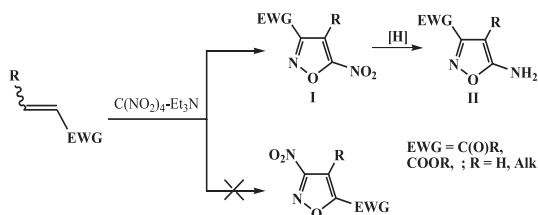


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Recently we have elaborated a new efficient method for the heterocyclization of electrophilic alkenes by the action of tetranitromethane (TNM) in the presence of triethylamine which afforded functionalized nitroisoxazoles [1]. The present investigation deals with X-ray analysis of thus obtained nitroisoxazoles and corresponding products of their reduction, aminoisoxazoles, for a reliable confirmation of their structure. X-ray analysis established that nitro- and amino-groups were located at position 5 of the isoxazole cycle. Thus, a new heterocyclization of electrophilic alkenes under TNM in the presence of triethylamine afforded 5-nitrosubstituted isoxazoles. In present time, X-ray study for first step of this reaction was made for five nitroisoxazoles I (see scheme). Second step of reaction was studied only for aminoisoxazole II (R = H, EWG = C(O)Et). The crystal structures of all nitroisoxazoles are formed only by Van der Waals force – no any non-classical hydrogen bonds found. Classical hydrogen (with amino-group as donor) bonds are found in crystal structure of aminoisoxazole.



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Keywords: small organic molecules, nitroisoxazoles, aminoisoxazoles

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New titanium nitrido complexes from $\{[TiCp^*(\mu-NH)]_3(\mu_3-N)\}$ and methyltriflate

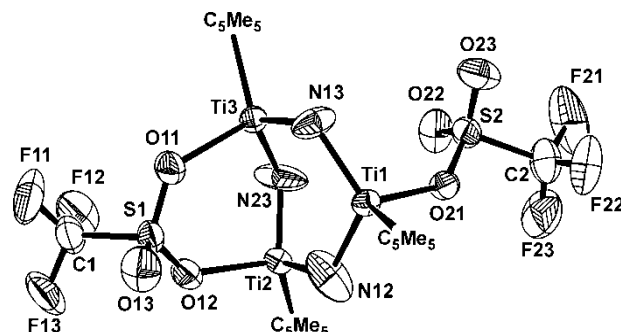
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Transition-metal nitrido complexes have been studied extensively in the past few decades. While nitrido complexes of Group 6-8 metals usually bear the nitrido ligands as a terminal functionality, the analogous derivatives of early transition metals exhibit polynuclear structures with μ_n -nitrido ligands bridging two or more metal centers. In many cases, the nitrido group is accompanied by amido or imido ligands, like those found in the imido-nitrido titanium complex $[\{TiCp^*(\mu-NH)\}_3(\mu_3-N)]$ (A) ($Cp^* = \eta^5-C_5Me_5$) [1]. Over the last few years we have been studying the capability of compound A to act as a tridentate ligand, as well as the reactivity of the nitrido group towards nucleophilic reagents [2] or Lewis acids [3].

In this communication, we will present the synthesis of new titanium(IV) nitrido complexes by treatment of compound A with an electrophilic chemical such as methyltrifluoromethanesulfonate (methyltriflate) in different molar ratios. The X-ray crystal structures determined for these compounds will be discussed. For instance, we show in figure the molecular structure of the singular trinuclear

imido-nitrido titanium complex obtained through reaction of A with 4 equivalents of methyltriflate. This compound presents nitrido, imido and triflate groups bridging the titanium centers.

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Keywords: titanium, nitrido ligand, methyltriflate

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Crystal structure of a carbacylamidophosphate organotin compound

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Organotin compounds continue to attract significant attention, owing to their applications in agriculture, biology, catalysis, organic synthesis, etc [1-3]. Besides, carbacylamidophosphates, which have $-C(O)NHP(O)-$ in their molecular core units are potential bidentate O,O-donor chelating ligands for metal ions, particularly for lanthanides [4]. Previously, diorganotin(IV) compounds of the N-benzoyl derivatives of carbacylamidophosphates [5,6] were synthesized, in which the coordination of the ligands were only through the oxygen atom of the phosphoryl group. The solid state results showed the monomeric *all-trans* octahedral complexes due to the fewer steric hindrance in the *trans* geometry. However, to our knowledge no organotin complexes of N-acetyl derivatives of carbacylamidophosphates are known.

In this work, N-monochloroacetyl, N', N''-bis(isopropyl) phosphoric triamide and its complex with $SnCl_2Me_2$ have been synthesized and characterized. The structure of complex has been determined by X-ray crystallography. Colorless crystals were obtained from a concentrated toluene solution at room temperature. The compound crystallizes in the triclinic system with space group $P\bar{1}$. Coordination of the ligand to tin (IV) occurs through both oxygen atoms of phosphoryl and carbonyl groups. This complex is binuclear structure with two non-equivalent Sn atoms. One adopts a distorted *trans*(C,C) *cis*(O_p,O_p) *cis*(Cl,Cl) octahedral configuration, while the other adopts a distorted trigonal bipyramidal geometry composed of two methyls, two chlorine atoms and carbonyl group.