π-Complexes of Copper(I) Halides with 3-(Allylamino)-(C₃H₅NHC₂H₄CN, Apn) and 3-(Diallylamino)-((C₃H₅)₂NC₂H₄CN, Dapn)-Propanenitrile. Syntheses and Crystal Structures of Compounds [CuCl(Apn)], [(H⁺Apn)Cu₂Cl₃], [(H⁺Dapn)CuCl₂], and [(H⁺Dapn)CuBr₂]

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Abstract—The π -complexes [CuCl(C₃H₅NHC₂H₄CN)] (I), [(C₃H₅NH₂C₂H₄CN)Cu₂Cl₃] (II), [((C₃H₅)₂NHC₂H₄CN)CuCl₂] (III), and [((C₃H₅)₂NHC₂H₄CN)CuBr₂] (IV) are obtained as single crystals by the ac electrochemical synthesis on copper wire electrodes from ethanolic solutions of 3-(allylamino)propanenitrile, 3-(diallylamino)propanenitrile, and CuX₂ (X = Cl, Br). Their crystal structures are determined. The crystals of compounds I, III, and IV are monoclinic, space group $P2_1/c$, Z = 4. The crystals of compound

II are triclinic, space group $P\overline{1}$, Z = 2. The unit cell parameters are a = 11.125(4), b = 8.769(4), c = 8.570(4) Å, $\beta = 90.94(4)^{\circ}$, V = 835.9(6) Å³ (**I**); a = 6.2566(4), b = 7.5975(6), c = 11.1251(8) Å, $\alpha = 90.896(6)^{\circ}$, $\beta = 92.827(5)^{\circ}$, $\gamma = 94.340(5)^{\circ}$, V = 526.57(7) Å³ (**II**); a = 11.656(4), b = 6.992(4), c = 14.681(5) Å, $\beta = 100.89(4)^{\circ}$, V = 1174.9(9) Å³ (**III**); a = 11.845(4), b = 7.282(4), c = 14.855(5) Å, $\beta = 100.37(4)^{\circ}$, V = 1260.4(9) Å³ (**IV**). The coordination mode of the Cu(I) atom in complex **I** includes two halogen atoms, the C=C bond, and the secondary amine N atom. The coordination environment in isostructural crystals of complexes **III** and **IV** is formed by the C=C bond and three halogen atoms as in complex **I**.

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The earlier studies of the π -complex formation of copper(I) with allyl derivatives of acyclic amines and nitriles showed differences in the influence of the donating amine N atoms and the donor-acceptor N atom of the cyano group on the formation of the coordination environment of the π -coordinated copper(I) atom [1]. The amine N atom and the C=C bond can coordinate to the same Cu(I) atom in the structures of 2CuCl · (diallylamine) [2], [Cu(diallylamine)(NO₃)] [3], and CuCl \cdot (allylamine) (V) [4], whereas the structures of copper(I) halides with allylnitriles (dienenitriles) exhibit the predominantly separate σ - and π coordination modes of Cu(I): $2CuCl \cdot (allyl cyanide)$ (VI, VII) [5, 6] and $2CuCl \cdot (1$ cyanobutadiene-1,3) [7]. Nevertheless, the cooperative coordination of Cu(I) involving the C=C bond and the CN group is observed, for example, in the structure of the π,σ complexes CuCl · (diallylcyanamide) [8], $CuCl \cdot (1-cvano-2-methylbutadiene-1.3)$ [9], and $[Cu(allyl cyanide)(NO_3)]$ [10]. Note that the protonated amine nitrogen atoms in the corresponding allyl derivatives have no possibility to coordinate to the metal atom, for instance, in the π complexes [H⁺(diallylamine)Cu(NO₃)₂] [11] and [{C₃H₅NH-C(NH₂)₂}Cu(NO₃)₂] [12].

It seemed of interest to study the coordination behavior with respect to Cu(I) of the N-allyl derivatives of aminonitriles, being ligands simultaneously containing amino and cyano groups along with C=C bonds. Therefore, we synthesized and studied by X-ray diffraction analysis single crystals of the copper(I) halide complexes with 3-(allylamino)propanenitrile (C₃H₅NHC₂H₄CN, **Apn**) and 3-(diallylamino)propanenitrile ((C₃H₅)₂NC₂H₄CN, **Dapn**): [CuCl(Apn)] (I), [(H⁺Apn)Cu₂Cl₃] (II), [(H⁺Dapn)CuCl₂] (III), and [(H⁺Dapn)CuBr₂] (IV).

EXPERIMENTAL

Synthesis of 3-(allylamino)propanenitrile. A mixture of acrylonitrile (6.8 mL, 0.1 mol) and allylamine (11.2 mL, 0.15 mol) was stored with cooling and continuous stirring (5 h, 20°C), preventing the temperature rise higher than 30°C [13]. Then the reaction mixture was heated for 1 h in a water bath with a reflux condenser at 60°C. The product was purified by distillation in a vacuum of a water-jet pump. The yield of Apn was 88% (15 mL).

Synthesis of 3-(diallylamino)propanenitrile. A mixture of acrylonitrile (7 mL, 0.11 mol) and diallylamine (16 mL, 0.13 mol) was heated with a reflux condenser in a water bath at 50–60°C for 8 h followed by storing at room temperature for 24 h [13]. After a small amount of unreacted starting substances was distilled off in a vacuum of a water-jet pump, the red-orange liquid was distilled at 130°C (20 mmHg). The yield of Dapn was 75% (13 mL).

Synthesis of I–VI. Colorless crystals of compound I were obtained by the ac electrochemical synthesis (U = 0.45 V, $I_{init} = 0.58$ mA) [14] on copper wire electrodes from CuCl₂ · 2H₂O (0.8 g, 4.5 mmol), Apn (0.6 mL, 5 mmol), and 95% C₂H₅OH (4.5 mL) for 4 days. Colorless crystals of complex II were obtained for 24 h under similar conditions using approximately the same amounts of reactants (an ethanolic solution of Apn was pretitrated with a concentrated aqueous solution of HCl to pH 4.5). Colorless crystals of compound III were obtained by the ac electrochemical synthesis [14] from $CuCl_2 \cdot 2H_2O(0.70 \text{ g})$, 4 mmol), Dapn (0.7 mL, 4.3 mmol), and 95% C_2H_5OH (4 mL) without HCl for 8 days. The crystals of compound III are also formed using an ethanolic solution of Dapn pretitrated with a concentrated aqueous solution of HCl to pH 4.5. Yellowish crystals of complex IV were grown for 5 days on copper electrodes under the conditions of ac electrochemical synthesis from CuBr₂ (0.67 g, 4 mmol), Dapn (0.7 mL, 4.3 mmol, was not titrated with HBr), and ethanol (4 mL). The density of crystals of I-IV was determined by the flotation method in a chloroform-bromoform mixture (Table 1).

X-ray diffraction analysis. After preliminary studies by the photomethod, diffraction sets obtained on KUMA-KM4/CCD and Rigaku AFC7 single-crystal diffractometers (CCD detector, MoK_{α} radiation, graphite monochromator, ω scan mode, low-temperature attachment) were used for structure determination of compounds I-IV. Corrections to the Lorentz and polarization factors were applied to reflection intensities. The X-ray experimental data were processed using the CrysAlisRED program [15] for compounds I, III, and IV. The Rigaku Crystal Clear program was used for processing the X-ray data for complex II [16]. Structures I-IV were solved by direct methods, and light atoms were revealed from the difference Fourier syntheses. An absorption correction was applied by the analytical method [17]. The structures of compounds I-IV were solved using the SHELX program package [18]. The hydrogen atoms in structures I-IV were found from the difference Fourier syntheses and refined in the riding model along with the non-hydrogen atoms. The crystallographic data for compounds **I**–**IV** and conditions of the diffraction experiment are collected in Table 1.

RUSSIAN JOURNAL OF COORDINATION CHEMISTRY Vol. 38 No. 2

Selected bond lengths and bonds angles are given in the captions for figures.

The coordinates of atoms and other parameters for compounds **I**–**IV** were deposited with the Cambridge Crystallographic Data Centre (nos. 811967, 811968, 811969, and 811970 for compounds **I**, **II**, **III**, and **IV**, respectively; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

The inorganic fragment Cu_2Cl_2 in structure I (Fig. 1) is identical to that in structure V [4]. The trigonal pyramidal coordination environment of Cu(I) is formed by the C=C bond, the amine nitrogen atom, the chlorine atom in the base of the pyramid, and the second Cl atom in the apical position. The C=C coordination bond length is 1.373(2) Å. The bridging function of the Apn molecule joins particular units Cu_2Cl_2 into a three-dimensional framework. It is surprisingly that the N atom of the cyano group of the Apn ligand is not coordinated by the metal atom.

The protonation of the amine N atom changes the coordination behavior of the Apn molecule: in the structure of π complex II, the N atom of the cyano group enters into the coordination environment of Cu(I). The structure of this compound consists of infinite ribbons $(Cu_2Cl_3)_n^{n-}$, similar to those described in the structures of $[H^+(diallylamine)Cu_2Br_3]$ [19] and π,σ complex VI [5]. The inorganic fragments are joined into the three-dimensional framework by the bridging cations H⁺Apn (Fig. 2). Unlike structure I, the trigonal pyramidal environment of one of the independent metal atoms in structure II contains the C=C bond and the Cl atoms, whereas the second Cu(I) atom is inside the slightly deformed tetrahedron with the nitrile N atom and three Cl atoms in the vertices. The structure of complex II is close to that of π,σ complex VI [5].

Isostructural π complexes III and IV substantially differ in structure from compounds I and II. They were formed during the synthesis in a medium of protonogenic ethanol, and the acidic medium appeared due to the solvolysis of CuCl₂ · 2H₂O or CuBr₂. The pronation of the amine nitrogen atom of Dapn imparts the cationic state H⁺Dapn to the ligand molecule and, hence, in structures III and IV it is coordinated to the Cu(I) atom by the C=C bond only (unlike II, where both the cyano group and the C=C bond are coordinated by the copper(I) atom). Owing to the bridging function of the halogen atoms, the polymeric anion

 $(CuX_2)_n^{n-}$, appears in which the Cu(I) atom coordinates the C=C bond and three X atoms (X = Cl, Br) (Fig. 3).

The C=C bond coordinated by copper(I) is elongated to 1.374(3) Å in **III** and to 1.365(5) Å in **IV**. Another double bond in the H⁺Dapn cation, which

2012

| Value | Value | | | | | | |
|---|---|--|--|--|--|--|--|
| I II III | IV | | | | | | |
| Empirical formula $C_6H_{10}N_2ClCu$ $C_6H_{11}N_2Cl_3Cu_2$ $C_9H_{15}N_2Cl_2Cu$ $C_9H_{15}N_2Cl_2Cu$ | I ₁₅ N ₂ Br ₂ Cu | | | | | | |
| <i>FW</i> 210 344.62 285.69 | 374.59 | | | | | | |
| Crystal size, mm $0.31 \times 0.30 \times 0.27$ $0.12 \times 0.1 \times 0.08$ $0.44 \times 0.21 \times 0.09$ $0.24 \times 0.21 \times 0.09$ | × 0.23 × 0.15 | | | | | | |
| Temperature, K 110(2) 200(1) 160(2) | 240(1) | | | | | | |
| Shape Prizms Blocks Prizms | Prizms | | | | | | |
| Diffractometer KUMA-KM4/CCD Rigaku AFC7 KUMA-KM4/CCD KUMA | A-KM4/CCD | | | | | | |
| Crystal system Monoclinic Triclinic Monoclinic M | onoclinic | | | | | | |
| Space group $P2_1/c$ $P\overline{1}$ $P2_1/c$ | $P2_{1}/c$ | | | | | | |
| Unit cell parameters: | | | | | | | |
| <i>a</i> , Å 11.125(4) 6.2566(4) 11.656(4) | 11.845(4) | | | | | | |
| b, Å 8.769(4) 7.5975(6) 6.992(4) | 7.282(4) | | | | | | |
| c, Å 8.570(4) 11.1251(8) 14.681(5) | 14.855(5) | | | | | | |
| α, deg 90 90.896(6) 90 | 90 | | | | | | |
| β, deg 90.94(4) 92.827(5) 100.89(4) | 100.37(4) | | | | | | |
| γ, deg 90 94.340(5) 90 | 90 | | | | | | |
| <i>V</i> , Å ³ 835.9(6) 526.57(7) 1174.9(9) 1 | 260.4(9) | | | | | | |
| Z 4 2 4 | 4 | | | | | | |
| $\rho_{calcd}, g/cm^3$ 1.662 2.179 1.615 | 1.974 | | | | | | |
| $\rho_{\text{meas}}, \text{g/cm}^3$ 1.66 2.19 1.72 | 1.98 | | | | | | |
| μ, mm ⁻¹ 2.86 4.76 2.28 | 8.05 | | | | | | |
| F(000) 424 340 584 | 728 | | | | | | |
| Index range hkl $-20 < h < 20$ $-8 < h < 8$ $-15 < h < 15$ -3 | 19 < <i>h</i> < 19 | | | | | | |
| -15 < k < 16 $-10 < k < 9$ $-9 < k < 9$ $-10 < k < 9$ $-9 < k < 9$ $-10 < 10 < 10$ $-10 < k < 9$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10 < 10$ $-10 < 10$ | -9 < <i>k</i> < 12 | | | | | | |
| -16 < l < 14 $-14 < l < 14$ $-19 < l < 19$ -2 | 24 < 1 < 25 | | | | | | |
| R_{int} 0.031 0.029 0.031 | 0.081 | | | | | | |
| Total number of reflec- tions21100392615859 | 21229 | | | | | | |
| Number of reflections428019202650with $F > 4\sigma(F)^*$ | 3766 | | | | | | |
| Number of refined param- 91 118 127 eters | 127 | | | | | | |
| 2θ _{max} , deg 84.0 58 57.4 | 73.8 | | | | | | |
| Weight scheme** $ \left[\sigma^2(F_o^2) + (0.0558P)^2 \right]^{-1} \left[\sigma^2(F_o^2) + [\sigma^2(F_o^2) + [\sigma^2(F_o^2) + (0.0717P)^2 + 0.9531P]^{-1} \right] \left[\sigma^2(F_o^2) + [\sigma^2(F_o^2) + (0.0558P)^2 - (0.0558P)^2 + 0.9531P]^{-1} \right] $ | $(5^{2}(F_{o}^{2}) + (100)^{2}(F_{o}^{2})^{-1})$ | | | | | | |
| $R(F > 4\pi(F)) \qquad \qquad 0.038 \qquad \qquad 0.047 \qquad \qquad 0.028 \qquad \qquad (0.0479P)^{-} + 0.9P]^{-}$ | 0.060 | | | | | | |
| wR = 0.033 = 0.020 = 0.020 | 0.107 | | | | | | |
| Goodness-of-fit 1.05 1.09 0.99 | 1.07 | | | | | | |
| $\Delta \rho_{-}/\Delta \rho_{-} e^{A^{-3}}$ 159/-074 087/-109 128/-042 0 | 81/-0.76 | | | | | | |

Table 1. Crystallographic data and experimental details for structures I-IV

Notes: * The correction to the Lorentz and polarization factors was applied.

** $P = (F_o^2 + 2F_c^2)/3.$



Fig. 1. Fragment of structure **I**. Selected bond lengths and angles: C(2)=C(3) 1.373(2) Å, C(3)C(2)C(1) 124.04(12)°; $C(2)^{i}CuC(3)^{ii}$ 38.97(6)°, Cu-m 1.9397(7) Å (*m* is the middle of the C=C bond); Cu-Cl 2.3168(9) Å, $Cu-Cl^{ii}$ 2.6437(10) Å, N(1)C(1)C(2)C(3) 98.90(14)°.(*i*) *x*, -y + 1/2, z + 1/2; (*ii*) -x, -y, -z + 2.



Fig. 2. Structure **II**. Selected bond lengths and bond angles: Cu(1)–N(1) 1.939(4), Cu–*m* 1.9969(5), C(5)=C(6) 1.353(6) Å; C(6)C(5)C(4) 122.4(4)°, C(6)^{*iii*}Cu(2)C(5)^{*iii*} 37.42(16)°. (*iii*) *x*, *y*, *z* – 1.

RUSSIAN JOURNAL OF COORDINATION CHEMISTRY Vol. 38 No. 2 2012



Fig. 3. Structural fragments of compounds **III** and **IV**. Selected geometric parameters for **III**: C(2)=C(3) 1.374(3), C(6)=C(5) 1.315(3) Å; C(3)Cu(1)C(2) 38.87(8)°; Cu-*m* 1.9467(6) Å; Cu(1)-Cl(1) 2.2830(12), Cu(1)-Cl(2) 2.2510(10) Å; N(1)C(1)C(2)C(3) 145.77(2)°, N(1)C(7)C(8)C(9) 172.37(2)°, N(1)C(4)C(5)C(6) 115.9(2)°. Selected geometric parameters for **IV**: Cu(1)-Br(1) 2.4154(13), Cu(1)-Br(1)^{*i*} 2.8904(10), C(2)=C(3) 1.365(5), C(6)=C(5) 1.297(6) Å; C(3)Cu(1)C(2) 38.13(13)°, Br(2)Cu(1)Br(1) 107.65(2)°, Br(2)Cu(1)Br(1)^{*i*} 92.92(4)°, Br(1)Cu(1)Br(1)^{*i*} 102.97(3)°. Cu-*m* 1.9738(7) Å; N(1)C(1)C(2)C(3) 146.5(3)°, N(1)C(4)C(5)C(6) 117.0(4)°. (*i*v) -*x* + 2, *y* + 1/2, -*z* + 1/2.

does not participate in metal atom coordination, is shortened to 1.315(3) Å in **III** and to 1.297(6) Å in **IV**.

As is known, hydrogen bonds play an important role in the formation and stabilization of a certain structure [20, 21]. Their role is also substantial in the strengthening of the structures of complexes I-IV, which is especially noticeable in ionic compounds II-IV (Table 2).

| Compound | Contact D–H···A | Distance, Å | | | Angle D–H…A, | Coordinates of stom A |
|----------|--------------------------|-------------|------|----------|--------------|--------------------------------|
| Compound | | D–H | Н…А | D····A | deg | Coordinates of atom A |
| Ι | N(1)-H(1)····Cl | 0.93 | 2.56 | 3.369(2) | 146 | -x, y - 1/2, -z + 3/2 |
| | $C(4)-H(4B)\cdots N(2)$ | 0.99 | 2.54 | 3.380(2) | 143 | -x - 1, y + 1/2, -z + 3/2 |
| II | $N(2)-H(1)\cdots Cl(2)$ | 0.90 | 2.48 | 3.222(3) | 141 | 1 - x, 1 - y, 1 - z |
| | $N(2)-H(2)\cdots Cl(1)$ | 0.90 | 2.44 | 3.215(3) | 145 | 2 - x, 1 - y, 1 - z |
| | C(3) - H(6) - Cl(3) | 0.97 | 2.82 | 3.590(4) | 137 | 2 - x, -y, 1 - z |
| | $C(6)-H(10)\cdots Cl(1)$ | 0.93 | 2.94 | 3.860(5) | 169 | 2 - x, -y, 1 - z |
| | $C(6)-H(11)\cdots Cl(1)$ | 0.93 | 2.62 | 3.550(6) | 175 | 3 + x, y, 1 + z |
| III | N(1)-H(1)-Cl(1) | 0.93 | 2.31 | 3.142(2) | 149 | <i>x</i> , <i>y</i> , <i>z</i> |
| | $C(1)-H(1A)\cdots Cl(1)$ | 0.99 | 2.81 | 3.553(2) | 132 | -x, y + 1/2, -z + 3/2 |
| | C(1) - H(1B) - N(2) | 0.99 | 2.52 | 3.364(3) | 143 | x, -y + 5/2, z - 1/2 |
| | $C(3)-H(3A)\cdots Cl(1)$ | 0.95 | 2.79 | 3.721(3) | 168 | x, y + 1, z |
| | $C(5)-H(5)\cdots N(2)$ | 0.95 | 2.60 | 3.539(3) | 169 | -x + 1, -y + 2, -z + 2 |
| IV | N(1) - H(1) - Br(1) | 0.92 | 2.50 | 3.305(3) | 146 | <i>x</i> , <i>y</i> , <i>z</i> |
| | C(1) - H(1B) - N(2) | 0.98 | 2.59 | 3.421(5) | 142 | x, -y + 1/2, z - 1/2 |
| | $C(3)-H(3A\cdots Br(1)$ | 0.94 | 2.88 | 3.802(4) | 166 | x, y + 1, z |
| | $C(8)-H(8A)\cdots Br(2)$ | 0.98 | 2.84 | 3.781(4) | 161 | -x+2, y+1/2, -z+1/2 |

Table 2. Hydrogen bonding geometry for compounds I-IV

RUSSIAN JOURNAL OF COORDINATION CHEMISTRY Vol. 38 No. 2 2012

It is most likely that the efficient hydrogen bonds $(C)H\cdots N(\equiv C)$ in structures **III** and **IV** diminishing the donating properties of the nitrile nitrogen atom in H⁺Dapn (and partially in H⁺Apn) favors the unexpected inert behavior of the cyano group towards the Cu(I) atom.

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REFERENCES

- Mys'kiv, M.G. and Oliinik, V.V., Koord. Khim., 1995, vol. 21, no. 4, p. 290.
- 2. Oliinik, V.V., Mys'kiv, M.G., and Pecharskii, V.K., *Zh. Strukt. Khim.*, 1993, vol. 34, no. 6, p. 43.
- Mys'kiv, M.G., Goreshnik, E.A., Pecharskii, V.K., and Oliinik, V.V., *Zh. Strukt. Khim.*, 1994, vol. 35, no. 1, p. 90.
- Fayad, Kh., Sobolev, A.N., and Mys'kiv, M.G., *Koord. Khim.*, 1991, vol. 17, no. 9, p. 1245.
- 5. Zavalii, P.Yu., Mys'kiv, M.G., and Gladyshevskii, E.I., *Kristallografiya*, 1986, vol. 31, no. 1, p. 88.
- Mys'kiv, M.G., Zavalii, P.Yu., Oliinik, V.V., and Fundamenskii, V.S., *Zh. Strukt. Khim.*, 1990, vol. 31, no. 4, p. 85.
- 7. Mys'kiv, M.G., Oliinik, V.V., and Zavalii, P.Yu., *Zh. Strukt. Khim.*, 1988, vol. 29, no. 4, p. 113.

- Oliinik, V.V., Mys'kiv, M.G., Zavalii, P.Yu., and Mazus, M.D., *Metalloorg. Khim.*, 1988, vol. 1, no. 6, p. 1247.
- Oliinik, V.V., Zavalii, P.Yu., Mys'kiv, M.G., and Fundamensiki, V.S., *Koord. Khim.*, 1986, vol. 12, no. 8, p. 1141.
- 10. Filinchuk, Ya.E., Oliinik, V.V., and Davydov, V.M., *Russ. J. Coord. Chem.*, 1997, vol. 23, no. 11, p. 791.
- 11. Olijnyk, V., Glowiak, T., and Mys'kiv, M., J. Chem. Cryst., 1995, vol. 25, no. 10, p. 621.
- 12. Filinchuk, Ya.E. and Mys'kiv, M., Pol. J. Chem., 2000, vol. 74, no. 7, p. 927.
- 13. Houben-Weil, *Methoden der organischen Chemie*, Stuttgart: Georg Thieme, vol. 4, 1949.
- 14. Ukrainian Patent no. 25450, Byull. Izobret., 1998, no. 6.
- CrysAlis RED. Version 1.171.31.8 (Release 12-01-2007. CrysAlis 171.NET), Oxford: Oxford Diffraction Ltd., 2007.
- 16. *Crystal Clear*, Woodlands (Texas, USA): Rigaku Corporation, 1999.
- 17. Clark, R.C. and Reid, J.S., Acta Crystallogr., Sect. A: Found. Crystallogr., 1995, vol. 51, no. 6, p. 887.
- 18. Sheldrick, G.M., SHELXS-97 and SHELXL-97. Program for the Solution and Refinement of Crystal Structures, Göttingen (Germany): Univ. of Göttingen, 1997.
- 19. Oliinik, V.V., Mys'kiv, M.G., and Aksel'rud, L.G., *Russ. J. Coord. Chem.*, 1996, vol. 22, no. 1, p. 67.
- 20. Desiraju, G.R., Acc. Chem. Res., 2002, vol. 35, no. 7, p. 565.
- 21. Beauchamp, D.A. and Loeb, S.J., *Chem.-Eur. J.*, 2002, vol. 8, no. 22, p. 5084.