research resources center for
Magnetic Resonance
St.Petersburg State University

Saint-Petersburg State University Research Park

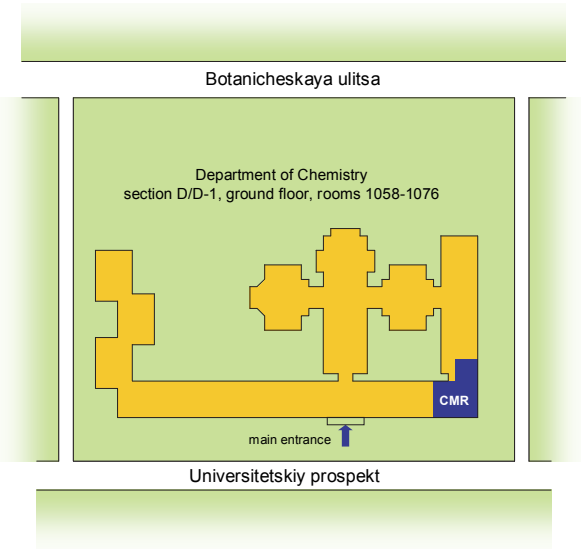
WHAT DO WE DO?

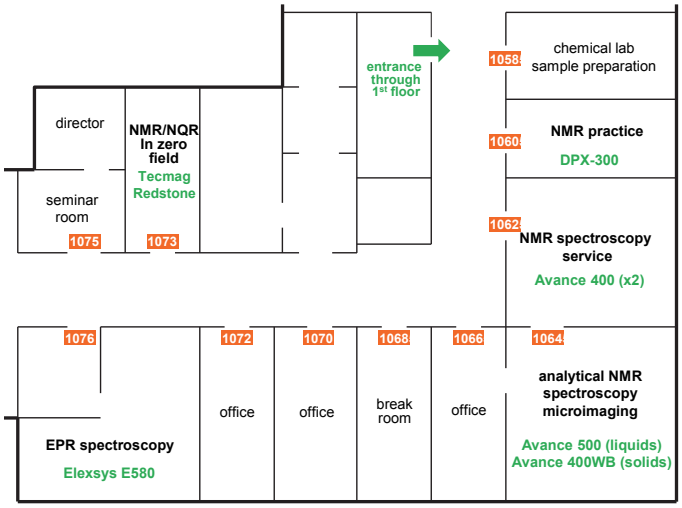
The Center for Magnetic Resonance provides access to the instrumentation, expertise, and infrastructure to carry out and support fundamental, applied and innovative research projects utilizing NMR, EPR and NQR spectroscopy as well as magnetic resonance microimaging. On commercial basis, all services of the Center could be provided to external users, such as medical, pharmacological and forensic organisations, as well as industry. Cooperations aimed for publications in peer-reviewed journals with academic research institutes and universities are possible as pro bono work.

HOW TO FIND US?

Center for Magnetic Resonance
St.Petersburg State University
Universitetskij pr. 26
198504 St.Petersburg, Russia

cmr.spbu.ru

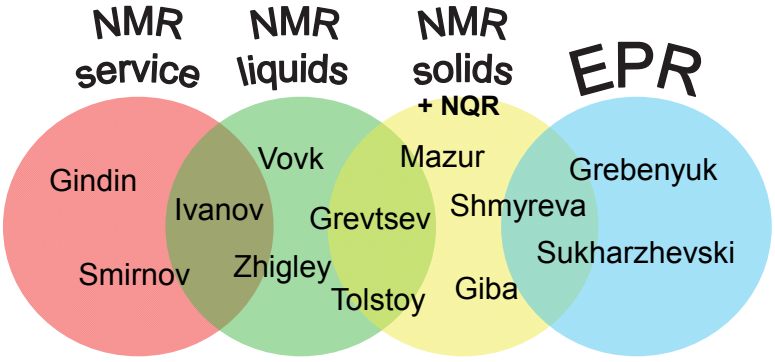




Phone numbers +7 812 ….

Room		Phone nr.	Local phone nr.
1058	Chemical lab		
1060	NMR Service	… 428-43-25	4325
1062	NMR Service	… 428-95-63	9563
1064	Analytical NMR		
1066	Office	… 363-69-25	5915 (IP phones only)
1068	Break room		5662 (IP phones only)
1070	Office		5916 (IP phones only)
1072	Office		5917 (IP phones only)
1073	zero-field NMR + NQR	… 428-95-64	9564
1075	Director	… 363-68-99	5661 (IP phones only)
1076	EPR spectroscopy	… 428-95-65	9565

STAFF



Ass. Prof. Dr. Peter Tolstoy, director
NMR of liquids, solutions and solids

peter.tolstoy@spbu.ru
Phone (cell): +7 (921) 430-81-91
Phone (room 1075): +7 (812) 363-68-99

Research interests: hydrogen bonding, intermolecular interactions, cryospectroscopy, combined NMR/UV

Dr. Vladimir Gindin, deputy director
NMR of liquids and solutions, NMR-service

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Phone (room 1062): +7 (812) 428-95-63
Phone (room 1070): +7 (812) 324-12-70, ext. 5916

Research interests: elucidation of structure and tautomerism of organic compounds and complexes

Sergey Smirnov, lead specialist
Liquid-state NMR spectroscopy, NMR-service

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Phone (room 1062): +7 (812) 428-95-63
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Research interests: high-resolution NMR, study of H-bonds by low-temperature NMR in liquified Freons

Alexander Ivanov, lead specialist
Liquid-state NMR spectroscopy, NMR-service

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Phone (room 1062): +7 (812) 428-95-63
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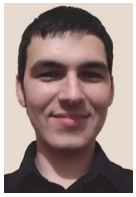
Research interests: structure elucidation of organic compounds using modern NMR spectroscopy techniques

Mikhail Vovk
Liquid-state NMR, relaxation, diffusion measurements

m.vovk@spbu.ru
Phone (room 1066): +7 (812) 363-69-25

Research interests: NMR relaxation, hydration of organic molecules in aqueous solutions



Artem Grevtsev
Liquid-state NMR spectroscopy

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Phone (room 1066): +7 (812) 363-69-25

Research interests: organic-inorganic composites, thin film solar cells, chalcopyrite nanoparticles

Elvira Zhigley
Liquid-state NMR spectroscopy

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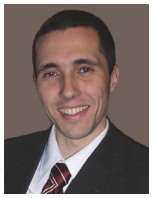
Phone (room 1066): +7 (812) 363-69-25

Research interests: NMR, laser-induced metal deposition, physical-chemical analytical methods



Dr. Anton Mazur
NMR spectroscopy of solids, MR microimaging

a.mazur@spbu.ru
Phone (room 1066): +7 (812) 363-69-25

Research interests: NMR of magnetically ordered systems, nanostructured materials, NMR relaxation in solids

Ivan Giba
NMR spectroscopy of solids

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Research interests: organometallic catalysis in organic molymer chemistry, solid state NMR spectroscopy and relaxation

Dr. Anna Shmyreva
NMR of magnetically ordered systems, NQR spectroscopy

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Phone (room 1073): +7 (812) 428-95-64
Phone (room 1072): +7 (812) 324-12-70, ext. 5917

Research interests: NMR of magnetic ordered systems, nanostructured materials, NQR

Dr. Stanislav Sukharzhevskii
EPR spectroscopy

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Phone (room 1076): +7 (812) 428-95-65

Research interests: EPR, application of MR in natural sciences, spectroscopic methods in geology and ecology

Ekaterina Grebenyuk
EPR spectroscopy

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Phone (room 1076): +7 (812) 428-95-65

Research interests: spectroscopic methods in chemistry, sol-gel process, nanocomposites

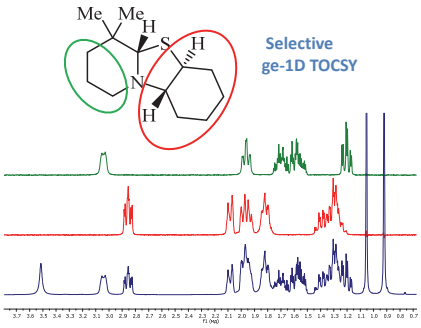
EQUIPMENT

2X BRUKER 400 MHZ AVANCE AND BRUKER 300 MHZ DPX

These instruments are dedicated to service routine measurements of 1D and 2D NMR spectra of liquids and solutions.

- Direct and inverse detection probes.
- Observed nuclei range from ^{1}H to ^{109}Ag.
- Possible $^{1}H\{^{19}F\}$ and $^{19}F\{^{1}H\}$ measurements.
- Possible ^{2}H measurements with ^{19}F lock.

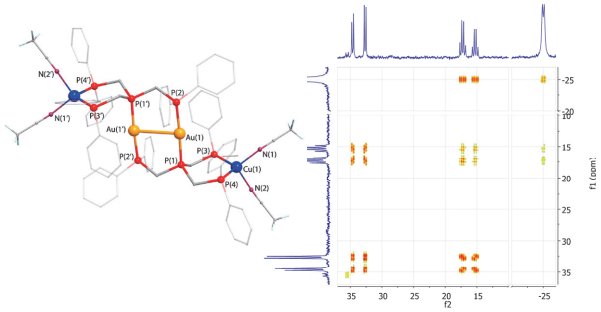
FULL RANGE OF NMR TECHNIQUES



Selective NMR experiments used to obtain sub-spectra of individual spin systems in order to establish the stereo-chemical structure of a complex molecule.

V.V. Sokolov, A.Yu. Ivanov, M.S. Avdontseva, A.A. Zolotarev, Chem. Heterocycl. Compd. 2014, 50, 550-556.

STRUCTURE BY 2D, 3D ETC. NMR



Homonuclear correlations such as ^{31}P-^{31}P COSY and other multi-dimensional techniques allow one to interpret long-range spin-spin couplings and determine structure of organometallic complexes.

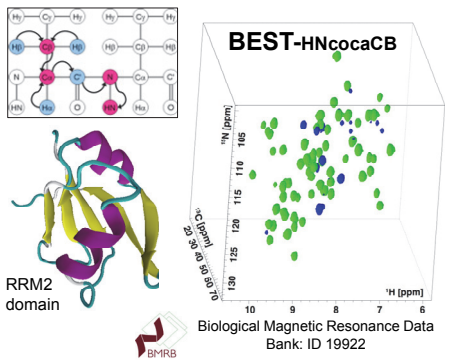
M.T. Dau, J.R. Shakirova, A.J. Karttunen, E.V. Grachova, S.P. Tunik et al., Inorg. Chem., 2014, 53, 4705-4715.

BRUKER 500 MHZ AVANCE

The spectrometer is suited for measurements of 1D, 2D and 3D NMR spectra of liquids and solutions.

- Long measurements at low temperature (down to 110 K).
- Three-channel architecture (from ^{1}H to ^{109}Ag).
- Diffusion measurements at temperatures up to 470 K.
- Combined NMR/UV-vis spectroscopy

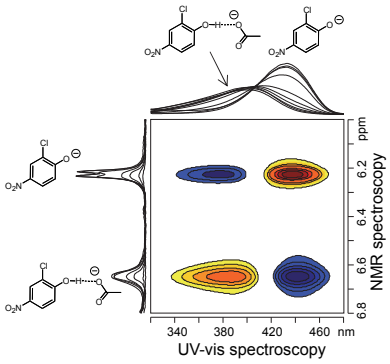
STRUCTURE DETERMINATION OF PROTEINS



Structure of RNA-recognition motif RRM2 in TDP-43 protein has been established using liquid state multi-dimentional NMR. TDP-43 constitutes a major component of the inclusion bodies formed in the brains of the patients with frontotemporal lobar degeneration.

N.R. Skrynnikov, S.O. Rabdano, I.S. Podkorytov et al., 2014, *unpublished*.

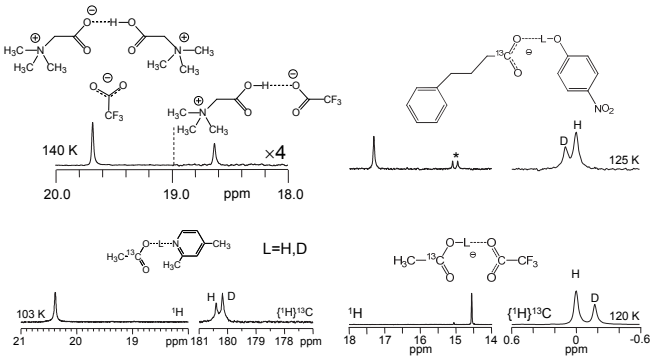
COMBINED NMR/UV-VIS SPECTROSCOPY



UV-Vis spectrometer build within the magnet of an NMR spectrometer allows one to obtain simultaneously NMR and UV-vis spectra and analyze the consistent set of spectroscopic data by 2D cross-correlation methods.

B. Koeppe, P.M. Tolstoy, E.T. J. Nibbering, T. Elsaesser, J. Phys. Chem. Lett., 2011, 2, 1106-1110.

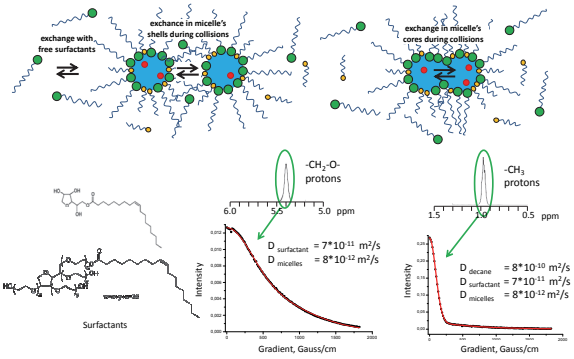
Low temperature NMR



Extra-low temperature NMR in liquefied gases as solvents (CDF_3/CDF_2Cl) allows to reach slow molecular and proton exchange regimes and resolve signals of H-bonded complexes of various stoichiometry & isotope composition.

B. Koeppe, J. Guo, P.M. Tolstoy, G.S. Denisov, H.-H. Limbach,
J. Am. Chem. Soc. 2013, 135, 7553-7566.

Diffusion-ordered NMR spectroscopy



Diffusion ordered NMR spectroscopy (DOSY) can be used to study sizes of micelles, their mobility and chemical exchange processes in complex microemulsions.

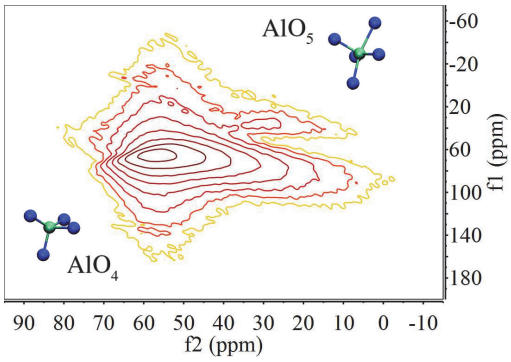
N.A. Smirnova, E.A. Safonova, A.S. Koneva, M. Vovk et al.,
2015, *unpublished*.

Bruker 400 MHz WB Avance

This NMR spectrometer is dedicated to the studies of samples in solid state: crystallines, powders, weakly ordered materials, gels, liquid crystals, amorphous compounds, nanostructures (zeolites, silicates) etc. The spectrometer allows one to study diffusion processes and obtain micro-tomographic image

- Long measurements at temperatures from 130 K to 870 K.
- Microtomography of objects up to 30 mm in linear size.
- Magic angle spinning up to 30 kHz.
- Diffusion measurements (gradients up to 3000 G/cm).

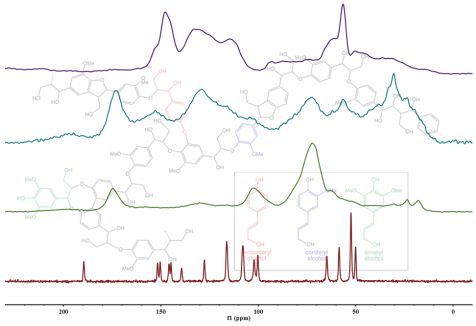
Structural studies of disordered materials



The study of the structural composition of the multicomponent glasses by MQ MAS and other methods allows one to create materials with predetermined physical and chemical properties

A.A. Osipov, V.E. Eremyashev, A.S. Mazur, P.M. Tolstoy, L.M. Osipova,
Phys. Chem. Glasses 2015, *accepted*.

Studies of complex mixtures of natural compounds



^{13}C NMR spectral analysis of complex mixtures of macromolecules of natural origin allows one to track the processes of decomposition (for example of lignins and its derivates) and soil formation.

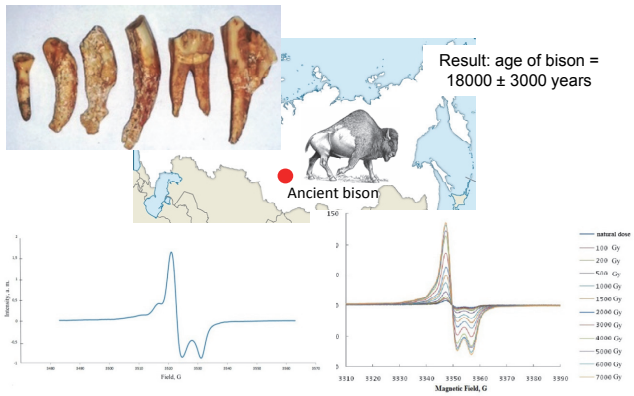
A.V. Vasiliev et al., A.I. Abakumov et al.,
2015, *unpublished*.

EPR spectrometer Bruker Elexsys E580

EPR spectrometer (λ = 3.2 cm, X-band) for studies of paramagnetic centers in solids, liquids, solutions, including aqueous solutions, and gases. Spectrometer is capable of measurements in CW-mode as well as in FT-mode.

- UV-irradiation of the samples (100 W, 200-2000 nm).
- Temperature range from 3.7 K to 500 K.
- ENDOR/TRIPLE measurements.

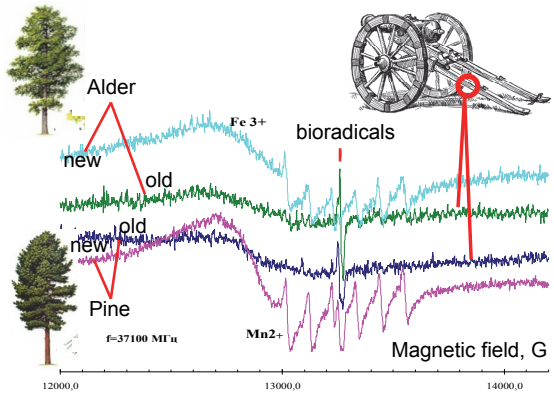
EPR-dating of tooth enamel



EPR spectra of radiation defects in tooth enamel of ancient bison. The nature of the paramagnetic centers and their development with time allows one to date such paleontological samples.

A.I. Antipov, A.Yu. Kultaeva, S.M. Sukharzhevski et al., 2014, *unpublished*

EPR-study of wood degradation



EPR spectra of old and new wood samples from the digs at Borodino battlefield. Signals of bioradicals and of Mn^{2+} and Fe^{3+} ions can be identified. These data can be used in historical reconstruction of the events in the year 1812.

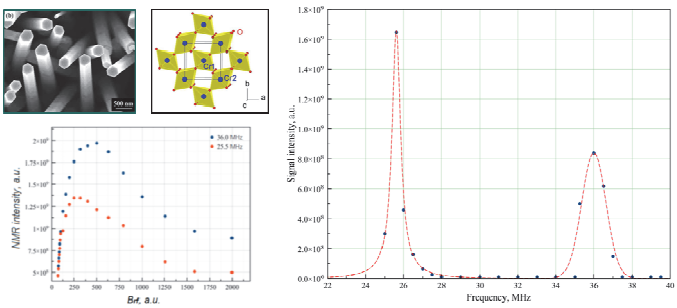
S.M. Sukharzhevski et al., *unpublished*

Tecmag Redstone NMR/NQR 1-500 MHz

This spectrometer allows one to obtain spectral and relaxation NMR and NQR parameters for solids, polymers, metals, glass and magnetically ordered systems.

- Observed frequencies in a zero external magn. field 20-120 MHz.
- Temperature range from 4 K to 500 K.
- Acquisition of weak NQR signals by multiple scans
- NQR spectra of ^{7}Li, ^{27}Al, ^{35}Cl, ^{63,65}Cu, ^{75}As, ^{93}Nb, ^{183}Ta etc nuclei.
- NMR of ^{11}B, ^{57}Fe,^{59}Co, ^{61}Ni etc. nuclei.
- Investigation of orientation dependence for monocrystals.

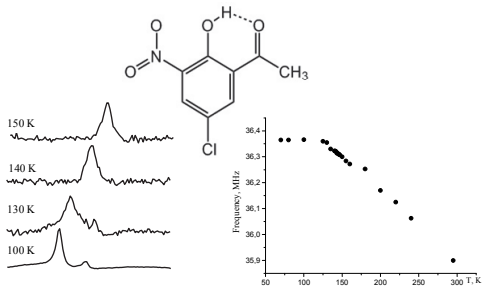
NMR of magnetically ordered nanoparticles



Two lines in the NMR spectrum of CrO_2 nano-rods with the structure of rutile show that there are two non-equivalent types of Cr ions instead of monovalent Cr^{4+}.

A.A. Shmyreva, O.M. Osmolowskaya et. al., 2015, *unpublished*.

35Cl NQR spectra of organic compounds

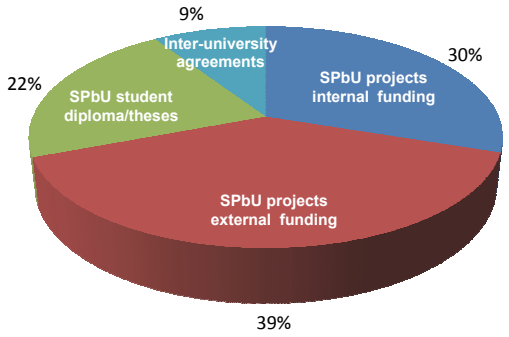


Molecule of 2-hydroxy-3-nitro-5-chloroacetophenone undergoes a phase transition at ca. 120 K, which can be detected by the 35Cl NQR spectroscopy.

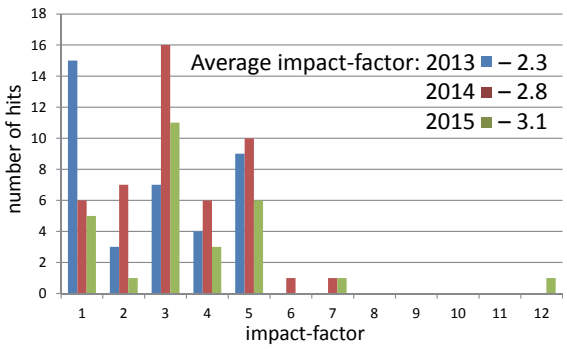
E.V. Kukushkina, A.I. Filarowski, P.M. Tolstoy et al., 2015, *unpublished*.

WORKLOAD AND PUBLICATION STATISTICS 2013-2015

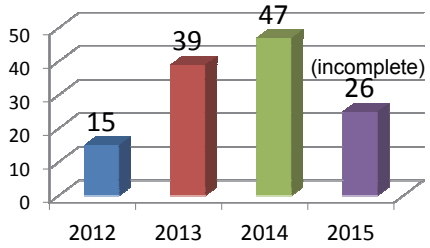
Measurement time distribution



Papers with acknowledgement to CMR



Papers published per year



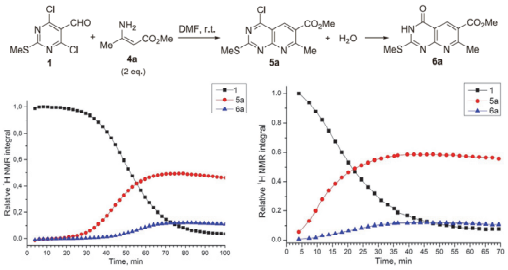
Users and projects:

~200 primary users: 75% chemists,
15% physicists,
10% other (biologists and geologists)

~200 projects: ca. 2 projects per week, av. duration 1.5 yrs

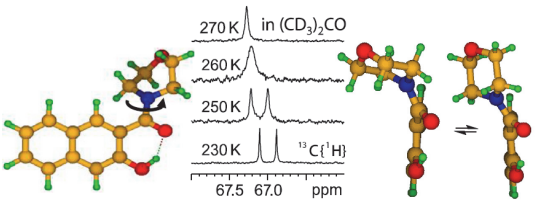
~50-100 requests per day: 90% routine,
10% other

SELECTED PUBLICATIONS 2015



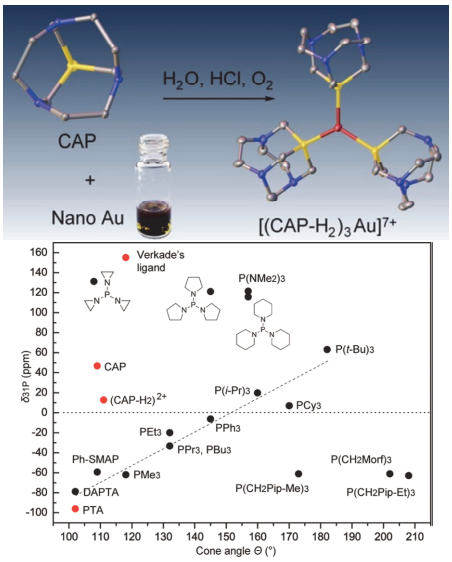
A new facile synthesis of pyrido[2,3-d]pyrimidin-4-ones via cyclocondensation of 4,6-dichloro-2-methylsulfanylpyrimidine-5-carbaldehyde with b-alkyl and b-aryl-b-aminoacrylic esters followed by hydrolysis of chlorine atom at position 4 of pyridopyrimidine ring has been developed. The reactions were monitored by ^{1}H NMR and the structure of the product was confirmed by 1H, 13C NMR, as well as HSQC and HMBC methods.

M.E. Chizhova, O.Yu. Bakulina, A.Yu. Ivanov, P.S. Lobanov, D.V. Dar'in, "Facile synthesis of pyrido[2,3-d]pyrimidines via cyclocondensation of 4,6-dichloro-2-methylsulfanylpyrimidine-5-carbaldehyde with β-substituted β-aminoacrylic esters", Tetrahedron 2015, 71, 6196-6203. DOI:10.1016/j.tet.2015.06.085.



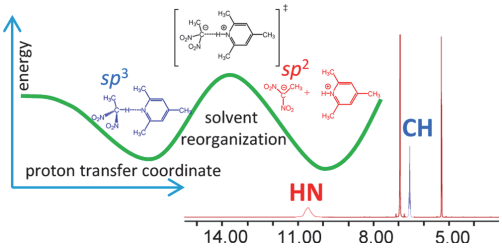
The conformational states of three b-hydroxynaphthylamides (morpholine, pyrrolidine and dimethylamine derivatives) have been analyzed by NMR, X-ray and DFT calculations. In aprotic solution the molecules contain intramolecular OHO hydrogen bonds, which change into intermolecular ones in solid state. The energy barriers for the amide group rotation around the CN bond were estimated from the line shape analysis of ^{1}H and ^{13}C NMR signals.

T. Kozlecki, P.M. Tolstoy, A. Kwocz, M.A. Vovk, A. Kochel, I. Polowczyka, P.Yu. Tretyakov, A. Filarowski, "Conformational state of β-hydroxy-naphthylamides and barriers for the rotation of the amide group", Spectrochim. Acta A, 2015, 149, 254–262. DOI:10.1016/j.saa.2015.04.052.



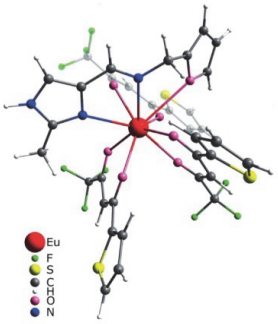
A tricyclic phosphine with previously unreported tris(homoadamantane) cage architecture is described. That water-soluble, air- and thermally stable ligand, 1,4,7-triaza-9-phosphatricyclo[5.3.2.1]tridecane (CAP) exhibits unusual chemical behavior toward gold and gold compounds: it readily reduces Au(III) to Au(0), promotes oxidative dissolution of nanocrystalline gold(0) with the formation of water-soluble trigonal CAP−Au(I) complexes, and displaces cyanide from $[Au(CN)_2]^-$ affording triangular $[Au(CAP)_3]^+$ cation.
Unusual chemical properties of various aminophosphines such as Verkade's ligand and tris(aziridinyl)phosphine has been linked to their low steric demand (measured as cone angle), which leads to significant downfield shift of ^{31}P NMR signals.

S.N. Britvin, A. Lotnyk, "Water-Soluble Phosphine Capable of Dissolving Elemental Gold: The Missing Link between 1,3,5-Triaza-7-phosphaadamantane (PTA) and Verkade's Ephemeral Ligand", J. Am. Chem. Soc., 2015, 137 (16), pp 5526–5535. DOI:10.1021/jacs.5b01851.



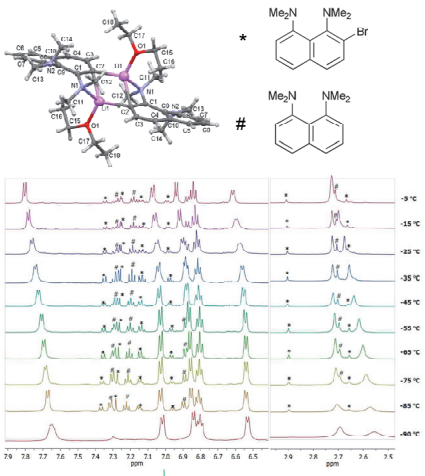
The intermolecular complex with a CHN hydrogen bond formed by 1,1-dinitroethane (DNE) and 2,4,6-trimethylpyridine (collidine) dissolved in CD_2Cl_2 was studied experimentally by ^{1}H NMR spectroscopy at 180−300 K. Equilibrium between the molecular CH···N form and the zwitterionic C−/HN+ form was detected in the slow exchange regime in the NMR time scale. QC calculations of the proton transfer pathway have revealed that the most indicative NMR parameter reflecting hybridization of a carbon atom is the spin-spin coupling constant ^{1}JCC, which starts to change rapidly as soon as a structure with a quasi-symmetric C··H··N bond is reached.

E. Yu. Tupikina, G.S. Denisov, P.M. Tolstoy, «NMR Study of CHN Hydrogen Bond and Proton Transfer in 1,1-dinitroethane Complex with 2,4,6- trimethylpyridine", J. Phys. Chem. A, 2015, 119, 659-668. DOI: 10.1021/jp511493m.



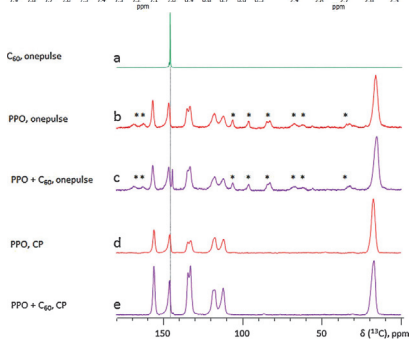
The Schiff base NNO ligand 1-(furan-2-yl)-*N*-[(2-methyl-1*H*imidazol-4-yl)methylene]methanamine was synthesized and structurally characterized by XRD crystallography, mass spectrometry, and NMR spectroscopy. Two lanthanide complexes [Ln(tta)$_3$(NNO)] (Ln = Eu^{III} and Lu^{III}; tta = thenoyltrifluoroacetone) were prepared and studied spectroscopically. The Eu complex shows bright red metal-centered phosphorescence under excitation into the ligand (π–π*) absorption bands with a quantum yield of ca. 80% and a lifetime of 580 µs. An organic light-emitting diode (OLED) device based on the Eu complex incorporated into a poly(9-vinylcarbazole) (PVK) matrix was prepared.

K.S. Kisel, G. Linti, G.L. Starova, V.V. Sizov, A.S. Melnikov, A.P. Pushkarev, M.N. Bochkarev, E.V. Grachova, S.P. Tunik, "Syntheses, Structures, and Photophysical Properties of Eu and Lu Diketonates with a Neutral Polydentate Imidazolylmethanamine Ligand", Eur. J. Inorg. Chem., 2015, Issue 10, 1734–1743. DOI: 10.1002/ejic.201403186.



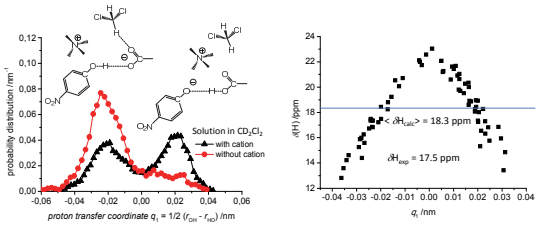
It has been found that 1,8-bis(dimethylamino)naphthalene (DMAN), unlike N,N-dimethylaniline, undergoes ring metallation in n-BuLi–TMEDA–Et$_2$O system with low selectivity and in poor total yield. The situation is significantly improved in t-BuLi–TMEDA–n-hexane system when 3- and 4-lithium derivatives become the only reaction products in good yield. The formation of 3-Li-DMAN is especially fortunate since no method of direct meta-functionalization of DMAN has been known to date. The relative stability and structure of DMAN lithium derivatives have been examined with the help of X-ray and multinuclear NMR measurements as well as DFT calculations.

A. S. Antonov, A. F. Pozharskii, V. A. Ozeryanskii, A. Filarowski, K. Yu. Suponitsky, P. M. Tolstoy, M. A. Vovk "Ring Lithiation of 1,8-Bis(dimethylamino)naphthalene: Another Side of the 'Proton Sponge Coin'", Dalton Trans. 2015, Advanced Article. DOI:10.1039/C5DT02482J.



The physicochemical features of nanocomposites of poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) and fullerene C_{60} were studied with the purpose of using these compounds as a selective layer of composite membranes in pervaporation coupling esterification of acetic acid with ethanol to produce ethyl acetate. The nature of the interaction between PPO and C^{60} molecules in composite was studied by NMR.

A. Penkova, G. Polotskaya, A. Toikka, "Pervaporation composite membranes for ethyl acetate production", Chem. Eng. Process., 2015, 87, 81-87, DOI: 10.1016/j.cep.2014.11.015.



A joint experimental and QC study on the influence of solvent dynamics on the protonation equilibrium in a strongly hydrogen bonded phenol-acetate complex in CD_2Cl_2 is presented. Experimentally, combined low-temperature ^{1}H and ^{13}C NMR and UV-vis spectroscopy was used, showing proton tautomerism between PhOH···$^-$OAc and PhO$^-$···HOAc forms. Computationally, *ab initio* molecular dynamics was used. The relative motion of counter-cation and "free" carbonyl group of the acid plays the major role in the OHO bond geometry and causes proton "jumps", *i.e.* interconversion of tautomers. Weak H-bonds between CH groups of the solvent and oxygen atom of carbonyl stabilize PhOH···$^-$OAc type of structures. Breaking of CH···O bonds shifts the equilibrium towards PhO$^-$···HOAc form.

S. Pylaeva, C. Allolio, B. Koeppe, G.S. Denisov, H.-H. Limbach, D. Sebastiani, P.M. Tolstoy, "Proton transfer in hydrogen bonded complex caused by solvation shells fluctuations: ab initio MD study of anionic phenolate-carboxylic acid and neutral pyridine-carboxylic acid systems", PCCP 2015, 17, 4634-4644. DOI: 10.1039/c4cp04727c.

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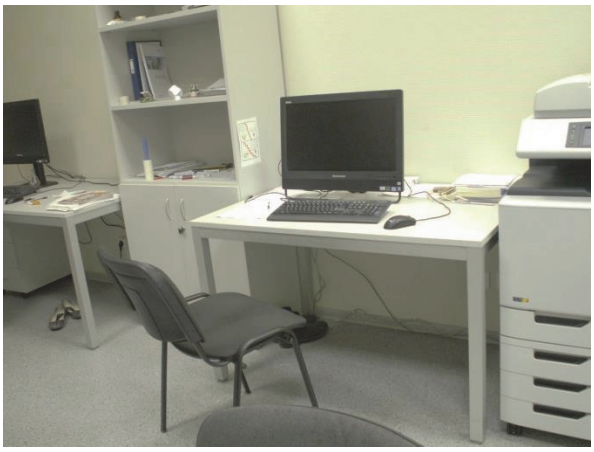
Personal desk space for visitors



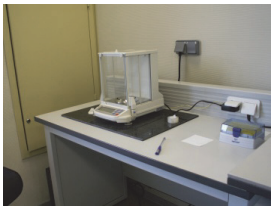
Break room

Common space

Chemical lab for sample preparation

Hoods

Rotary evaporator

High vacuum line

Scales