Лаборатория физико-химических методов анализа строения вещества
Химический факультет МГУ

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Москва, 2012

NMR/MS: Structure elucidation in organic chemistry

Method	Minimal amount of sample, mg		Concentrations range, M
	Routine hardware	***Advanced hardware***	
X-Ray (single crystal)	50	10	-
Nuclear Magnetic Resonance (NMR)	5.0 – 0.01	**10^{-2} – 10^{-6}**	**1.0 – 10^{-6}**
Mass-spectrometry	10^{-3} – 10^{-6}	10^{-6} – 10^{-12}	10^{-5} – 10^{-12}
Microscopy	~ 10^{-3}	10^{-3} – 10^{-6}	-

Joint NMR/MS studies in organic chemistry

ELSEVIER

Available online at www.sciencedirect.com
ScienceDirect
Focus Article, *Mendeleev Commun.*, 2010, **20**, 125–131

Mendeleev
Communications

Mechanistic insight into organic and catalytic reactions by joint studies using mass spectrometry and NMR spectroscopy

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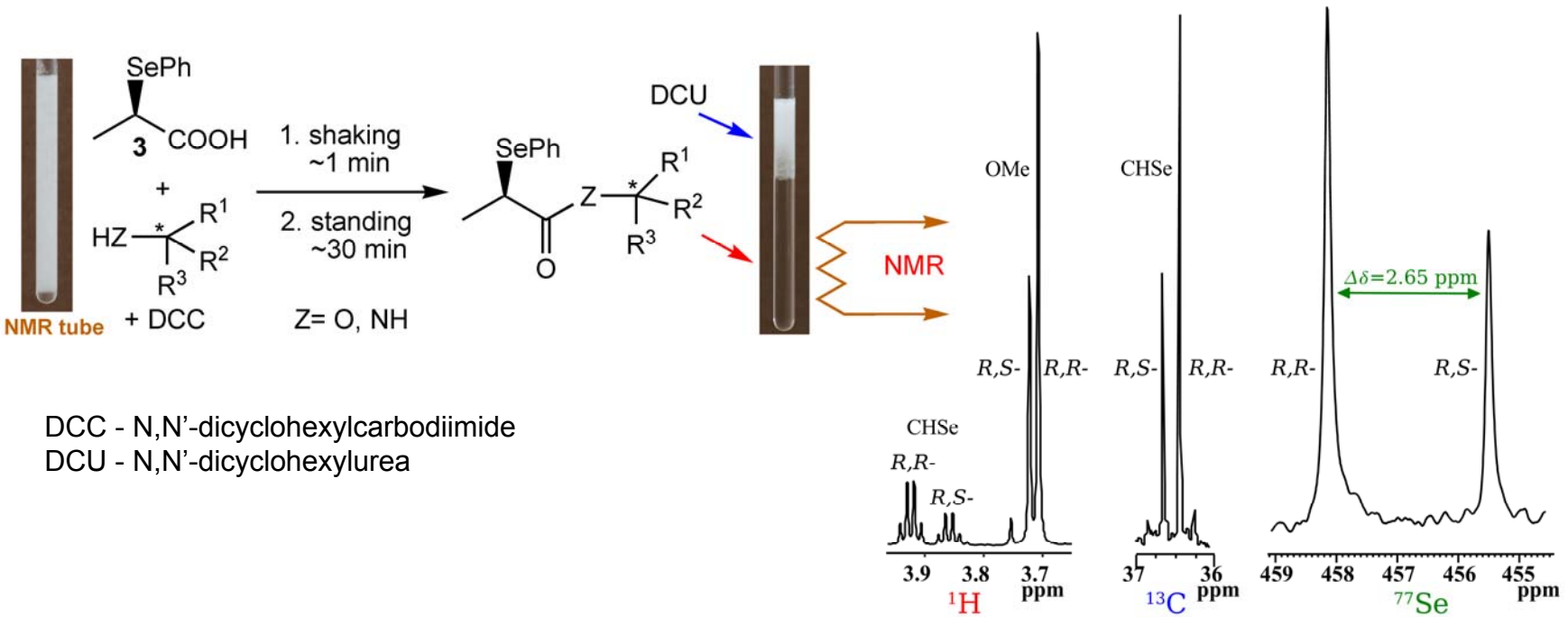
DOI: 10.1016/j.mencom.2010.05.001

Utilization of NMR spectroscopy and mass spectrometry for joint mechanistic and structural studies is a well-known practice. Several opportunities have appeared in recent years because of new hardware development and design of novel experimental procedures. Recent progress in this area and leading examples of new development, as well as already distinguished techniques, are discussed.

Mendeleev Commun., **2010,** 20, 125-131.

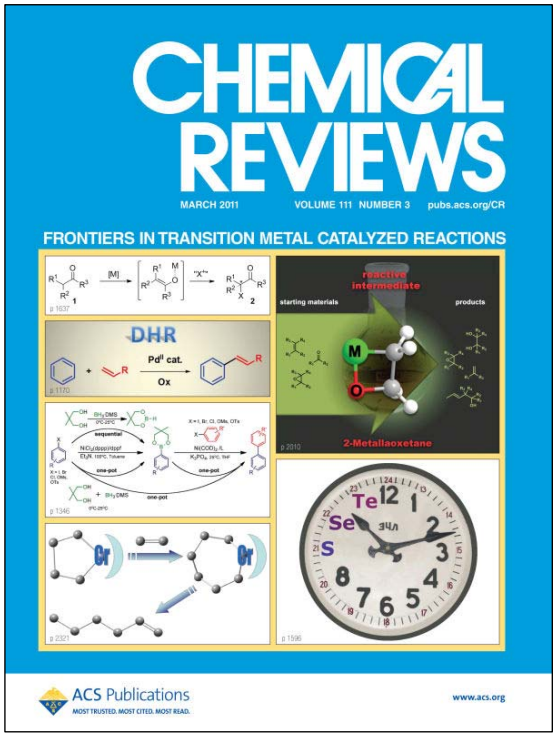
Sensing Chirality by NMR



DCC - N,N'-dicyclohexylcarbodiimide
DCU - N,N'-dicyclohexylurea

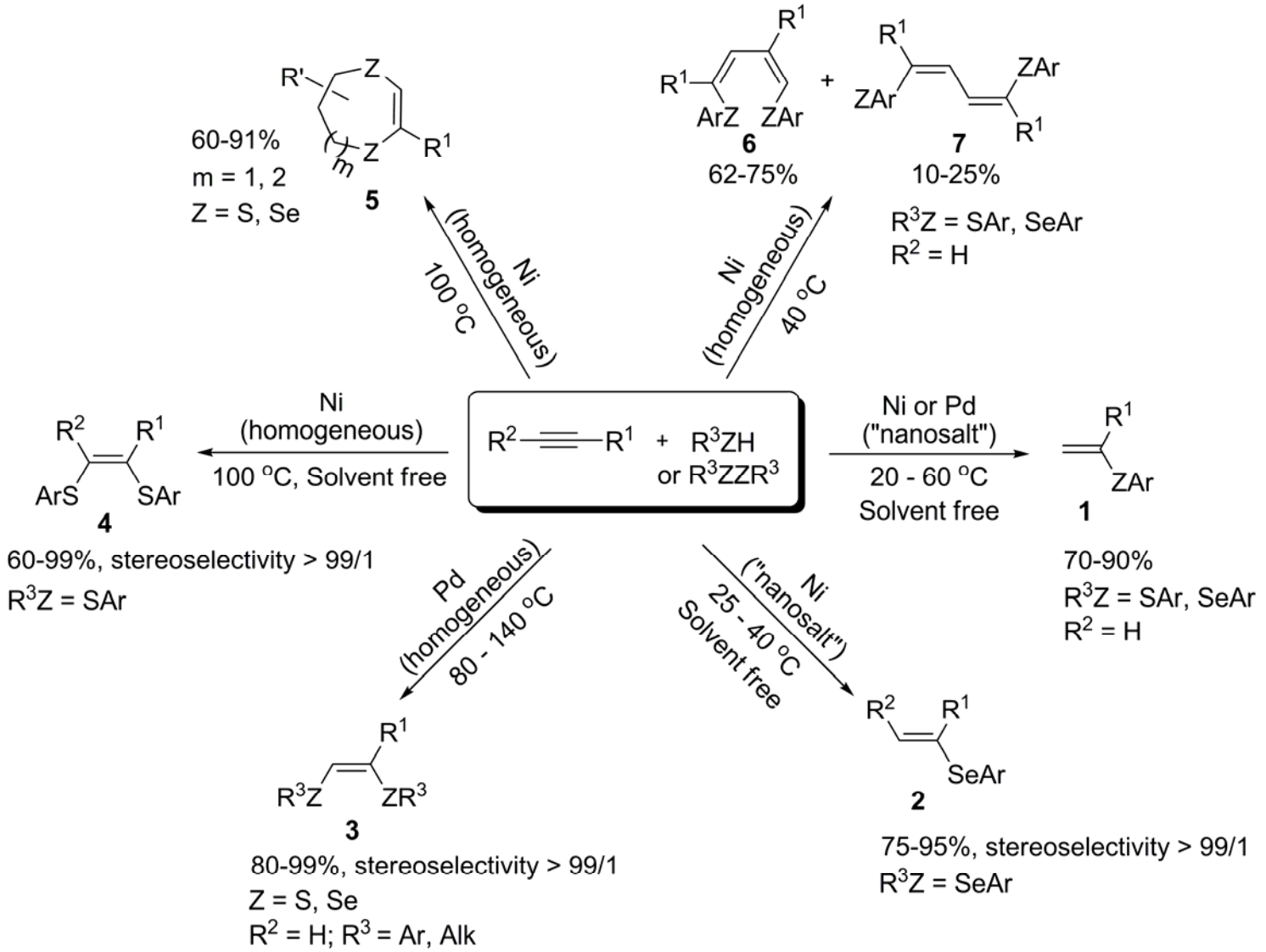
- Easy determination of enantiomeric purity of crude samples and isolated products.
- Monitoring of ee.
- Absolute configuration (if standard is known).
- **Without chromatography!**

Chem. Commun., **2010**, 46, 3212; *Green Chem.*, **2011**, 13, 1735.



"Transition-Metal-Catalyzed C-S, C-Se, and C-Te Bond Formation via Cross-Coupling and Atom-Economic Addition Reactions"
Chem. Rev. **2011**, 111, 1596.

Application in organic synthesis



Review: *Chem. Rev.* **2011**, *111*, 1596.

J|A|C|S
COMMUNICATIONS

Published on Web 05/19/2007

New Approach for Size- and Shape-Controlled Preparation of Pd Nanoparticles with Organic Ligands. Synthesis and Application in Catalysis

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One-dimensional (1D) nanostructures have attracted much attention because of their potential application in numerous fields of science and engineering. Presently, a tremendous amount of research activity is under way for the use of 1D nanostructures (such as nanowires, nanotubes, nanobelts, and nanofibers) as well-defined building units for the fabrication of various nanoscale devices.[1] In addition to the field of nanoscale devices precursors, 1D nanostructures with organic ligands could be of great importance for carrying out highly selective chemical transformations in a catalytic manner.[1d,p] Special procedures have been developed for the preparation of inorganic (1D) nanostructures based on metal chalcogenides M_xZ_y (M = Zn, Sn, In, Cd, Ga, Pb, Pd; Z = O, S, Se).[2,3] However, these methods involve harsh reaction conditions, and they are unsuitable for the preparation of nanostructures with organic ligands. Not surprisingly, the application of 1D nanostructures with organic ligands remains unexplored.[3]

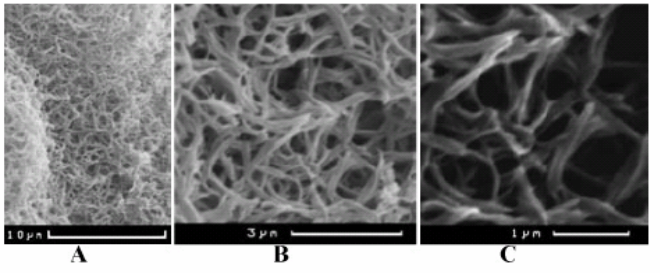

Figure 1. Low magnification (A, 1000×) and high magnification (B, 8000×, C, 16000×) SEM images of $[Pd(SCy)_2]_n$ (**4a**).

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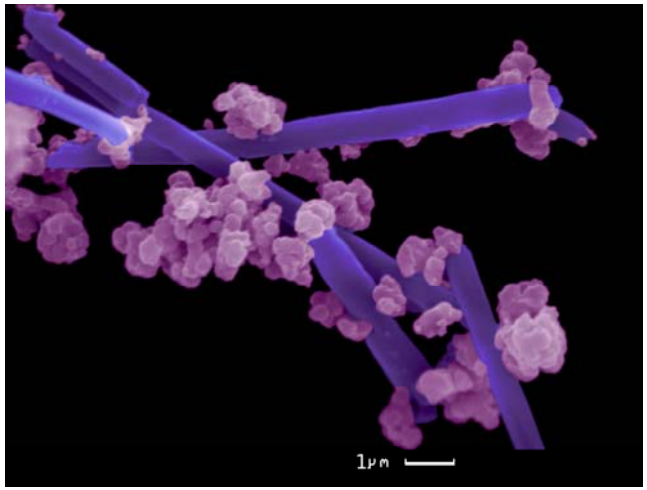
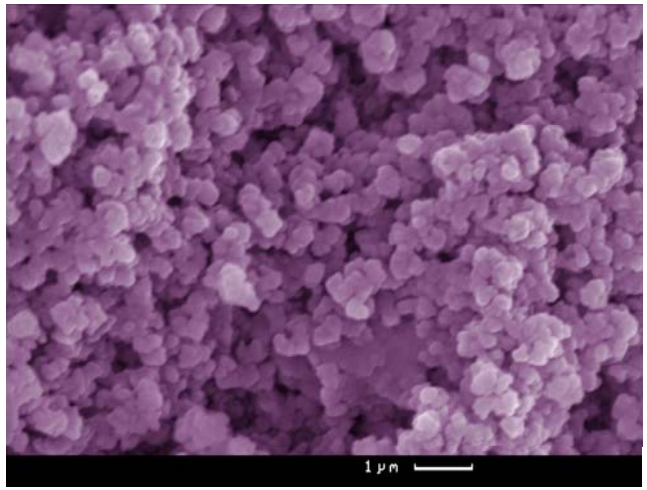
‡ Lomonosov Moscow State University.
§ Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences.
# New Mexico Highlands University.

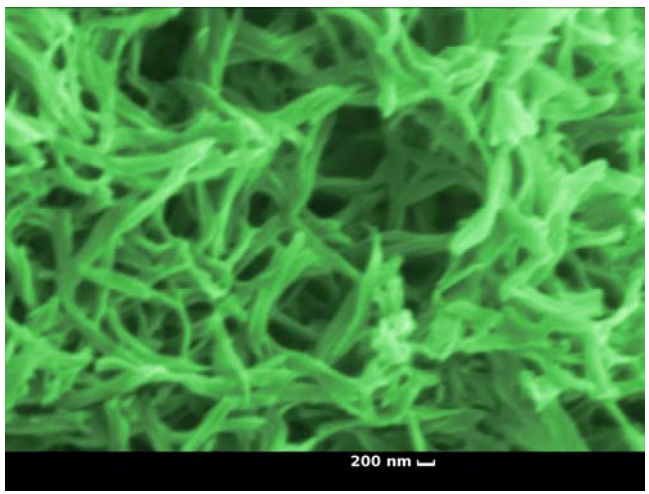
Electron microscopy study of “Nanosalts”



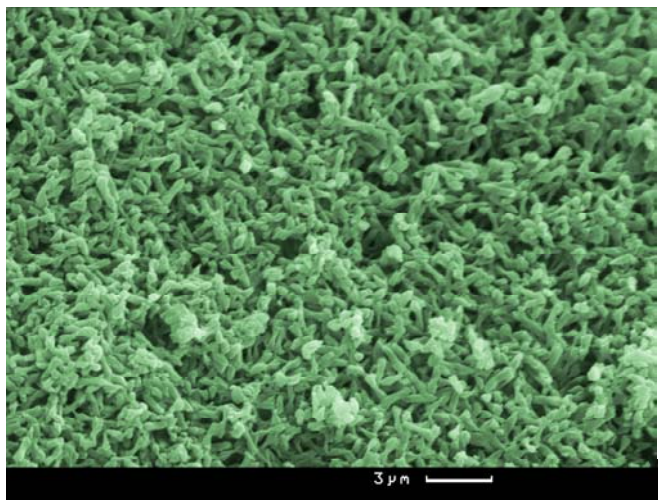
$[Ni(SePh)_2]_n$/support



$[Ni(SePh)_2]_n$



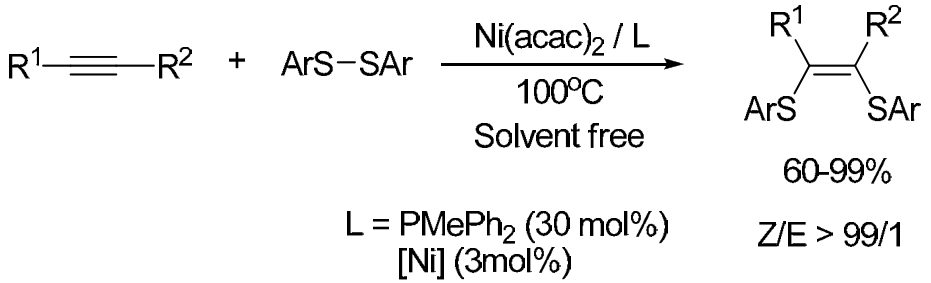
$[Pd(SCy)_2]_n$

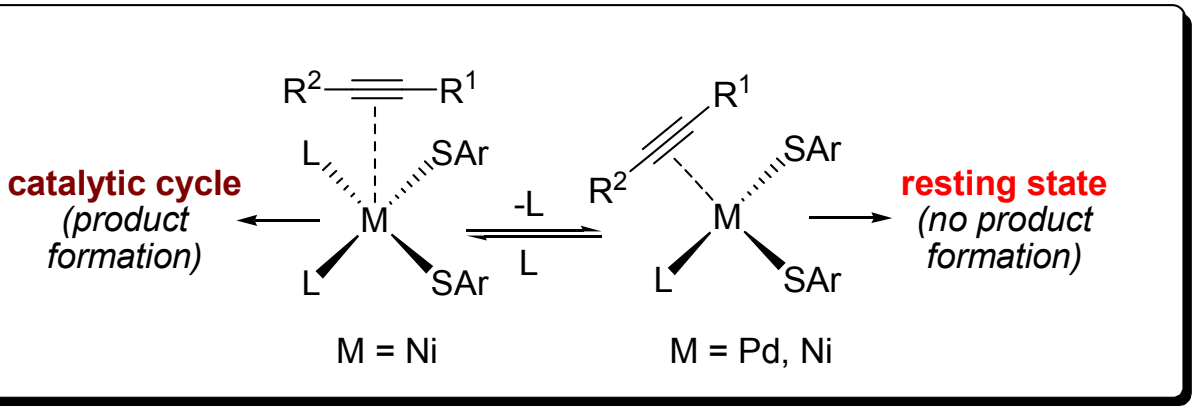


$[Pd(SBn)_2]_n$

Invited Perspective article on “Nanosalts”: *Dalton Trans.*, **2011**, 40, 4011.

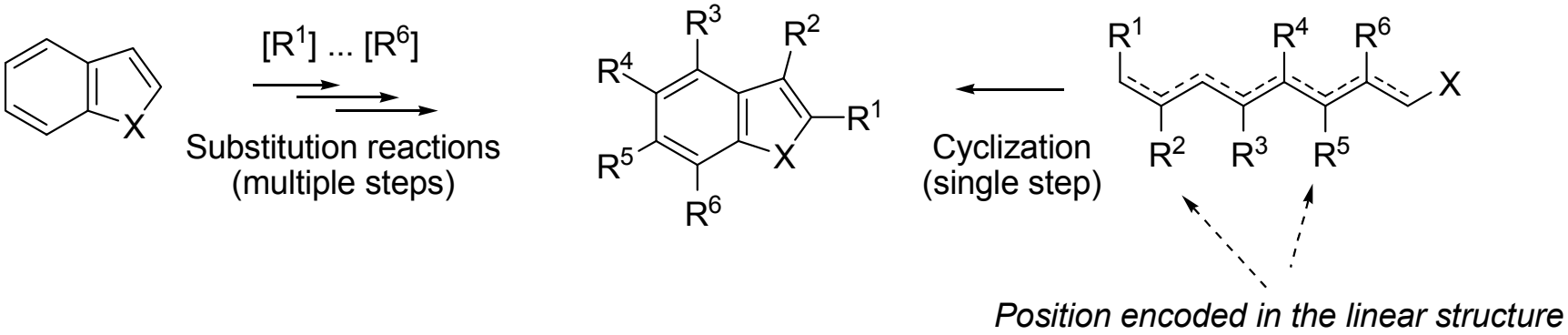
Catalytic addition to internal alkynes





Chem. Eur. J. **2010**, 16, 2063; *Chem. Eur. J.*, **2011**, 17, 12623.

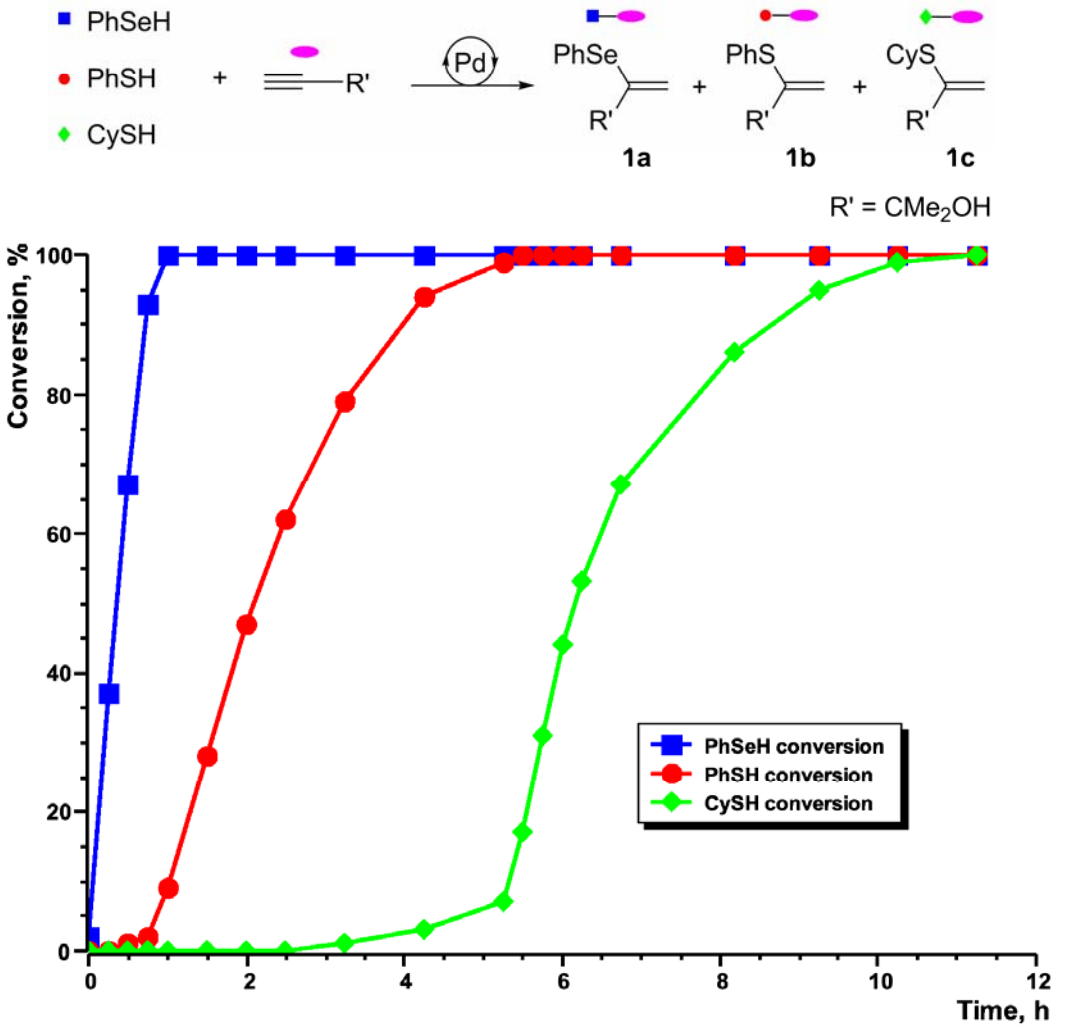
Cycloaddition and cycloaromatization involving alkynes

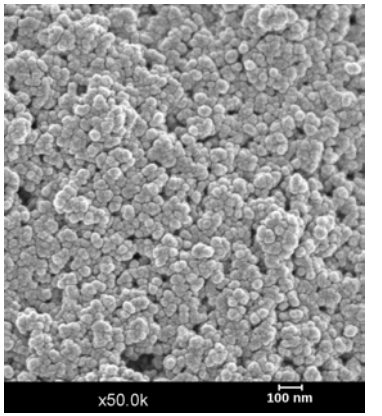


Linear Encoding of Functional Groups in the Synthesis of Aromatic and Heterocyclic Compounds

Chem. Sci., **2011**, 2, 2332.

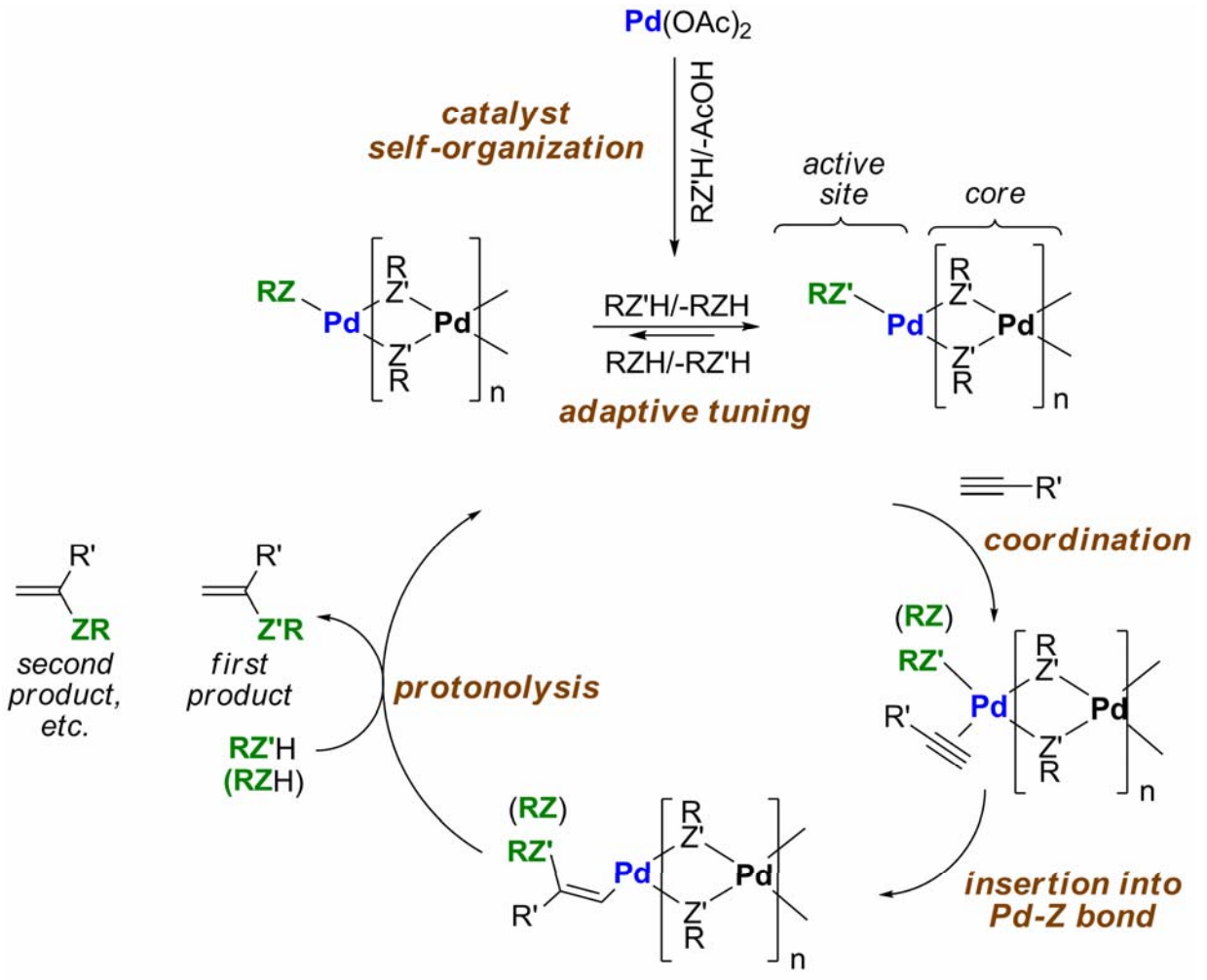
Adaptive tuning of Pd catalysts





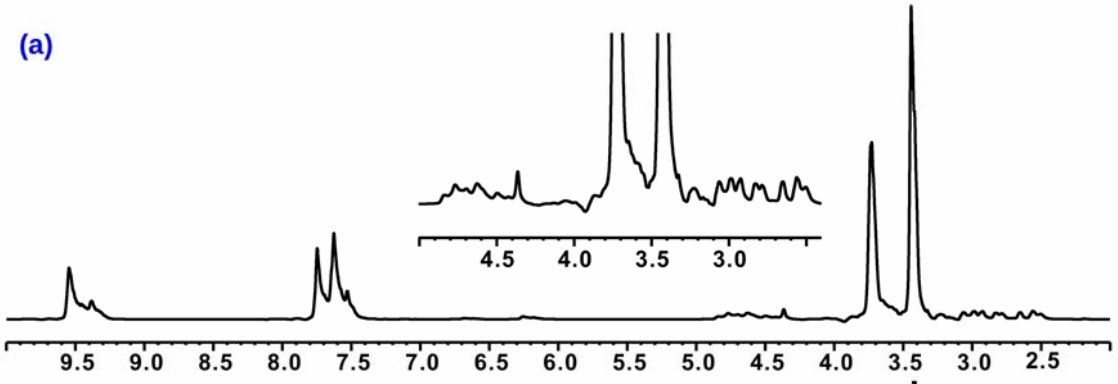
J. Am. Chem. Soc. **2012**, *134*, 6637.

Understanding adaptivity of Pd catalysts



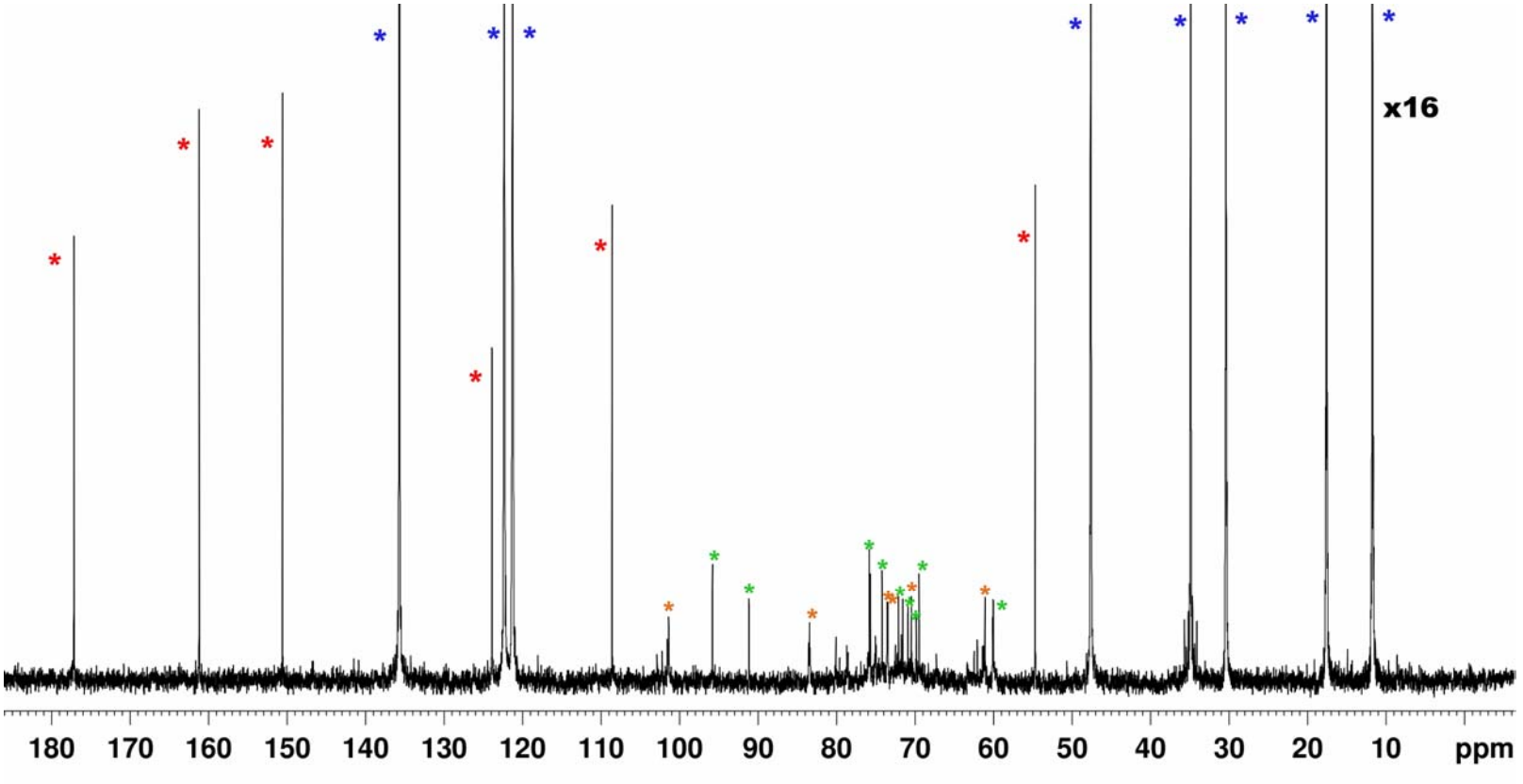
J. Am. Chem. Soc. **2012**, *134*, 6637.

High resolution NMR in ionic liquids



Chem. Sus. Chem., **2012**, 5, 783.

High resolution NMR in ionic liquids



$^{13}C\{^{1}H\}$ NMR spectrum (150 MHz, 80 ^{o}C) of the native IL sample of reaction mixture: [BMIM][Cl] (blue), glucose (green), 5-HMF (red), and borate complex (orange).

Chem. Sus. Chem., **2012**, 5, 783.

Chem. Rev. **2011,** *111,* 418–454

Characterization of Molecular Systems and Monitoring of Chemical Reactions in Ionic Liquids by Nuclear Magnetic Resonance Spectroscopy

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V.P.Ananikov, *Chem. Rev.* **2011**, 111, 418.