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COPPER(I) CHLORIDE π-COMPLEXES WITH 2,5-bis(ALLYLTHIO)-1,3,4-THIADIAZOLE: SYNTHESIS AND STRUCTURAL FEATURES

Yu. I. Slyvka, B. R. Ardan, and M. G. Mys′kiv

Three new Cu(I) π-complexes with 2,5-bis(allylthio)-1,3,4-thiadizole (Bialtia) ([Cu_3Cl_3(Bialtia)$_2$] (**1**), [Cu_3Cl_3(Bialtia)] (**2**), and [Cu_2Cl_2(Bialtia)] (**3**)) are obtained by the alternating current electrochemical synthesis and studied by single crystal X-ray diffraction. A distinctive feature of Bialtia is its ability to coordinate to Cu(I) atoms by nitrogen atoms of the thiadiazole core and two allyl groups. The formation of island inorganic {Cu_3Cl_3} moieties occurs in **1** through π coordination of two independent Bialtia molecules by three metal atoms. Due to the absence of one Bialtia molecule in **2**, inorganic {–Cu_3Cl_3–} subunits join into infinite chains {[Cu_3Cl_3(Bialtia)]}$_n$. The structure of [Cu_2Cl_2(Bialtia)] (**3**) contains two Cu(I) atoms with different coordination environments: distorted tetrahedral and trigonal.

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Among multiple thiadiazole derivatives known to exist in four isomeric forms, 1,3,4-thiadiazoles are the most studied class of compounds used in pharmacology, agriculture, and also as corrosion inhibitors, luminophores, and precursors in the synthesis of transition metal organic compounds [1-4]. Although many 1,3,4-thiadiazoles, including their allyl derivatives, were first obtained in the 19th century, until recently there were no any crystal structural information on their copper(I) π-complexes in the literature [5, 6]. However, the alternating current electrochemical synthesis method enabled us to prepare a series of Cu(I) π-complexes with 2-allylamino-5-methyl-1,3,4-thiadiazole (Mepeta), 2-allylamino-5-phenyl-1,3,4-thiadiazole (Phepeta), and 5-allylthio-1,3,4-thiadiazole-2-amine (Pesta), which were studied by single crystal X-ray diffraction [6-9].

When there are simultaneously the 1,3,4-thiadiazole (or 1,2,3-triazole) core and a labile allyl group bonded to it (whose C=C bond acts as Pearson's soft base [10]), it appears to be favorable for the selective involvement of atoms of inorganic anions in Cu(I) coordination and promotes the formation of previously unknown inorganic moieties, in particular, the $Cu^I\cdots FSiF_5^{2-}$ bonds [9, 11-13]. The prevailing tendency of the Mepeta and Pesta ligands to form dimeric $[Cu_2(Mepeta)_2]^{2+}$ and $[Cu_2(Mepeta)_2]^{2+}$ moieties with their N,N,(C=C)-chelate bridging π,σ coordination to the copper(I) ion was also established.

In order to study the coordination features of diallyl derivatives of 1,3,4-thiadiazoles, namely, 2,5-bis(allylthio)-1,3,4-thiadizole $C_8H_{10}N_2S_3$ (Bialtia), with respect to copper(I) chloride, three new π-complexes with the composition

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$[Cu_3Cl_3(Bialtia)_2]$ (**1**), $[Cu_3Cl_3(Bialtia)]$ (**2**), and $[Cu_2Cl_2(Bialtia)]$ (**3**) were synthesized and analyzed by single crystal X-ray diffraction.

EXPERIMENTAL

Synthesis. 1,3,4-Thiadiazole-2,5-dithiol was obtained according to the known procedure from hydrazine sulfate and carbon disulfide [14]. 2,5-Bis(allylthio)-1,3,4-thiadizole (Bialtia) was prepared by allylation of 1,3,4-thiadiazole-2,5-dithiol with allyl chloride in the presence of $NaHCO_3$ in an ethanol solution. The yield was 96%. 2,5-Bis(allylthio)-1,3,4-thiadizole is a yellow liquid at room temperature. Bialtia: ^1H NMR (Bruker Avance spectrometer, 500 MHz, MeOD) δ 5.97 ppm (ddt, J = 17.0 Hz, 10.0 Hz, 7.0 Hz, 2H, =CH), 5.32 ppm (dd, J = 16.9 Hz, 1.2 Hz, 2H, CH_2=), 5.19 ppm (dd, J = 10.0 Hz, 0.5 Hz, 2H, CH_2=), 3.89 ppm (d, J = 7.0 Hz, 4H, CH_2). ^{13}C NMR (126 MHz, MeOD) δ, 167.23 ppm (C-ring), 133.53 ppm (=CH), 119.91 ppm (CH_2=), 38.16 ppm (CH_2).

$[Cu_3Cl_3(Bialtia)_2]$ compound (**1**) was obtained as colorless prismatic crystals on copper electrodes by the alternating current electrochemical synthesis [15] from 4 ml of an ethanol solution of 0.22 g (1.3 mmol) of $CuCl_2{\cdot}2H_2O$ and 0.16 g (0.7 mmol) of Bialtia in one day at the alternating current voltage of 0.4 V.

Single crystals of $[Cu_3Cl_3(Bialtia)]$ (**2**) were also obtained by the alternating current electrochemical synthesis from 4 ml of an ethanol solution of 0.22 g (1.3 mmol) of $CuCl_2{\cdot}2H_2O$ and 0.25 g (1.1 mmol) of Bialtia. Needle-shaped yellowish crystals of **2** formed on copper electrodes in five days at the alternating current voltage of 0.6 V. When this reactor was kept in a refrigerator at a temperature of –18 °C for two weeks, yellowish prismatic crystals (on the reactor bottom) of $[Cu_2Cl_2(Bialtia)]$ compound (**3**) formed.

Single crystal X-ray diffraction. A set of integrated reflection intensities for the crystals of compounds **1** and **3** was measured on a Kuma KM-4-CCD single crystal diffractometer (MoK_α radiation, graphite monochromator, Mercury CCD detector); and for the crystal of complex **2,** on an Agilent Xcalibur (CuK_α radiation, graphite monochromator, Onyx CCD detector). All three structures were solved and refined using the SHELXT and SHELXL software with the OLEX^2 interface [16-18]. The positional and thermal parameters of all non-hydrogen atoms (except for carbon C11, C12, and C13 atoms of the disordered allyl group of the organic ligand in **2**) for **1-3** were refined by the full-matrix least-squares method on F^2. In **1,** the Cu1 atom, together with C atoms of the allyl group coordinated to it, is disordered over two positions with their respective occupancies of 0.833(5) (Cu1A, C12A, C13A) and 0.167(5) (Cu1B, C12B, C13B). In **2**, carbon atoms of one of the allyl groups are also disordered over two positions with their respective occupancies of 0.66(2) (C11A, C12A, C13A) and 0.34(2) (C11B, C12B, C13B). The positions of hydrogen atoms were calculated geometrically and refined with a fixed C–H distance and $U_{iso}(H) = 1/2U_{eq}(C)$. Details of the single crystal X-ray diffraction and main crystal structural data for compounds **1-3** are given in Table 1.

Atomic coordinates and other parameters of compounds **1-3** have been deposited with the Cambridge Structural Database (Nos. CCDC 1543062-1543064 respectively) at the address of www.ccdc.cam.ac.uk/data_request/cif.

RESULTS AND DISCUSSION

$[Cu_3Cl_3(Bialtia)_2]$ π-complex (**1**) crystallizes in the triclinic crystal symmetry; the asymmetric unit of the structure contains two organic ligand molecules and three independent copper and chlorine atoms. One of the Bialtia molecules acts as the chelate-bridging π,σ ligand coordinating to the Cu(I) atom by two olefinic C=C bonds of two S-allyl groups and two N13 and N14 atoms of the 1,3,4-thiadiazole core (Fig. 1, Table 2). The Cu1 and Cu2 atoms have a similar distorted tetrahedral environment consisting of the nitrogen atom, the olefinic bond, and two bridging chlorine atoms ($\tau_4 = 0.82$ for Cu1A, $\tau_4 = 0.84$ for Cu1B, and $\tau_4 = 0.80$ for Cu2) [19, 20]. The Cl1 atom, on the one hand, joins Cu1 and Cu2 in a five-membered $\{N_2Cu_2Cl\}$ ring and, on the other hand, together with two other bridging Cl2 and Cl3 atoms, enters into the coordination

TABLE 1. Details of Single Crystal X-Ray Diffraction and the Main Crystal Structural Data for Compounds **1-3**

Characteristic	**1**	**2**	**3**
Empirical formula	$C_{16}H_{20}Cl_3Cu_3N_4S_6$	$C_8H_{10}Cl_3Cu_3N_2S_3$	$C_8H_{10}Cl_2Cu_2N_2S_3$
M, g/mol	757.69	527.33	428.34
T, K	150(2)	120(2)	150(2)
Crystal symmetry	Triclinic	Orthorhombic	Orthorhombic
Radiation	MoK_α	CuK_α	MoK_α
Space group	$P\bar{1}$	$P2_12_12_1$	$Pca2_1$
Unit cell parameters			
a, Å	9.863(3)	8.328(3)	11.033(4)
b, Å	9.937(3)	10.495(4)	8.345(3)
c, Å	14.138(4)	16.733(5)	14.382(4)
α, deg	78.63(3)	90	90
β, deg	88.30(3)	90	90
γ, deg	70.42(3)	90	90
V, Å^3	1278.9(7)	1462.5(9)	1324.2(8)
Z	2	4	4
ρ_{calc}, g/cm^3	1.97	2.40	2.15
μ, mm^{-1}	3.29	13.98	4.06
F(000)	756	1032	848
Crystal size, mm	0.39×0.20×0.12	0.56×0.12×0.10	0.45×0.29×0.26
$\theta_{min}\div\theta_{max}$, deg	2.9 ÷ 36.8	6.8 ÷ 89.1	3.1 ÷ 36.8
Limits of Miller indices	$-16 \le h \le 16$, $-15 \le k \le 15$, $-23 \le l \le 23$	$-10 \le h \le 5$, $-13 \le k \le 13$, $-21 \le l \le 21$	$-13 \le h \le 18$, $-11 \le k \le 12$, $-19 \le l \le 17$
Number of reflections: measured / independ.	20777 / 10065	10209 / 3262	11747 / 3996
used in refinement, $I \ge 2\sigma(I)$	7650	3016	3731
Number of independent parameters	311	171	155
S on F^2	1.022	1.026	1.041
$R[F^2 > 2\sigma(F^2)]$	0.0495	0.0510	0.0493
$wR(F^2)$	0.1288	0.1380	0.1361
Max / min of residual electron density, e/Å^3	0.94 / –0.77	1.25 / –0.64	1.37 / –1.09

environment of the third Cu3 atom, thus forming three fourth of the cube ($-Cu_3Cl_3-$). Interestingly that in nearly twenty structurally studied complexes with the N,(C=C)-coordination of heteroatomic ligands to Cu(I) there are monomeric CuCl, polymeric, or, more often, dimeric Cu_2Cl_2 moieties, whereas the island Cu_3Cl_3 moieties (Fig. 1) have not previously been found [11, 21-23]. The distorted tetrahedral environment of Cu3 ($\tau_4 = 0.79$) in **1**, in addition to three mentioned halogen atoms, includes yet another olefinic C=C bond of one of the allyl groups of the second Bialtia molecule. The Cu3–Cl1 distance (2.977(1) Å) is much longer than the other Cu–Cl bonds in complex **1**, but is shorter than the maximum contact between Cu(I) and Cl (3.15 Å) in the structure of olefinic copper(I) π-complexes with allyl derivatives of heterocyclic compounds [11].

Complex $[Cu_3Cl_3(\text{Bialtia})]$ (**2**) crystallizes in the orthorhombic crystal symmetry, in the acentric group $P2_12_12_1$; the asymmetric unit of the structure contains three independent copper and chlorine atoms and only one organic ligand molecule (Fig. 2, Table 3). The coordination of the Bialtia molecule in **2**, which is similar to that of the chelate-bridging π,σ-ligand in **1**, occurs via two N atoms of the heterocycle and two C=C bonds of the allyl groups. The Cu1 ($\tau_4 = 0.88$) and Cu2 ($\tau_4 = 0.86$) atoms in complex **2** have a distorted tetrahedral environment. The absence of the olefinic bond π-coordinated to the Cu3 atom

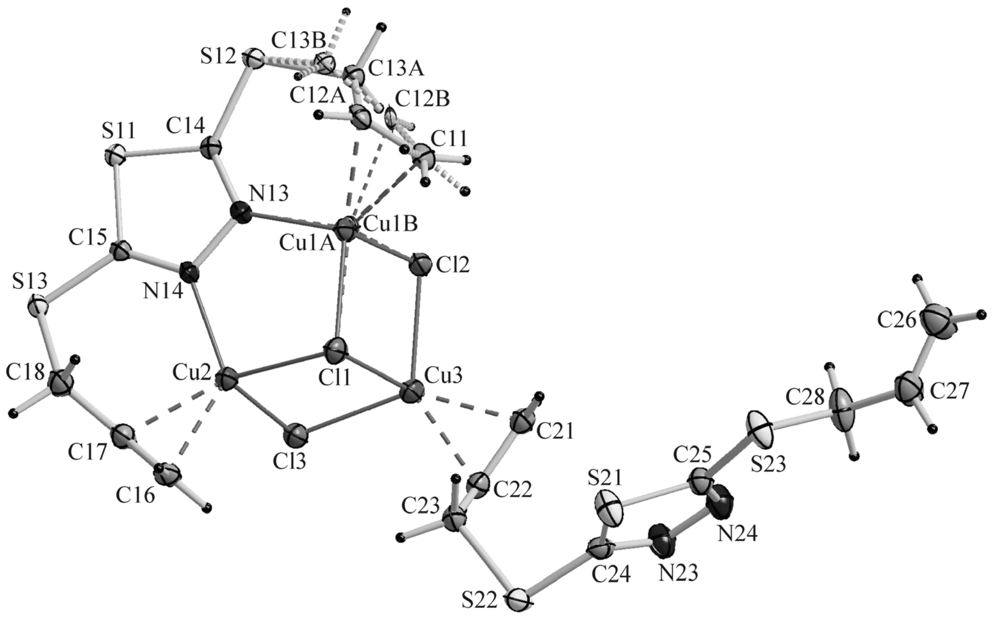

Fig. 1. Asymmetric unit of the structure of [$Cu_3Cl_3(Bialtia)_2$] complex (**1**). Disordered allyl group is depicted by dashed lines.

TABLE 2. Main Bond Lengths and Bond Angles in the Structure of Complex **1**

Bond	*d*, Å	Bond	*d*, Å	Angle	ω, deg	Angle	ω, deg
Cu1A–N13	2.002(3)	Cu2–Cl3	2.526(1)	N13–Cu1A–*m*1	112.6(1)	Cl3–Cu2–*m*2	100.19(9)
Cu1A–Cl1	2.289(2)	Cu2–*m*2	1.966(3)	Cl1–Cu1A–*m*1	132.3(1)	Cl1–Cu2–Cl3	96.31(4)
Cu1A–Cl2	2.598(2)	Cu3–Cl2	2.268(1)	Cl2–Cu1A–*m*1	105.3(1)	Cl1–Cu3–*m*3	105.63(8)
Cu1A–*m*1	1.944(3)	Cu3–Cl3	2.265(1)	Cl1–Cu1A–Cl2	98.12(8)	Cl2–Cu3–*m*3	123.05(8)
Cu2–N14	2.023(2)	Cu3–*m*3	1.947(3)	N14–Cu2–*m*2	114.95(9)	Cl3–Cu3–*m*3	126.01(8)
Cu2–Cl1	2.323(1)	Cu3–Cl1	2.977(1)	Cl1–Cu2–*m*2	131.88(9)	Cu1A–Cl1–Cu2	92.68(7)

Note. *m*1, *m*2, and *m*3 are the middles of the C11=C12A, C16=C17, and C21=C22 bonds respectively.

($\tau_4 = 0.82$) leads to the polymeric association of individual {Cu_3Cl_3} moieties through the appeared Cu3–Cl1^i contact of 2.287(3) Å (^{i} –0.5+*x*, 1.5–*y*, 1–*z*) in an infinite chain {[Cu_3Cl_3(Bialtia)]}$_n$ (Fig. 3*a*).

The structure of [Cu_2Cl_2(Bialtia)] complex (**3**) differs in the environment of two π-coordinated Cu1 and Cu2 atoms (Fig. 2*b*). The tetrahedral environment of the Cu1 atom includes the nitrogen atom of the 1,3,4-thiadiazole core, the C=C bond of the allyl group, and two halogen atoms; the bridging Cl1 atom joins the Cu(I) atoms in a five-membered {N_2Cu_2Cl} ring, whereas the Cl2 atom is purely terminal. Unlike **3**, in the [$Cu_2Cl_2(C_{12}H_{20}N_2)$] π complex with allylacetone azine, where the identical metal inorganic core with a five-membered {N_2Cu_2Cl} ring forms, the Cl2 atoms join individual links into a polymer chain (Fig. 3*b*) [24]. The trigonal environment of the Cu2 atom in **3** consists of only the C=C bond, the N atom of the heterocycle, and the mentioned bridging Cl1 atom because the nearest Cu2–Cl2 distance (3.922(2) Å) substantially exceeds the sum of the van der Waals radii of the respective atoms. The tendency of the π-coordinated copper(I) atom to bond to the terminal chlorine atom has previously been noted in the structures of CuCl compounds with 2-allylamino-5-phenyl-1,3,4-thiadizole [7], 3-allyl-2-iminobenzothiazole [25], 1-allyl-3-methylpiperazine [26], and 3-allylthio-4-allyl-5-phenyl-4*H*-1,2,4-triazole [27].

In the structures of **1-3** the hydrogen atoms of the allyl group are involved in the formation of a branched network of C–H….X hydrogen contacts (X = Cl, S, N) (Table 4). To visualize the geometry of these bonds for the fragment of the structure of **3**, the Hirshfeld surface was analyzed and the respective fingerprint diagrams with the selected H…Cl and H…S

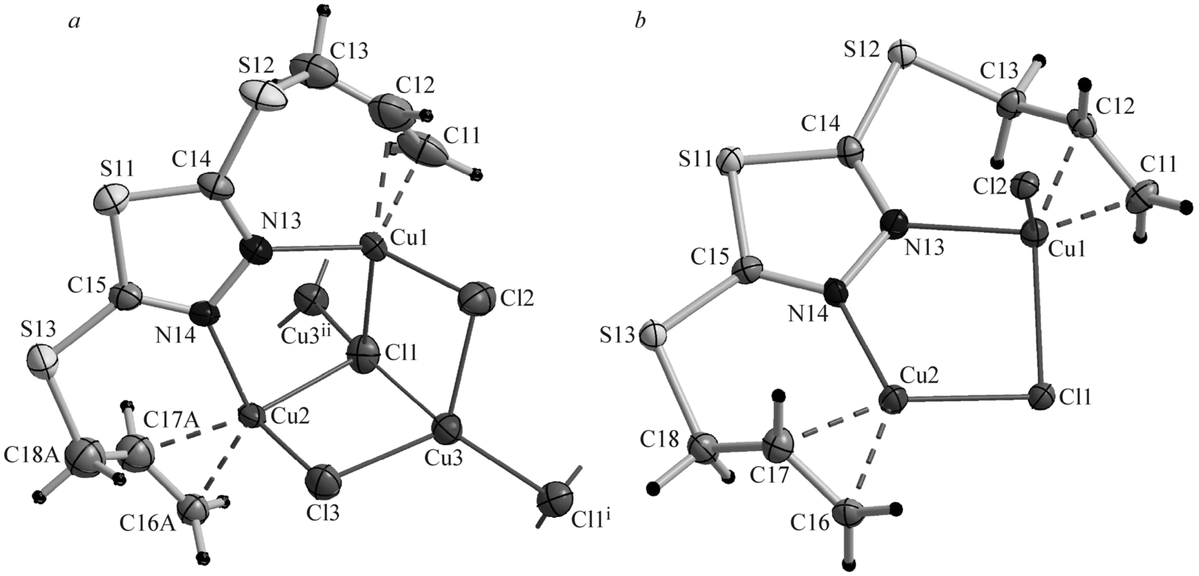

Fig. 2. Fragment of [Cu_3Cl_3(Bialtia)] structure (**2**) (*a*) and the asymmetric unit of [Cu_2Cl_2(Bialtia)] structure (**3**) (*b*). For clarity, the disordered part of the allyl group at the Cu2 atom is omitted in **2**. Symmetry codes: ^{i} –0.5+*x*, 1.5–*y*, 1–*z*; ^{ii} 0.5+*x*, 1.5–*y*, 1–*z*.

TABLE 3. Main Bond Lengths and Bond Angles in the Structures of Complexes **2** and **3**

Compound	**2**	**3**	Compound	**2**	**3**
Bond	*d*, Å		Angle	ω, deg	
Cu1–N13	2.027(8)	2.105(6)	N13–Cu1–*m*1	111.8(4)	109.4(3)
Cu1–Cl1	2.429(3)	2.384(2)	Cl1–Cu1–*m*1	118.9(4)	122.9(3)
Cu1–Cl2	2.348(3)	2.352(2)	Cl2–Cu1–*m*1	108.2(4)	119.4(3)
Cu1–*m*1	2.040(9)	1.995(6)	Cl1–Cu1–Cl2	101.93(9)	103.07(6)
Cu2–N14	2.033(7)	1.975(6)	N14–Cu2–*m*2	111.0(6)	117.0(3)
Cu2–Cl1	2.370(3)	2.224(2)	Cl1–Cu2–*m*2	123.5(6)	137.4(3)
Cu2–Cl3	2.383(3)		Cl3–Cu2–*m*2	115.6(6)	
Cu2–*m*2	1.976(9)	1.909(6)	Cl2–Cu2–Cl3	101.32(9)	
Cu3–Cl1	2.287(3)		Cu1–Cl1–Cu2	90.49(8)	89.80(6)
Cu3–Cl2	2.255(3)		Cl1–Cu3–Cl2	114.9(1)	
Cu3–Cl3	2.234(3)		Cl1–Cu3–Cl3	125.4(1)	
Cu3–Cl1^{i}	2.287(3)		Cl1–Cu3–Cl1^{i}	101.9(1)	

Note. *m*1 and *m*2 are the middles of the C11=C12 and C16=C17 (C16A=C17A in the structure of **2**) bonds respectively. Symmetry code: ^{i} –0.5+*x*, 1.5–*y*, 1–*z*.

contacts were presented (Fig. 4). The contribution of the H...H contact to the surface is 19.9%. The Hirshfeld surface and the corresponding fingerprint diagrams of the contacts were constructed using the CrystalExplorer software [28].

Thus, the formation of π-complexes **1-3**, in particular, their inorganic constituents, is due to the participation of two spatially directed allyl groups of the Bialtia molecule in the coordination of the metal atom. The coordination behavior of the Bialtia ligand with four centers active with respect to copper(I) substantially differs from that in the previously studied compounds with 2-allylamino-5-methyl-1,3,4-thiadiazole and 5-allylthio-1,3,4-thiadiazole-2-amine [6-9] prone to form solely dimeric $\{Cu_2L_2\}^{2+}$ moieties regardless of the nature of the inorganic anion.

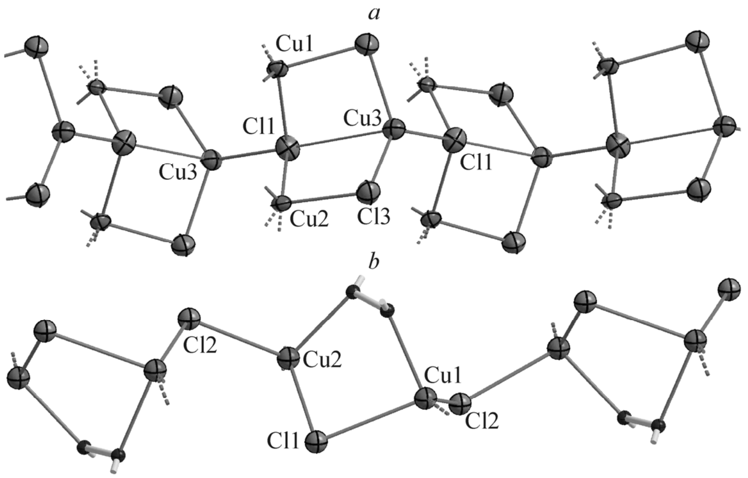

Fig. 3. Inorganic polymer in the structure of **2** (*a*) and in the structure of the [$Cu_2Cl_2(C_{12}H_{20}N_2)$] π complex [24] with allylacetone azine (*b*).

TABLE 4. Geometry of Selected Hydrogen Bonds in the Structures of **1**, **2**, and **3**

Bond	Symmetry code	Distance, Å			Angle, deg
D–H···A		D···H	H···A	D···A	D–H···A
		1			
C13A–H13B···Cl3	*x*, 1+*y*, *z*	0.99	2.85	3.607(3)	134
C13B–H13C···Cl3	*x*, 1+*y*, *z*	0.99	2.77	3.560(9)	137
C12A–H12A···N24	–1+*x*, 1+*y*, *z*	0.95	2.38	3.327(4)	173
C27–H27···S22	2–*x*, –*y*, 1–*z*	0.95	2.88	3.711(4)	147
		2			
C13–H13B···Cl2	0.5+*x*, 0.5–*y*, 1–*z*	0.99	2.84	3.46(1)	122
C18B–H18B···Cl2	1–*x*, 0.5+*y*, 1.5–*z*	0.99	2.63	3.48(5)	144
C12–H12···S12	–0.5+*x*, 0.5–*y*, 1–*z*	0.95	2.90	3.72(1)	145
		3			
C13–H13B···Cl2	–0.5+*x*, 1–*y*, *z*	0.99	2.85	3.524(7)	126
C12–H12···S13	1–*x*, 1–*y*, –0.5+*z*	0.95	2.96	3.827(7)	153

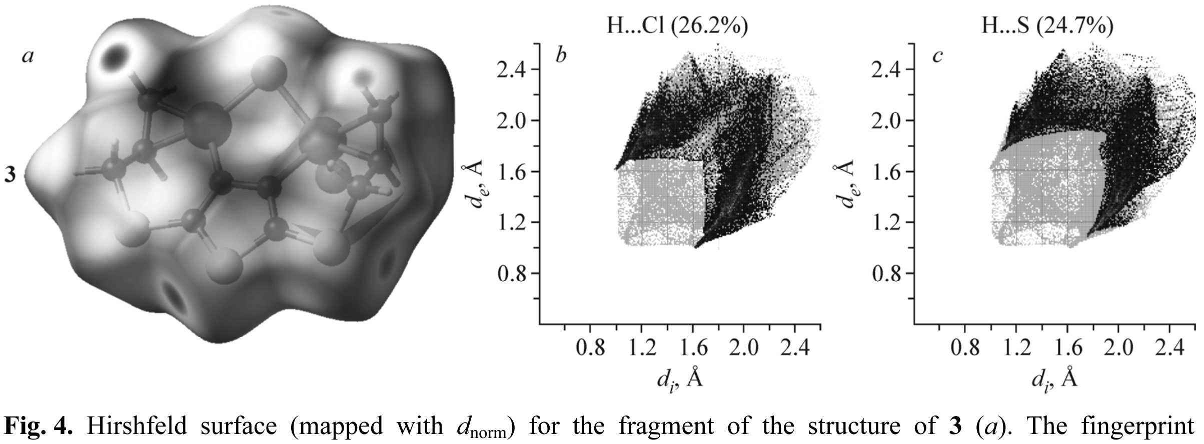

Fig. 4. Hirshfeld surface (mapped with d_{norm}) for the fragment of the structure of **3** (*a*). The fingerprint diagrams with the selected contacts H…Cl (*b*) and H…S (*b*, *c*).

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