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Crystal structure of nickel(II) complex with 2-{1-[6-(1-selenosemicarbozonoethyl)-2-pyridyl]ethylidene}hydrazine carbonitrile

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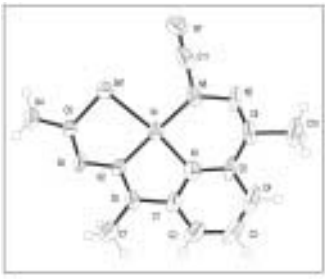
Keywords: selenium compounds, coordination compounds, crystal and molecular structure

A nickel(II) complex with 2-{1-[6-(1-selenosemicarbozonoethyl)-2-pyridyl]ethylidene}hydrazine carbonitrile was synthesized by reaction of Ni(II) acetate and 2,6-diacetylpyridine bis(selenosemicarbazone) (H_2dapsesc) in ethanolic solution, and purified by dissolution in DMF and precipitation by slow addition of ethanol.
The obtained complex was characterized by elemental analysis and spectroscopic measurements (IR, ^{1}H NMR, ^{13}C NMR), which showed that modification of one side chain of the symmetrical H_2dapsesc ligand with elimination of hydrogen selenide occurred during the reaction. The structure of the complex was determined by X-ray analysis. The unsymmetrical bideprotonated ligand coordinates the metal in a square planar geometry by a N_3Se chelation, which generates two five-membered and one six-membered chelation rings. The overall neutral complex is planar and the crystal packing is based on N-H ...N hydrogen bonds and on long Ni ...Ni contacts (3.71Å).



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X-ray crystal and molecular structures of thymidine and guanosine 3',5'-cyclic monophosphates

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Keywords: biological phosphates, cyclic phosphates, X-ray crystal and molecular structure

The biological role of nucleoside 3',5'-cyclic monophosphates has been well known for many years. Some of them, like cAMP, have been extensively investigated as regards their structure for long, but cTMP has never been studied by the use of X-ray crystallography [1]. The results of structural investigations on thymidine 3',5'-cyclic monophosphate sodium salt heptahydrate, Na(cTMP)•7H_2O, along with the guanosine 3',5'-cyclic monophosphate sodium salt tetrahydrate reinvestigated, Na(cGMP)•4H_2O, and guanosine 3',5'-cyclic monophosphate in the form of hydrated acid, cGMP•4H_2O, will be presented. The Na^+ salts crystallize with one anion in the asymmetric unit, while the acid cGMP - with one zwitterion. The orientation of the nucleobase with respect to the sugar ring is different in cTMP and cGMP crystals, i.e. *anti* and *syn*, respectively. The sugars (deoxyribose in cTMP and ribose in cGMP) both exist in twist conformation ($_4T^3$), which is characteristic of the cyclic nucleotides. The six-membered dioxaphosphorinane O/P/O/C/C/C rings adopt a distorted chair conformation with the significant flattening of the ring at the P atom. The coordination spheres of sodium cations in both salts are composed of six O atoms: one carbonyl O2 and five from water molecules (in Na(cTMP)•7H_2O), or all the six O atoms from water molecules (in Na(cGMP)•4H_2O). Na^+ ions and water molecules in the latter form $[Na(H_2O)_4]_n^+$ polymeric chains, among which the cGMP anions are located. The arrangement of the cGMP zwitterions in the crystal of the acid form is very similar to that observed in the salt form due to location of water molecules in the channels occupied by $[Na(H_2O)_4]_n^+$ chains in the crystal of the salt. That is accompanied by the similar molecular geometry of the cGMP in both salt and acid forms, which is shown in Fig. 1.

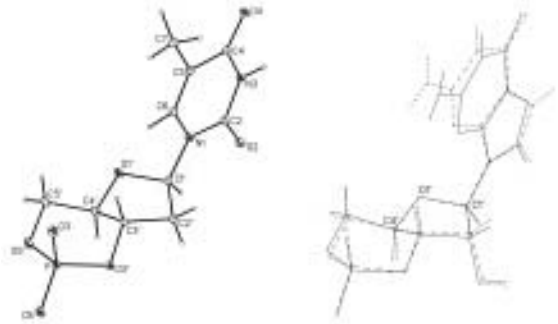

Fig. 1. The molecular geometry of the anion in cTMP (left) along with the comparison of the cGMP structures obtained from the salt and acid crystals (right).

[1] Cambridge Structural Database (*CSD*, Version 5.27 of November 2005); Allen, F. H. *Acta Cryst.* 2002, *B58*, 380-388.