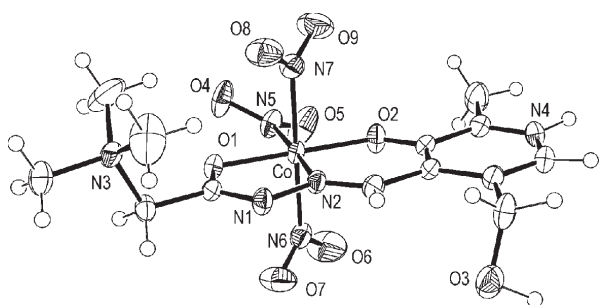


s13.m38.p17 **Synthesis and Crystal Structure of [Co(L)(NO₂)₃]₂H₂O (L=piridoxylene Girar-T hydrazone).** Ljiljana Vojinovic^a, Sladana Novakovic^b, Goran Bogdanovic^b, Vukadin Leovac^a, Valerija Cesljevic^a and Katalin Mészáros Szécsényi^a, ^aDepartment of Chemistry, University of Novi Sad, ^bInstitute of Nuclear Science "Vinca", Serbia and Montenegro. E-mail: ljilja@ih.ns.ac.yu

Keywords: Cobalt(III) complexes; Girar-T hydrazone; Crystal structure

Dark red monocrystals of title complex were prepared by the reaction of water solutions of Na₃[Co(NO₂)₆] and L×2H₂O in mole ratio 1:1. The Co(III) is sited in an octahedral environment. The equatorial plane is formed by a tridentate coordination of the ligand (L) and one NO₂ molecule, while the other two NO₂ molecules are in axial positions. The ligand is coordinated to the Co(III) atom through O3, N1 and O4 atoms, forming two fused chelate rings, one being five-membered and one a six-membered one. All atoms in the chelate and phenyl rings are approximately coplanar. The angle between the mean plane of the equatorial NO₂ and the mean plane defined by the two chelate rings is 28.1(3)°. The Co-N bond lengths for the two axial NO₂ are about 0.03 Å longer than that with the equatorial NO₂. The two axial NO₂ are coplanar. The complex molecule possesses eight oxygen atoms capable of acting as proton acceptors in the hydrogen bonds, but there are only two groups (one N-H and one O-H) able to participate as proton donors. As a consequence, six oxygen atoms from NO₂ ligands form weak C-H...O hydrogen bonds with only one exception for O4 atom which participates in two strong O-H...O hydrogen bonds. Because of that the N-O4 bond is significantly longer than the rest of N-O bonds in the NO₂ ligands.

Data were collected on an Enraf-Nonius CAD-4 diffractometer using Mo K α radiation ($\lambda = 0.71069$ Å) and $\omega/2\theta$ scans in 1.41 to 25.47° θ range and were corrected for Lorentz and polarization effects. The structure was solved by direct methods and difference-Fourier methods, and refined by full matrix least-squares using SHELXL97 program to a final R = 0.0431 for 2938 reflections with I > 2 σ _I. Crystallographic data: CoC₁₃H₂₂N₇O₁₀, Mr = 495.3, monoclinic, space group P 2₁/c, a = 14.936(3) Å, b = 7.773(3) Å, c = 17.463(3) Å, $\beta = 104.26(3)^\circ$, V = 1964.9(9) Å³, Z = 4, D_c = 1.674 Mg/m³, $\mu(\text{MoK}\alpha) = 0.944$ mm⁻¹, Mo K α radiation, $\lambda = 0.71073$ Å, GooF = 1.055.

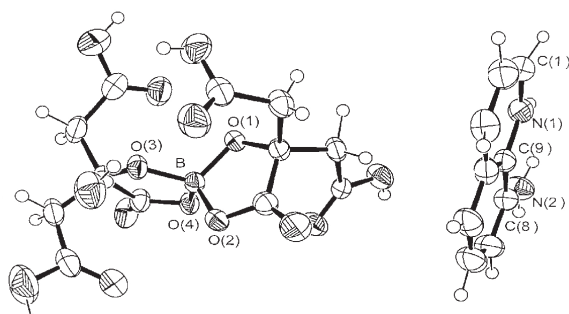


The structure of [Co(L)(NO₂)₃] with 50% probability ellipsoid.

s13.m38.p18 **Synthesis and Crystal Structure of 8-Aminoquinolinium Bis(citrate)borate Tetrahydrate.** I. Zviedre^a, S. Belyakov^b, J. Schwartz^a, ^aInstitute of Inorganic Chemistry of the Riga Technical University, 34 Miera Str. Salaspils, LV-2169, LATVIA, ^bLatvian Institute of Organic Synthesis, 21 Aizkraukles Str. Riga, LV-1006, Latvia. E-mail: irena@nki.lv

Keywords: Crystal structure; Boron coordination compounds

A new complex 8-aminoquinolinium bis(citrate)borate tetrahydrate C₉H₉N₂[(C₆H₆O₇)₂B]·4H₂O has been synthesized [1] in continuation of the systematic crystal chemical investigations of boron coordination compounds with organic cations. Well-shaped single crystals of it have been obtained and their full X-ray structural investigation has been carried out. The crystal structure is formed from univalent cations (HL)⁺, spiran-type complex anions [(C₆H₆O₇)₂B]⁻ and four crystallization water molecules. The complex anions are formed



by two citric acid residues bidentically coordinated to the central symmetrical distorted BO₄ tetrahedron. The bonds B-O(carb.) (aver. 1.494(3) Å) are systematically longer than B-O(hydr.) (aver. 1.461(3) Å). The heterocyclic bond angles at boron atom (104.47(16)^o in average) are the smallest ones from the other O-B-O angles (112.03(17)^o in average). The average bond lengths within the citric acid residue: C(sp³)-O(H) 1.417(3); in the terminal carboxyl groups C(sp²)=O aver. 1.206(4), C(sp²)-OH 1.316(4) Å. The length of the C-C bond of C(sp³)-C(sp³) type is 1.538(3) Å; that of C(sp²)-(sp³) 1.496(4) Å. The boron-containing five-membered rings C-O-B-O-C are planar in the limits of ± 0.02 Å. The oxygen atoms in carbonyl groups adjacent to heterocyclic system are coplanar to the atoms of cycle. The dihedral angle corresponding to the crossing of two main mean-square planes of heterocycles is 92.35(10)^o. The cation is protonated at the nitrogen atom N(1). The values of the distances N-C are: N(1)-C(1) 1.330(5) Å; N(1)-C(9) 1.361(4) Å; C(ar)-N(2)H₂ - 1.335(5) Å. The quinolinium rings are planar in the limits of ± 0.01 Å. Amino group is declined from the plane by 0.10 Å. All four water molecules, all hydroxyl groups of complex anion terminal carboxyl groups, amino group and protonated nitrogen atom participates in fashion of the hydrogen bond system. Crystals are triclinic, space group P $\bar{1}$: a=9.601(1) Å, b=10.688(2) Å, c=15.608(3) Å; $\alpha=88.568(1)^\circ$, $\beta=76.740(1)^\circ$, $\gamma=63.523(2)^\circ$; V=1389.9(3) Å³; Z=2; d_x=1.453 gcm⁻³; R=0.0433, wR2=0.0897 for 3467 independent reflections with I \geq 2 σ (I) (MoK α radiation).

[1] I. Zviedre, D. Zaruma. *Latvian Journal of Chemistry*, (2003), N4, 387.