B*	119·6 (3)
Tl*A*	N1*A*	C5*A*	134·8 (4)	Tl*B*	N1*B*	C5*B*	130·3 (4)
N2*A*	N1*A*	C5*A*	106·7 (5)	N2*B*	N1*B*	C5*B*	107·4 (4)
N1*A*	N2*A*	C3*A*	110·1 (5)	N1*B*	N2*B*	C3*B*	109·6 (4)
N1*A*	N2*A*	B*A*	122·0 (6)	N1*B*	N2*B*	B*B*	123·2 (5)
C3*A*	N2*A*	B*A*	127·9 (7)	C3*B*	N2*B*	B*B*	127·1 (5)
N2*A*	C3*A*	C4*A*	108·5 (6)	N2*B*	C3*B*	C4*B*	108·4 (5)
C3*A*	C4*A*	C5*A*	105·8 (5)	C3*B*	C4*B*	C5*B*	105·3 (5)
N1*A*	C5*A*	C4*A*	108·9 (5)	N1*B*	C5*B*	C4*B*	109·3 (5)
N1*A*	C5*A*	C11*A*	124·0 (5)	N1*B*	C5*B*	C11*B*	121·7 (5)
C4*A*	C5*A*	C11*A*	127·1 (5)	C4*B*	C5*B*	C11*B*	128·9 (5)
C5*A*	C11*A*	C12*A*	121·5 (5)	C5*B*	C11*B*	C12*B*	122·1 (5)
C5*A*	C11*A*	C16*A*	121·6 (6)	C5*B*	C11*B*	C16*B*	119·3 (5)
C12*A*	C11*A*	C16*A*	116·8 (3)	C12*B*	C11*B*	C16*B*	118·6 (6)
C11*A*	C12*A*	C13*A*	120·6 (6)	C11*B*	C12*B*	C13*B*	118·6 (5)
C12*A*	C13*A*	C14*A*	123·1 (7)	C12*B*	C13*B*	C14*B*	124·0 (6)
C13*A*	C14*A*	C15*A*	115·4 (6)	C13*B*	C14*B*	C15*B*	117·2 (6)
C13*A*	C14*A*	C17*A*	123·3 (6)	C13*B*	C14*B*	C17*B*	121·5 (6)
C15*A*	C14*A*	C17*A*	121·3 (5)	C15*B*	C14*B*	C17*B*	121·1 (5)
C14*A*	C15*A*	C16*A*	121·9 (6)	C14*B*	C15*B*	C16*B*	121·8 (6)
C11*A*	C16*A*	C15*A*	122·1 (6)	C11*B*	C16*B*	C15*B*	119·6 (6)
N2*A*	B*A*	N2*A*^{i}	112·7 (6)	N2*B*	B*B*	N2*B*^{i}	110·1 (5)

Symmetry transformation (i) $-y, x-y, z$.

correction term (Zachariasen, 1963) was also refined [final value $3{\cdot}20\,(6)\times10^{-7}$]. The final cycle of refinement included 229 variable parameters, $R = 0{\cdot}017$, $wR = 0{\cdot}018$, goodness-of-fit 1·10, $w = 1/[\sigma F_o^2 + 0{\cdot}04F_o^2]$. Max. shift/e.s.d. 0·04; density in final difference map $\pm0{\cdot}28$ e Å^{-3}; there were no chemically significant features. Refinement of the enantiomorph of the structure gave a greater R value (0·027) implying that the initial assignment of the absolute stereochemistry was by chance correct. Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a PDP-11/73 computer using *SDP-Plus* (Frenz, 1983). Atomic coordinates and details of molecular geometry are given in Tables 1 and 2.* Fig. 1 is a view of the two independent molecules prepared using *ORTEP*II (Johnson, 1976). Fig. 2 is a view down the threefold axis showing the relative orientations of the *p*-tolyl groups.

Discussion. There are two independent molecules in the crystal structure, each lying on a threefold axis which passes through the Tl and B atoms (Fig. 1). The two molecules (*A* and *B*) pack so that their phenyl rings intermesh. In molecule *A* the *p*-tolyl ring is almost coplanar [interplanar angle 1·8 (9)°] with the pyrazolyl ring to which it is bonded; in molecule *B* the corresponding ring planes form a dihedral angle of 29·6 (7)°. This rotation allows the *A* and *B* tolyl rings to pack more efficiently and reduces the dihedral angle between them to a value of 8·5 (8)°. This is seen most clearly in Fig. 2 which shows a view down the threefold axis. It is also clear from Fig. 2 that both $B(N{-}N)_3Tl$ cages are twisted; the B—N—N—Tl torsion angles are 20·0 (8) and 14·2 (6)° for the *A* and *B* molecules respectively (in an ideal cage with 3*m* symmetry, the B—N—N—Tl torsion angle would be 0°).

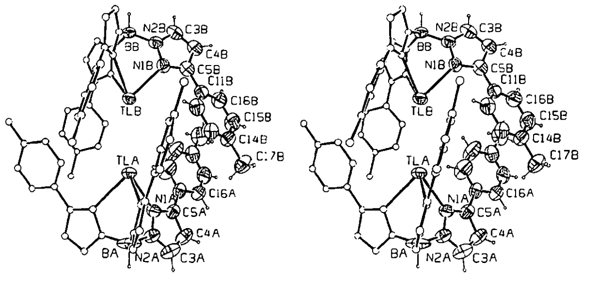

Fig. 1. A stereoview of the two independent molecules *A* and *B* showing our numbering scheme. For the atoms of the asymmetric unit, ellipsoids are shown at the 50% probability level; for clarity, symmetry-related atoms have been assigned spheres of arbitrary size.

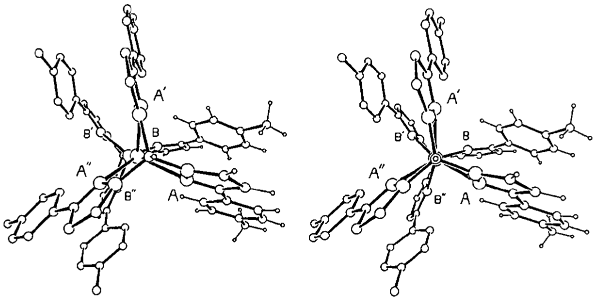

Fig. 2. A stereoview down the threefold axis showing the orientation of the pyrazolyl rings; molecule *A* is above molecule *B*; for clarity the atoms have all been assigned spheres of arbitrary size and H atoms are only shown on the atoms in the asymmetric unit. The ′ and ″ labels indicate the symmetry transformations $-y, x-y, z$ and $y-x, -x, z$ which should be applied to the coordinates of Table 1 to produce the complete molecular structures.

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* A drawing of the data crystal, lists of structure factors, thermal parameters, calculated hydrogen coordinates, mean plane data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54203 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The Tl—N distances in (1) of 2·568 (4) and 2·587 (5) Å are typical of covalent bonding between these atoms. In hydridotris(3-*tert*-butyl-1-pyrazolyl)-thallium(I) (which has crystallographic mirror symmetry; Cowley *et al.*, 1989) the corresponding distances are 2·582 (6) and 2·587 (9) Å. The Tl—N distances in (*N*,*N*′,*N*″-trimethyl-1,4,7-triazacyclononane)thallium(I) hexafluorophosphate are 2·59 (2)–2·63 (1) Å (Weighardt, Kleine-Boymann, Nuber & Weiss, 1986). The acute N—Tl—N angles of 75·2 (2) and 78·2 (2)° are due to steric demands of the pyrazolyl ligand; the values in the *tert*-butyl derivative are 75·2 (2) and 78·3 (2)°.

The van der Waals radius of a Tl atom given by Bondi (1964) is 1·96 Å; the Tl···Tl separation of 3·8636 (4) Å in (1) thus probably implies that there is little significant interaction between the two Tl atoms in this crystal structure; closer approach of the thallium atoms is precluded in any case as it would lead to unacceptably short intermolecular contacts between the phenyl rings of the two molecules. The packing in the 3-*tert*-butyl derivative is quite different from that reported here, presumably because of the greater bulk of the *tert*-butyl groups. In [{η^5-$(PhCH_2)_5C_5$}Tl] a quasi dimer is formed about an inversion centre with Tl···Tl 3·632 Å (Schumann, Janiak, Khan & Zuckermann, 1988). Maroni & Spiro (1968) claim that normal coordinate analysis of the IR and Raman spectra of $Tl_4(OR)_4$ derivatives fails to predict the observed spectrum unless some Tl···Tl bonding is assumed; the only X-ray data available are from an incomplete analysis using two-dimensional data of $Tl_4(OMe)_4$ where Tl···Tl separations in the range 3·81–3·86 Å are reported (Dahl, Davis, Wampler & West, 1962).

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Structure and Conductivity of a New Phase of Di[3,4;3′,4′-bis(ethylenedithio)-2,2′,5,5′-tetrathiafulvalenium] Dichlorocuprate(I); β-(BEDT-TTF)$_2$CuCl$_2$

By Xianhui Bu and Philip Coppens

Department of Chemistry, State University of New York at Buffalo, Buffalo, New York 14214, *USA*

and Barbara Lederle and Michael J. Naughton

Department of Physics and Astronomy, State University of New York at Buffalo, Buffalo, NY 14260, *USA*

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Abstract. β-$(C_{10}H_8S_8)_2[CuCl_2]$, $M_r = 903{\cdot}8$, triclinic, $P\bar{1}$, $a = 6{\cdot}623\ (2)$, $b = 9{\cdot}723\ (2)$, $c = 12{\cdot}658\ (3)$ Å, $\alpha = 85{\cdot}98\ (2)$, $\beta = 79{\cdot}72\ (2)$, $\gamma = 79{\cdot}95\ (2)°$, $V = 789{\cdot}0\ (4)$ Å^3, $Z = 1$, $D_x = 1{\cdot}90$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 19{\cdot}01$ cm^{-1}, $F(000) = 455$, room temperature, $R(F) = 0{\cdot}049$, $wR(F) = 0{\cdot}057$ for 1382 unique reflections. The structure consists of layers of partially oxidized BEDT-TTF cations [BEDT-TTF = bis(ethylenedithio)tetrathiafulvalene] separated along the **c** direction by discrete, linear $CuCl_2^-$ anions. Intermolecular S···S distances shorter than the sum of the van der Waals radii (3·6 Å) are found

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