

o.m12.p19 Coordination peculiarities of 5-pyridinesubstituted N-methyl isoxazolidines: syntheses, characterization and crystal structures of Pd(II), Zn(II) complexes. A.B. Lysenko^a, S.V. Shishkina^b, O.V. Shishkin^b, F.L. Ortiz^c, R.D. Lampeka^a. ^aDepartment of Inorganic Chemistry, Kiev University, Vladimirska St. 64, Kiev, Ukraine. ^bScientific Research Department of Alkali Halide Crystals, STC "Institute for Single Crystals", Lenina st., 60, 310001 Khar'kov, Ukraine. ^cScientific Experimental Department of Organic Chemistry, Crta.Sacramento s/n 04120 Almeria, Spain.

Keywords: isoxazolidines, complexes, syntheses.

Our interest in the synthesis and studies of isoxazolidine derivatives because of their relevance in organic synthesis^{1,2} and their role as biologically active substances has led us to consider co-ordination abilities these compounds, which have not been studied yet. The metal complexes with isoxazolidines as ligands are attracted our attention as one of the methods of purification and separation of isomeric isoxazolidines that is also important from a biological point of view. A few Pd(II), Zn(II) complexes with 5-pyridinesubstituted N-methyl isoxazolidines (2-(2-methyl-3-phenyl-isoxazolidin-5-yl)-pyridine (**L**¹), 2-(2-methyl-3-ferrocenyl-isoxazolidin-5-yl)-pyridine (**L**²), 4-(2-methyl-3-phenyl-isoxazolidin-5-yl)-pyridine (**L**³)) have been prepared and characterised by ¹H, ¹³C, ³¹P 1-D and HMBC, ROESY, COSY 2-D NMR spectroscopy. The configurations of isoxazolidines have been assigned by analysis of NMR coupling constants and by nuclear Overhauser effect. It has been shown that isoxazolidines may act as bidentate (N,N-; O,N-) or monodentate (N-pyridine) donors. The molecular structure of *trans*-[PdCl₂(**L**²)PEt₃] has been established by means X-ray crystallography. The crystal structure of *trans*-[PdCl₂(**L**²)PEt₃] contains square-planar coordination polyhedrons of palladium ions. The **L**² is coordinated in a monodentate manner *via* the pyridine nitrogen atom. The isoxazolidine fragment has an envelope conformation.

o.m12.p20 Crystal structures and absolute configurations of two new amino acids obtained from monoterpene ketones. J. Holband¹, G. Wójcik¹, S. Lochynski², J. Kuldo². ¹Institute of Physical and Theoretical Chemistry; ²Institute of Organic Chemistry, Biochemistry and Biotechnology, Wrocław University of Technology, Wyb. Wyspińskiego 27, 50-370 Wrocław, Poland.

Keywords: terpene, stereogenic center, crystal structure.

Terpenes are known as useful chiral synthons in syntheses of structural variety of optically active compounds displaying interesting biological properties such as local anesthetics¹ or antiarrhythmics and cardiodepressive compounds², insect growth³ regulators or pyrethroids⁴. Two terpene substrate ((+)-3-carene and (-)-menthol) were used as synthons for the preparation of amino acids with fixed stereogenic centers. These compounds are potential inhibitors of GABA neuroreceptors.

The main step in synthesis of amino acids from terpenes is the Backmann rearrangement. During this process the inversion of configuration on the amine carbon may occur. X-ray diffraction method seemed to be the best way of determination absolute configuration of chiral, reacting center. Each starting substrate (mentioned above) had at least one fixed, nonreacting, prochiral centers, so we could treat them as reference configurations in our crystal investigations.

X-ray data were collected with MoK α radiation at 298 K on a KUMA KM4 diffractometer equipped with CCD camera. Crystal data: **1**) (1R,2S,2'R)-(+)-2-[2-amino-1-propyl]3,3-dimethylcycloprop-1-ylacetic acid; C₁₀H₁₉NO₂, P2₁, *a*=6.295(1) Å, *b*=6.713(1) Å, *c*=12.836(3) Å, β =100.20(3)°, *V*=533.86(17) Å³, *Z*=2, D_c =1.153 Mg/m³, crystal size 0.90x0.67x0.18 mm, $2\theta \leq 63.18$, 4585 refl. measured, 3238 unique refl., 3087refl. with $I > 2\sigma(I)$, *R*₁=0.048, 131 param. **2**) (3R,6S)-(-)-6-amino-3,7-dimethyloctanoic acid; P2₁2₁2₁, C₁₀H₂₁NO₂, *a*=5.719(1) Å, *b*=10.923(2) Å, *c*=17.389(3) Å, *V*=1086.3(3) Å³, *Z*=4, D_c =1.145 Mg/m³, crystal size 0.59x0.13x0.15 mm, $2\theta \leq 63.73$, 9332 refl. measured, 4365 unique refl., 3182 refl. with $I > 2\sigma(I)$, *R*₁=0.046, 132 parameters.

The structures of amino acids indicated that the amino acid molecules appear as zwitterions. In all the studied crystals a network of medium and weak intermolecular N-H...O hydrogen bonds occur.

Besides, we have determined the crystal structure of the lactame: (1S,3R,7R)-(-)-3,8,8-trimethyl-4-azabicyclo[5.1.0]octan-5-one, which was obtained as an intermediate from (+)-3-carene. The nonreacting, chiral center of our interest has the same configuration as the amino acid **1**. It means that hydrolysis of the lactame does not change the configuration.

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