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Crystal and Molecular Structures of the Complexes of Cobalt Dichloride with 1-Isopropenylimidazole and 1-Vinylimidazole

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Abstract—The crystal and molecular structures of bis(1-isopropenylimidazole)dichlorocobalt ($C_{12}H_{16}Cl_2 \cdot N_4Co$) [$R$ 0.036 (R_W 0.089) for 3229 unique reflections with $I > 2\sigma(I)$] and tetra(1-vinylimidazole)dichlorocobalt ($C_{20}H_{24}Cl_2N_8Co$) [R 0.031 (R_W 0.072) for 1863 unique reflections with $I > 2\sigma(I)$] were determined. In these molecular complexes, the monodentate terminal 1-alkenylimidazole ligands coordinate to the metal via a "pyridine" nitrogen atom. In $C_{12}H_{16}Cl_2N_4Co$, the Co atom has a distorted tetrahedral 2N,2Cl coordination. The coordination polyhedron of cobalt in $C_{20}H_{24}Cl_2N_8Co$ is a strongly elongated 4N,2Cl octahedron. The Co–N and Co–Cl bonds [Co–N 2.015(2) and 2.032(4) Å; Co–Cl 2.229(2) Å] in the tetrahedral complex $C_{12}H_{16}Cl_2N_4Co$ are shorter than those in the octahedral complex $C_{20}H_{24}Cl_2N_8Co$ [Co–N 2.134(2) and 2.157(2) Å; Co–Cl 2.518(1) Å]. In the structures of both complexes there are short contacts involving the Cl atoms.

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Among antianemic drugs containing such microelements as Fe, Cu, and Co, the Co compounds stimulate hemogenesis the most efficiently, enhancing also the activity of enzymes and hormones [1]. Since the determination of the structure of vitamin B_{12}, whose active center is the cobalt complex with a macrocyclic ligand derived from 5,6-dimethylbenzimidazole, active studies were performed on simulation of the structure and spectral parameters of the Co–N coordination core by various biomimetic synthetic complexes [2–7]. The results of our studies also demonstrate a high potential of complexes of cobalt salts with 1-alkenylimidazoles for their use as hemogenesis stimulants [8–10]. Kobazol drug, an effective hemogenesis stimulant with a broad spectrum of action, exhibiting erythro- and leucopoiesis-stimulating, immunomodulating, and antibacterial effect and approaching in the action mechanism natural hemogenesis stimulants, was developed at the Irkutsk Institute of Chemistry, Siberian Division, Russian Academy of Sciences on the basis of tetra(1-vinylimidazole)dichlorocobalt [9].

Recently we developed a convenient procedure for preparing 1-isopropenylazoles [10]. X-ray structural data for their complexes are lacking, but there are ample data on the structure of coordination compounds of CoX_2 (X = Cl, ClO_4, 1/2 SiF_6, NO_3) with 1-vinyl derivatives of imidazole and 2-methylimidazole of various compositions [11–13], including tetra(1-vinylimidazole)dichlorocobalt [14, 15]. However, in [14] the structure solution was based on an incorrect choice of the space group, and in [15] the intermolecular contacts and molecular packing in the crystal are not discussed.

In this study, with the aim to determine the steric structure and elucidate the effect of the complexation on the geometric parameters of the azole ligand with exocyclic unsaturated substituents, we performed a comparative single crystal X-ray diffraction study of bis(1-isopropenylimidazole)dichlorocobalt **I** and tetra(1-vinylimidazole)dichlorocobalt **II**.

In the monomeric molecule of bis(1-isopropenylimidazole)dichlorocobalt **I** (for the coordinates and temperature factors of atoms, see Table 1), the Co atom has a distorted tetrahedral coordination; the verticed of the tetrahedron are occupied by the nitrogen atoms of two independent monodentate terminal ligands L and L^A and by two chlorine atoms (Fig. 1).

Table 1. Coordinates (×10^4) and temperature factors ($Å^2 \times 10^3$) of atoms in the structure of bis(1-isopropenylimidazole)dichlorocobalt **I**

Atom	x	y	z	U_{eq}
Co^1	9792(1)	928(1)	2323(1)	49(1)
Cl^1	10136(1)	1923(1)	3893(1)	73(1)
Cl^2	12306(1)	–821(1)	1155(1)	62(1)
N^1	8027(2)	–92(2)	2725(1)	50(1)
N^2	6420(2)	–1403(2)	3517(1)	52(1)
N^{1A}	8497(2)	2875(2)	1417(1)	52(1)
N^{2A}	7791(2)	4654(2)	60(1)	49(1)
C^1	6907(3)	–46(3)	2016(2)	62(1)
C^2	5920(3)	–841(3)	2492(2)	64(1)
C^3	7699(3)	–920(2)	3619(2)	51(1)
C^4	5713(3)	–2343(3)	4324(2)	65(1)
C^5	4153(5)	–2343(5)	4259(4)	96(1)
C^6	6804(5)	–3239(5)	5152(3)	112(2)
C^{1A}	6866(3)	4139(3)	1822(2)	74(1)
C^{2A}	6414(3)	5235(3)	1007(2)	72(1)
C^{3A}	9000(2)	3214(2)	354(2)	49(1)
C^{4A}	7943(3)	5431(2)	–1015(2)	56(1)
C^{5A}	6878(4)	7020(3)	–1080(3)	78(1)
C^{6A}	9316(5)	4413(3)	–1961(2)	93(1)

The angular distortions in the coordination polyhedron are typical of cobalt 2N,2Cl tetrahedra: The ClCoCl angle is the largest (116.1°), the NCoC angle is the smallest (101.7°), and the ClCoN angles are intermediate (107.7°–111.5°) (Table 2). The Co–Cl [2.229(4), 2.241(3) Å] and Co–N [2.015(2), 2.032(4) Å] bonds are similar in the length to the corresponding bonds in the related tetrahedral complexes of cobalt with 1,1'-divinyl-2,2'-biimidazole [16] (mean Co–N distance 2.024 Å) and 1-vinyl-2-hydroxymethylimidazole (2.026 Å) [17] and are considerably shorter than the corresponding bonds in octahedral cobalt complexes.

Table 2. Interatomic distances (d) and bond angles (ω) in the structure of bis(1-isopropenylimidazole)dichlorocobalt **I**

Parameter	Molecule L	Molecule L^A
Bond	d, Å	
$Co^1–Cl^1$	2.229(4)	
$Co^1–Cl^2$	2.241(3)	
$Co^1–N^1$	2.015(2)	2.032(4)
$N^1–C^1$	1.382(3)	1.365(3)
$N^1–C^3$	1.313(3)	1.307(4)
$N^2–C^2$	1.380(4)	1.367(4)
$N^2–C^3$	1.346(3)	1.354(3)
$N^2–C^4$	1.442(3)	1.433(4)
$C^1–C^2$	1.331(4)	1.344(4)
$C^4–C^5$	1.333(4)	1.336(4)
$C^4–C^6$	1.470(5)	1.444(4)
Angle	ω, deg	
$Cl^1Co^1Cl^2$	116.1(1)	
$N^1Co^1Cl^1$	111.5(1)	107.7(2)
$N^1Co^1Cl^2$	109.9(1)	109.1(2)
$N^1Co^1N^{1A}$	101.7(1)	
$N^1C^3N^2$	110.3(2)	112.1(2)
$C^3N^2C^4$	126.4(2)	127.0(2)
$C^1C^2N^2$	106.6(2)	106.4(2)
$C^5C^4N^2$	118.3(3)	119.6(2)
$C^3N^1C^1$	106.5(2)	105.0(2)
$C^1N^1Co^1$	122.7(2)	125.8(2)
$C^3N^2C^2$	107.3(2)	106.0(2)
$C^2N^2C^4$	126.4(2)	126.9(2)
$C^2C^1N^1$	109.4(2)	110.5(2)
$N^2C^4C^6$	116.2(2)	116.0(2)
$C^5C^4C^6$	125.5(3)	124.4(3)

In the imidazole rings of the ligands in **I**, the delocalization of the π-electron density over the bonds is significant but nonuniform (Table 2). The shortest bond, $N^1–C^3$ (1.305–1.313 Å), is shorter than the N=C bonds in pyridine rings (typical length 1.336 Å) [18]. The $N^1–C^1$ and $N^2–C^2$ distances [mean length 1.374(4)±0.006 Å] in the rings are close to the standard length of the single $N–C_{sp^2}$ bond in imidazoles (1.375 Å) [18]. In the isopropenyl substituents, the bonds are delocalized to a greater extent than in the heterorings: The $C^4–C^5$ distance [1.335(4)±0.002 Å] is appreciably longer than the standard value for the C=C bond in vinyl groups of imidazole (1.275 Å) [16, 17] (see also data below for **II**). The $N^2–C^4$ and $C^4–C^6$ bonds have usual length (1.433–1.470 Å, Table 2). The C^5 and C^{5A} atoms of the methylene groups of the unsaturated substituents are in the cis positions to the C^2 and C^{2A} atoms of the heterorings, respectively. The $C^2N^2C^4C^5$ torsion angles in ligands L and L^A of **I** (–18.2° and +15.3°, respectively, Table 3) are simi-

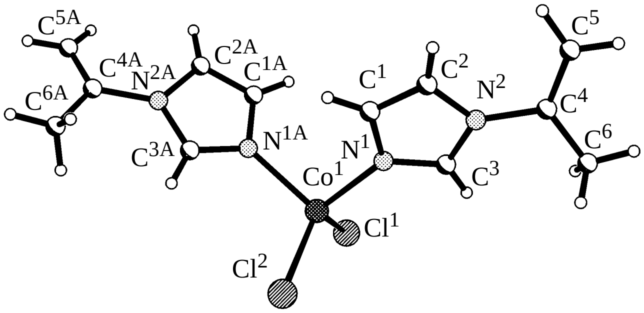

Fig. 1. Structure of bis(1-isopropenylimidazole)dichlorocobalt **I**.

lar in value but opposite in sign, i.e., the planes of the isopropenyl substituents are turned relative to the imidazole plane to opposite sides: by 161.8° in L and 15.6° in L^A. The C^5 and C^6 atoms deviate from this plane by –0.351 and +0.427 Å in L and by +0.394 and –0.249 Å in L^A, respectively. The dihedral angle between the heteroring planes in **I** is 108.6°. The L and L^A molecules are turned relative to the NCoN plane to different extents (by 148.6° and 59.5°), which is most likely associated with the packing requirements (Fig. 2). In complex **I** there are short contacts between the C atoms of both ligand molecules and Cl atoms. Owing to the short contacts $Cl^1\cdots H^3–C^3$, $Cl^1\cdots H^6–C^6$, and $Cl^2\cdots H^{2A}–C^{2A}$ (Table 4) with the geometry typical of hydrogen bonds, the molecules of the complex, multiplied by the inversion centers 0 1/2 0 and 0 1/2 1/2, form columns oriented along the crystallographic *x*-axis (Fig. 3). In addition, in the structure of **I** there are short contacts between the Cl atoms and ligands of the adjacent columns, combining the columns in a three-dimensional framework (Fig. 2).

In the monoclinic cell of tetra(1-vinylimidazole)dichlorocobalt **II** (for the coordinates and temperature parameters of atoms, see Table 5), the cobalt atoms occupy special positions in the inversion centers. The coordination surrounding of the Co atom is formed by four N atoms of monodentate terminal ligands L^1 and L^{1A} occupying the equatorial vertices of a strongly elongated octahedron and by two Cl atoms (Fig. 4). The Co–N bond lengths in **II** [2.134(1) and 2.157(1) Å, Table 6] are typical of an octahedrally coordinated Co^{2+} atom and are close to those found previously, e.g., in complexes of cobalt dichloride with pyridine (**III**) (2.183 Å) [19] or picolylpyrrolidin-2-one (**IV**) (2.145 Å) [20]. The Co–N bonds in **II** are appreciably longer than in the tetrahedral complexes. In particular, in **I** the Co–N bonds (2.032 and 2.015 Å) are considerably (by 0.123 Å on the average) shorter than in **II**. The Co–Cl bond in **II** [2.518(1) Å] is noticeably longer than in **III** (2.447 Å) and **IV** (2.472 Å) and considerably longer (by 0.285 Å on the average) than in tetrahedral complex **I**. This elongation can be caused by various factors, including the Jahn–Teller effect [21]. Bivalent cobalt (electronic configuration d^7) in the octahedral ligand field should have the $t_{2g}^6e_g$ state, which should result in the distortion (elongation) of the octahedron [21]. Also, the elongation of the Co–Cl bond in **II** may be caused by the participation of the Cl atoms in short contacts, similar in the geometry to hydrogen bonds, with one of the independent ligands (Table 7, Fig. 5). Owing to the presence of these contacts involving the vinyl group of L^{1A} only, the L^{1A} molecule is more planar than L^1.

Table 3. Selected torsion angles (τ, deg) in the molecule of bis(1-isopropenylimidazole)dichlorocobalt **I**

Angle	Molecule L	Molecule L^A
$Co^1N^1C^1C^2$	178.8	178.9
$Co^1N^1C^3N^2$	–178.7	–178.4
$C^2N^2C^4C^5$	–18.2	15.3
$C^3N^2C^4C^5$	162.3	–163.2
$C^2N^2C^4C^6$	161.4	–166.7
$C^3N^2C^4C^6$	–18.1	14.8

Similarly to **I**, the structure of **II** is predetermined by the transoid arrangement of the exocyclic C=C bonds relative to the $C^3–N^1$ bond of the ligands (Table 8). The vinyl groups in L^1 and L^{1A} deviate from the imidazole ring planes to opposite sides by 164.7° and 4.6°, respectively (Table 8); the C^4 and C^5 atoms of L^{1A} deviate from the heteroring plane to a considerably lesser extent (by 0.011 and –0.065 Å) than those of L^1 (by –0.017 and –0.303 Å). The imidazole rings of L^1 and L^{1A} are also oriented differently relative to the equatorial plane of the octahedron: The respective dihedral angles are 69° and 134°. The difference in the intermolecular contacts of L^1 and L^{1A} results in a certain difference in their geometry and is, most likely, responsible for a small tetragonal distortion in the equatorial plane of the octahedron (the NCoN angles are in the range 87.2°–92.8°, Table 6). The imidazole ring planes in **II** are turned relative to each other by 112.6°. The bond lengths in

Table 4. Short contacts in the structure of bis(1-isopropenylimidazole)dichlorocobalt **I**

Fragment	*d*(C–H), Å	*d*(H···Cl), Å	*d*(C···Cl), Å	∠(CHCl), deg	Symmetry code for Cl
$C^3–H^3\cdots Cl^1$	0.93	2.76(4)	3.690(4)	176(3)	2 – *x*, –*y*, 1 + *z*
$C^6–H^6\cdots Cl^1$	0.93	2.87(5)	3.641(4)	138(3)	2 – *x*, –*y*, 1 + *z*
$C^1–H^1\cdots Cl^2$	0.94	2.90(4)	3.787(4)	158(3)	2 – *x*, –*y*, –*z*
$C^2–H^2\cdots Cl^2$	0.93	2.80(5)	3.721(5)	169(3)	1 + *x*, *y*, *z*
$C^{2A}–H^{2A}\cdots Cl^2$	0.93	2.82(5)	3.748(4)	176(3)	1 + *x*, 1 – *y*, *z*

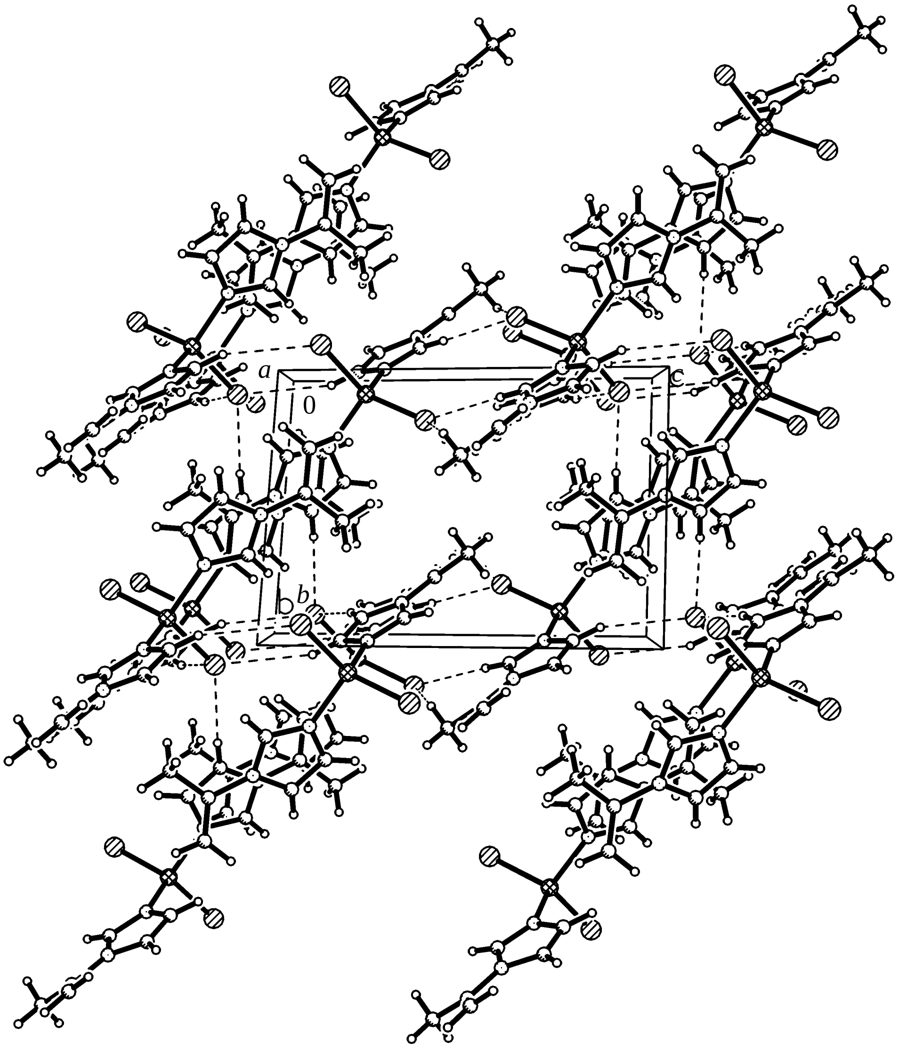

Fig. 2. Molecular packing of bis(1-isopropenylimidazole)dichlorocobalt **I** in the crystal (projection onto *yz* plane).

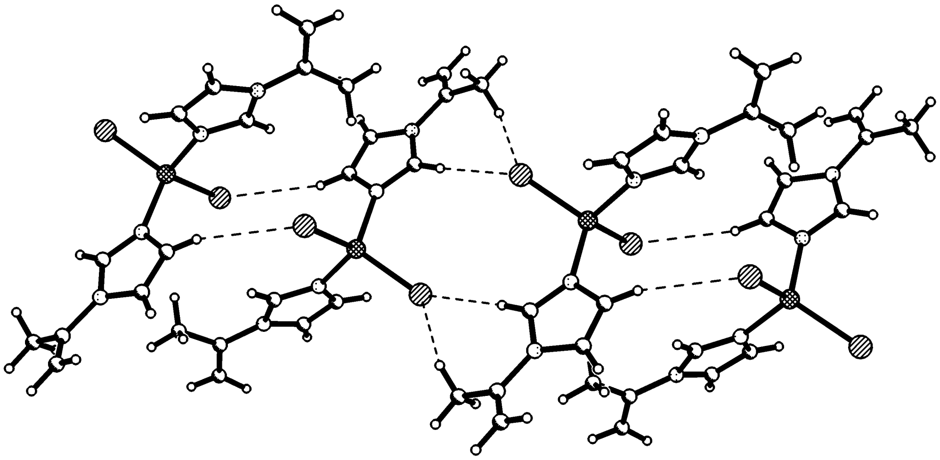

Fig. 3. Fragment of the structure of bis(1-isopropenylimidazole)dichlorocobalt **I**.

the imidazole rings in the ligands of **II** are the same as in ligands L and L^A in **I** (cf. Table 2). The C^4–C^5 bond length in the vinyl group of **II**, 1.275(3) Å, is close to that in the previously studied vinylimidazole complexes of cobalt dichloride [16, 17].

Figure 6 shows the molecular packing of **II** in the crystal. The complexes, multiplied by sliding reflec-

Table 5. Coordinates ($\times 10^4$) and temperature factors (Å$^2 \times 10^3$) of atoms in the structure of tetra(1-vinylimidazole)dichlorocobalt **II**

Atom	*x*	*y*	*z*	U_{eq}
Cl^1	2825(1)	–1026(1)	415(1)	45(1)
N^1	1845(3)	1026(1)	1070(2)	41(1)
N^2	4423(3)	1711(1)	2219(2)	50(1)
N^{1A}	492(3)	394(1)	–1720(2)	40(1)
N^{2A}	1975(3)	789(1)	–3087(2)	47(1)
C^1	1321(4)	1725(2)	1687(2)	51(1)
C^2	2900(4)	2145(2)	2400(2)	58(1)
C^3	3706(4)	1048(2)	1411(2)	48(1)
C^4	6447(5)	1864(2)	2749(3)	71(1)
C^5	7187(6)	2354(2)	3701(3)	82(1)
C^{1A}	–822(4)	500(2)	–2878(2)	56(1)
C^{2A}	75(4)	739(2)	–3721(2)	59(1)
C^{3A}	2140(3)	575(2)	–1884(2)	42(1)
C^{4A}	3539(5)	1020(2)	–3526(3)	66(1)
C^{5A}	3432(7)	1176(3)	–4675(4)	101(1)

Table 6. Interatomic distances (*d*) and bond angles (ω) in the structure of tetra(1-vinylimidazole)dichlorocobalt **II**

Parameter	Molecule L	Molecule L^A
Bond	*d*, Å	
Co^1–Cl^1	2.5188(6)	
Co^1–N^1	2.167(2)	2.134(2)
N^1–C^1	1.375(3)	1.369(3)
N^1–C^3	1.305(3)	1.305(3)
N^2–C^2	1.361(4)	1.365(3)
N^1–C^3	1.345(3)	1.346(3)
N^2–C^4	1.445(4)	1.418(3)
C^1–C^2	1.352(4)	1.345(4)
C^4–C^5	1.273(5)	1.278(4)
Angle	ω, deg	
Cl^{1}Co^{1}Cl^{1a}	180.0	
N^{1}Co^{1}Cl^1	89.6(5)	89.2(5)
N^{1}Co^{1}Cl^{1a}	90.4(5)	90.8(5)
N^{1a}Co^{1}Cl^1		90.8(5)
N^{1a}Co^{1}Cl^{1a}		89.2(5)
N^{1a}Co^{1}N^1	180.0	180.0
N^{1}Co^{1}N^{1A}	92.8(7)	
N^{1}Co^{1}N^{1Aa}	87.2(7)	
N^{1}C^{3}N^2	112.4(2)	112.1(2)
C^{3}N^{2}C^4	122.9(2)	124.0(2)
C^{1}C^{2}N^2	106.7(2)	107.0(2)
C^{5}C^{4}N^2	124.4(4)	125.0(3)
C^{3}N^{1}C^1	105.1(2)	105.5(2)
C^{1}N^{1}Co^1	127.0(2)	128.0(2)
C^{3}N^{2}C^2	106.4(2)	106.0(2)
C^{2}N^{2}C^4	130.8(2)	130.0(2)
C^{2}C^{1}N^1	109.5(2)	109.5(2)

[a] Symmetry code (–*x*, –*y*, –*z*).

tion planes *n*, are arranged in the centered motif. As already mentioned, in the structure of **II** there are short contacts with Cl atoms (Table 7), but, in contrast to **I**, they involve only one of the ligands, L$_{1A}$. These interactions link the molecules of **II** in columns (Fig. 5) oriented along *x*-axis coinciding with the shortest unit cell parameter *a*. The columns are arranged so that the planes of the heterocycles of the like ligands of the adjacent molecules, related by the inversion centers 0 0 1/2 and 0 1/2 0 and located in the closely lying parallel planes (2.5 and 3 Å), are shifted relative to each other by considerably longer distances exceeding 4 Å. There are only van der Waals contacts between the columns.

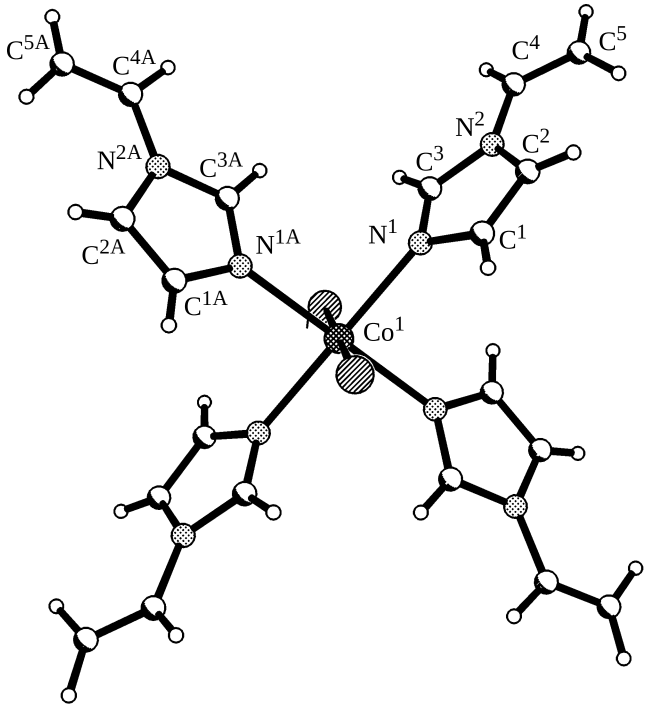

Fig. 4. Structure of tetra(1-vinylimidazole)dichlorocobalt **II**.

EXPERIMENTAL

Blue single crystals of **I** ($C_{12}H_{16}Cl_2N_4Co$) and violet single crystals of **II** ($C_{20}H_{24}Cl_2N_8Co$) were isolated in the form of well-edged prisms. The experimental data were obtained on a SMART CCD-1000 automatic diffractometer at room temperature [λMoK_α radiation, graphite monochromator, ω scanning, $2\theta_{max}$ 58.46° and 50.10° for **I** and **II**, respectively]. The SAINT program [22] was used for processing the ex-

Table 7. Short contacts in the structure of tetra(1-vinylimidazole)dichlorocobalt **II**[a]

Fragment	*d*(C–H), Å	*d*(H···Cl), Å	*d*(C···Cl), Å	∠(CHCl), deg
$Cl^1\cdots H^{3A}–C^{3A}$	0.95(3)	2.75(3)	3.633(3)	155(2)
$Cl^1\cdots H^{4A}–C^{4A}$	0.94(4)	2.81(4)	3.688(3)	156(3)

[a] Symmetry code for Cl: 1 – *x*, –*y*, –*z*.

perimental data and taking into account the absorption. Crystals of **I** are triclinic, space group $P\bar{1}$: *a* 8.426(7), *b* 8.942(16), *c* 11.89(3) Å; α 87.33(9)°, β 77.49(8)°, γ 65.31(7)°; *V* 794(3) Å^3, *Z* 2, ρ_{calc} 1.448 g cm^{-3}, μ_{Mo} 14.09 cm^{-1}. Crystals of **II** are monoclinic, space group $P2_1/n$: *a* 7.3395(13), *b* 15.103(3), *c* 11.121(2) Å; α 107.30(13)°, *V* 1177(4) Å^3, *Z* 2, ρ_{calc} 1.429 Mg m^{-3}, μ 9.80 cm^{-1}.

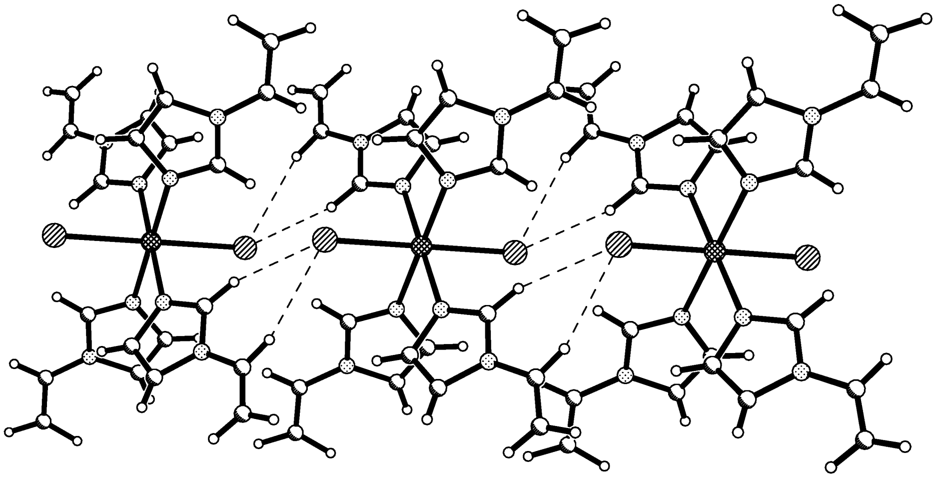

Fig. 5. Fragment of the structure of tetra(1-vinylimidazole)dichlorocobalt **II**.

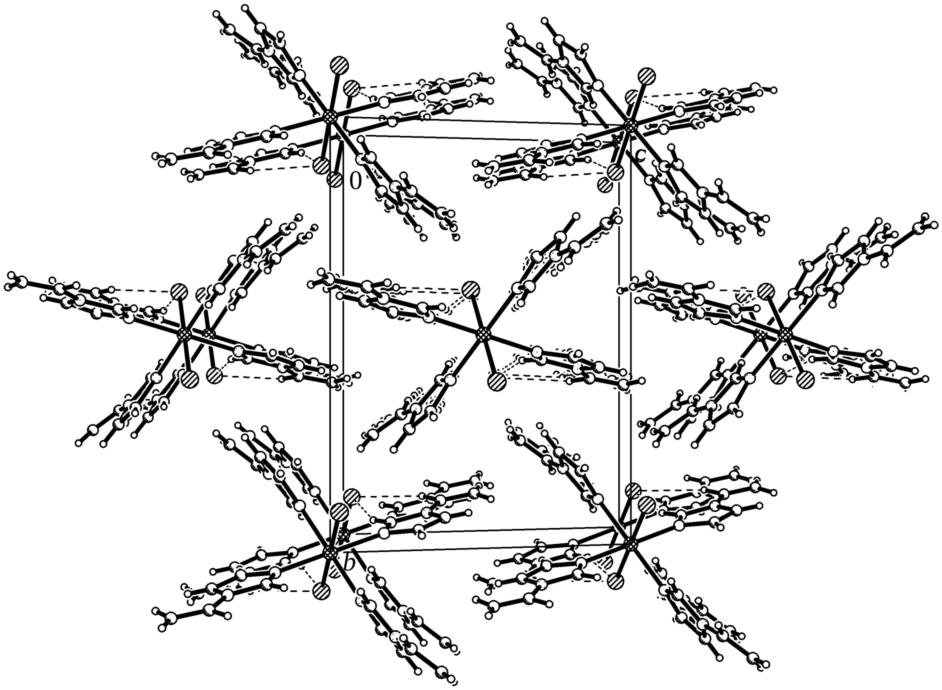

Fig. 6. Molecular packing of tetra(1-vinylimidazole)dichlorocobalt **II** in the crystal (projection onto *yz* plane).

Table 8. Selected torsion angles (τ, deg) in the molecule of tetra(1-vinylimidazole)dichlorocobalt **II**

Angle	Molecule L	Molecule L^A
$Co^1N^1C^1C^2$	170.9	–178.8
$Co^1N^1C^3N^2$	–171.0	178.9
$C^2N^2C^4C^5$	14.5	–4.9
$C^3N^2C^4C^5$	–164.4	175.8

The structures of **I** and **II** were solved by the direct method and refined by the full-matrix least-squares method in the anisotropic approximation for nonhydrogen atoms. The hydrogen atoms were localized in differential Fourier syntheses and were refined in the isotropic approximation. The final R factors are as follows: for **I** [3229 unique reflections with $I > 2\sigma(I)$], R 0.036 (R_W 0.089); for **II** [1863 unique reflections with $I > 2\sigma(I)$], R 0.031 (R_W 0.072). The computations were made with SHELXTL PLUS program package (PC-Version 5.0) [23].

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