

Laboratory of physical-chemical analysis methods

LABORATORY OF CATALYTIC RESEARCH

TOMSK STATE UNIVERSITY



aboratory of Catalytic Research (LCR) is a multidisciplinary R&D center dealing with a wide range of fundamental and applied research in the field of chemistry and catalysis, surface chemistry, organic synthesis, biochemistry, polymer chemistry, physical-chemical analysis, and materials sciences.

The laboratory has been tracing its history since 1965, and was founded on the basis of department of physical chemistry as an applied research laboratory.

Our team consists of



including

13 PhDs 9 Doctors



The laboratory is tooled up with a complex of modern research and analytical equipment.

60 developments of LCR

have been implemented in chemical industry of the USSR and the Russian Federation

Over the last 5 years we achieved:

- * More than 30 Russian patents and 15 know-hows
- * More than 150 publications in Russian and international journals
- * More than 35 projects (federal purposeful programs, grants of the RFBR, sponsored research, etc.)
- * 800M rubles raised for R&D
- * More than 20 winners of the program "Participant of the youth science and innovation competition"

The following departments are a part of joint scientific team:

- Department of catalysis
- Department of organic synthesis
- Laboratory of physical-chemical analysis methods
- Department of sorption studies

- Department of polymers and monomers
- Department of materials science
- Laboratory of translational cell and molecular biomedicine
- Collective Use Center for Sorption and Catalytic Studies

ORGANIC SYNTHESIS

Key areas:

* Development of methods to synthesize valuable glyoxal-based organic compounds for various branches of industry (petrochemical, pharmaceutical, defense industries, etc.)

* Studies of peculiarities of organic reactions

* Development of basis to design low-tonnage chemical production facilities

The developed technologies to synthesize chemical products:

The implemented products



Crystalline glyoxal (TS 2417-07508902-213-2013. manufacturer: JSC "FRPC "Altai")



Glvcoluril (TS 2478-001-80061471-2011 with chng. 1, manufacturer: LLC "Glyoxal-T")



Glyoxal-sodium bisulfite adduct (TS 2438-012-67017122-2014. manufacturer: LLC "Novochem")



Sodium glycolate (TS 2432-011-67017122-2014. manufacturer: LLC "Novochem")



2-Methylimidazole (TS 2478-001-61226910-2013)



Imidazole



5-nitroimidazole



Dimetridazole



Metronidazole



Tetramethylolglycoluril

Equipment for synthesis

- * Büchi Glass Uster reactors. 10 and 15 l
- * A set of chemical reactors, 100 ml 1 l
- * Spray drier Büchi B-290
- * Freeze drier Alpha 1-2 LDZ Plus



coluril

Tetraacetylglyacid



Tetrachloroglycoluril



Tetrabromoglycoluril





HO

acid

5



Quinoxaline



4,5-Dihydroxyimidazolidine-2-tion

- * Rotary evaporator Heidolph Laborota 4003 Control
- * Rotary evaporator Büchi R-II
- * Rotary evaporator Büchi Rotavapor R-220
- * Rotary evaporator Heidolph





Sodium glyoxylate



Allantoin

CATALYSIS AND SORPTION STUDIES

Key areas:

- * Synthesis of metal and oxide catalysts
- * Study of catalytic activity and optimization of catalyst performance conditions
- * Kinetic and mechanistic studies of various catalytic reactions
- * Development of pilot catalytic processes,
- * Catalytic process performance under conditions close to industrial ones

- * Studies of catalytic processes under elevated pressures
- * Studies of solid materials using non-isothermal kinetics approaches (temperature-programmed desorption, temperature-programmed oxidation, temperature-programmed reduction, temperature-programmed reaction)
- * Studies of textural properties (specific surface area, porosity) of catalysts and sorbents



Key topics:

* Oxidative coupling and cross-coupling of methane

* Selective oxidation of diols into valuable organic compounds

* Development of catalysts for photocatalytic purification of water solutions

* Development of catalytic processes for bioethanol processing

- * Total oxidation of hydrocarbons
- * Catalytic synthesis of dimethyl ester
- * Olefin production from raw hydrocarbons

* Development of catalysts for lowtemperature oxidation of carbon monoxide

* Catalytic liquid-phase hydrogenation of monosaccharides

* Catalytic dehydrogenation of C4-C5 alkanes

The implemented products:

– During 1965-1980 new catalysts for methanol oxidation had been developed and implemented at 5 plants of USSR (Kiviõli, Severodonetsk, Tomsk, etc.)

- During 2005-2009 a PAG-2A catalyst had been developed (TS 2174-003-02069318-2009, manufacturer: LLC "Glyoxal-T") The catalyst is used to manufacture dialdehydes via partial oxidation of diols (ethylene glycol, propylene glycol, etc.) to glyoxal and methylglyoxal, respectively. The catalyst was implemented at "Novochem" LLC (Tomsk)

Developments

- Total oxidation catalyst OU-G (joint development with LLC "Glyoxal-T")

The catalyst is used for total oxidation of harmful, toxic and volatile components of off-gases of chemical, petrochemical, paint-and-lacquer and other branches of industry.

– Catalyst for low-temperature CO oxidation

The catalyst is used to purify air from carbon monoxide. It effectively works at room temperature and does not contain gold and platinum group metals. – Catalyst for acetaldehyde manufacturing via ethanol dehydrogenation

The catalyst is used to process ethanol or bioethanol into acetaldehyde. The produced acetaldehyde possesses high purity, and hydrogen is formed as a useful by-product.



Equipment

- Lab-scale catalytic unit to study the processes at elevated pressures (up to 80 atm)
- Lab-scale flow-type high-pressure catalytic unit to study metathesis reactions (up to 55 atm)
- Modular pilot catalytic unit to study gas-phase oxidative processes
- Catalytic unit to study heterogeneous photocatalytic reactions
- Flow catalytic unit to study liquidphase processes
- Continuous and pulse reactor to carry out liquid-phase catalytic processes
- Catalytic unit to study kinetics of heterogeneous catalytic reactions
- A line of gas chromatographs
 "Chromatec Crystal 5000.1" and
 "Crystal 5000.2"

- A line of technological equipment to prepare pilot batches of catalysts
- Grain strength measurer IPG-1M
- Analyzer of chemical and physical sorption AutoChem 2950 HP combined with quadruple mass spectrometer UGA-300

– Chemisorption analyzer ChemiSorb 2750 combined with mass spectrometer QMS-300

 Analyzer of specific surface and porosity Tristar 3020

 Analyzer of specific surface and porosity Tristar 3Flex

– Synchronous thermal analysis device Netzsch STA 449 F1 combined with mass spectrometer QMS 403 CF Aeolos



PHYSICAL-CHEMICAL ANALYSIS

Key areas:

- * Development of analysis methods and analytical support of research carried out by LCR of TSU
- * Fundamental and applied research in the field of clinical metabolomics, "omix" studies, analytical support of advanced medical research
- $\boldsymbol{\star}$ Fundamental and applied research in the field of analytical
 - A wide range of analytical research and analysis using up-to-date physical-chemical methods (GC, GC/MS, HPLC, HPLC/MS/MS, MP-AES, HCNS(O)-analysis, TLC, Karl Fisher titration, etc.).

chemistry of organic compounds

- * Analytical support of preclinical and clinical studies of drugs in accordance with principles of GLP and GCP
- * Analytical support of low-tonnage production of chemical products on the basis of TSU-affiliated enterprises



have been developed and implemented at the laboratory

GAS CHROMATOGRAPHY

- * Compositional analysis of gases
- * Determination of concentration of organic compounds in water solutions
- * Compositional analysis of oil and oil products

18.5 J0m x 0 53mm 10 x 50,m 260E470P S/N 12711A1

* Imitation distillation of oil

LIQUID CHROMATOGRAPHY (HPLC)

- * Determination of content of organic compounds in water solutions
- * Analysis of heterocyclic compounds
- * Determination of molecular-mass distribution of various polymers using gel permeation chromatography
- * Determination of content of water- and fat-soluble vitamins



MASS-SPECTROMETRIC METHODS (HPLC/MS/MS, GC/MS)

* Qualitative and quantitative analysis of samples using gas and liquid chromato-mass-spectrometry

* Analysis of biological samples (determination of active compounds, metabolites, pharmacokinetic studies)

* Analysis for trace amounts of compounds



ELEMENTAL ANALYSIS

- * Determination of metal content in various objects using MP-AES



OTHER METHODS



- * Determination of water mass fraction using coulometric and volumetric titration on Karl Fischer
- * Refractometry
- * Spectroscopy in UV and visible regions
- * Moisture analysis of solids using gravimetrical method
- * Instrumental thin-layer chromatography analysis combined with MS detection
- * Determination of melting and boiling points
- * Potentiometric titration
- * pH measurements, ionometry





Equipment

A line of gas chromatographs
 "Chromatec Crystal 5000.1" and
 "Crystal 5000.2"

– HPLC Agilent 1200 (Agilent technologies, USA)

– HPLC Agilent 1260 (Agilent technologies, USA)

– Chromato-mass-spectrometer Finnigan Trace DSQ (Thermo Electron Chomatography and Mass Spectrometry Division, USA)

– Liquid chromatograph UltiMate 3000 (Dionex, USA) with triple quadruple mass-spectrometer API 2000 (AB Sciex, USA)

– Autotitrator 870 Titrino plus (Metrohm AG, Switzerland)

– Autotitrator Metrohm 899 Coulometer (Metrohm AG, Switzerland)

– Weighing drymeter MX-50 (A&D Company Ltd., Japan) – Microwave plasma atomic emission spectrometer Agilent 4100 (Agilent technologies, USA)

– Elemental analyzer EuroEA 3000 (EuroVector, Italy)

– Autotitrator DL15 (Mettler Toledo, USA)

– Melting point analyzer M-560 (Buchi, Switzerland)

– A set for high performance thin-layer chromatography Camag (Camag, Switzerland)

– Refractometer NAR-2T (ATAGO, Japan)

– Automatic polarimeter AP-300 (ATAGO, Japan)

– Spectrophotometer UV-1800 (Shimadzu, Japan)

MORE INFORMATION ON OUR ANALYTICAL CAPABILITIES CAN BE FOUND ON www.lpcma.tsu.ru

RESEARCH IN THE FIELD OF POLYMER CHEMISTRY

Key topics:

- * Production of glycolide and lactide monomers as well as biodegradable polymers and compolymers on the basis thereof
- * Synthesis and studies of properties of composites based on synthetic hydroxyapatite and oligomers of glycolic and lactic acids
- * Ion-plasma modification of polymer surfaces

Equipment

Ion-plasma complex for surface processing by ion and electron beams MEVVA V.RU

RESEARCH IN THE FIELD OF BIOMEDICINE

Key topics:

- * Development of innovative implantation materials based on biocompatible polymers with anti-inflammatory properties
- * Development of a system of tumor immune status marker to determine stability to cancer chemotherapy
- * Development of innovative system to determine places of undetected chronical inflammation leading to cardiovascular diseases
- * Synthesis and investigation of ligandmixed complexes of d-metals having antimicrobial activity

Equipment

Confocal microscope ZEISS LSM 780 NLO

RESEARCH IN THE FIELD OF MATERIALS SCIENCES

Key topics:

* Production and investigation of ultra- and nanodispersed powders to produce composite materials

* Development of modified compositions based on ultradispersed powders of refractory metals for out-of-furnace processing of castings made of black and non-ferrous foundry

* Development of technologies to form new high-impact, heat-proof and corrosion-resistant

metallic materials

* Fundamental and applied research in the field of structure-phase states and mechanical properties of metallic materials

* Physical basis of new technological processes of surface and bulk strengthening of materials

* Mechanistic studies of structural and phase transitions in metals, ceramic materials and dielectrics under radiation impact

Equipment

– Equipment for metallic sample preparation

– Equipment to study structure and phase states of materials (optical microscopy, transmission microscopy, XRD analysis)

IMPLEMENTATION OF DEVELOPMENTS AND INDUSTRIAL RELATIONS



A number of projects have been carried out by the LCR in cooperation with its industrial partners:

- technology to produce glyoxal via vapor-phase oxidation of ethylene glycol was developed in 2008-2009 (LLC "Novochem", Tomsk)
- technology to synthesize crystalline glyoxal was developed in 2010-2012 (FRPC "Altai", Byisk)
- technology to produce 2-methylimidazole was developed in 2011-2013 (LLC "Aldo-Pharm", Tomsk)

- technology to produce modified urea-formaldehyde resins and low-toxic wooden boards on the basis thereof (emission class E0.5) has been carrying out together with LLC "Tomlesdrev"

n 2014, Engineering chemical and technological center (ECTC) was founded by TSU together with SibSMU and industrial partners.

To implement the technologies

6 companies producing low-tonnage chemistry

have been established, including LLC "Novochem", CJSC "Aldomed", LLC "Glyoxal-T", LLC "Compachem", LLC "Glitergo". The companies comprise the "Novochem" group of companies.



The following services are rendered by the ECTC:



Development of technologies to produce low-tonnage chemical products at pilot level (including new catalytic technologies)



Construction and operation of pilot units



Production of pilot batches of products

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Drafting a basis to design industrial productions

Products implemented into industry

- * Glyoxal synthesis catalyst "PAG-2A", TS 2174-003-02069318-2009
- * Glyoxal, 40% water solution, TS 2633-003-67017122-2011
- * Crystalline glyoxal, TS 2417-07508902-213-201314.
- * Glycoluril, TS 2478-001-80061471-2011 with chng. 1
- * Disinfectant "Dinovis", TS 9392-002-30407785-2012

- * Disinfectant "Aldosteril", TS 9392-003-30407785-2013
- * Modifiers for black (MC) and non-ferrous (MC-M) foundry, TS 1760-001-64101572-2011
- * Technical sodium glycolate, TS 2432-011-67017122-2014
- * Glyoxal bisulfite adduct, TS 2438-012-67017122-2014



PARTNERS AND CUSTOMERS

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Partners

Industrial enterprises:

- JSC "FRPC "Altai"
- JC "SCTB "Katalizator"
- CJSC "Promkataliz"
- LLC "Tomlesdrev"

Russian universities and research institutes

- Boreskov Institute of catalysis SB RAS
- Research institute of pharmacology and regenerative medicine after E.D.
 Goldberg
- Tomsk oncology research institute
- Cardiology research institute (Tomsk)
- Novosibirsk institute of organic chemistry after N.N. Vorozhtsov SB RAS
- Prokhorov General physics institute
- Kurdyumov Institute for metal physics
- National University of Science and Technology "MISIS"
- Siberian State Medical University

- Tomsk Polytechnic University
- Tomsk agricultural institute
- Zelinskiy Institute of Organic Chemistry RAS
- Northern (Arctic) Federal University
- Tomsk state university of architecture and building

Foreign universities and research institutes

- Leiden University Medical Center (Leiden, Netherlands)
- Institut de recherches sur la catalyse et l'environnement de Lyon (Lyon, France)
- Åbo Akademi University (Turku/⊠bo, Finland)
- Universita' di Firenze Sesto Fiorentino (Fi) (Italy)
- Fraunhofer-Institut für Chemische Technologie ICT (Pfinztal, Germany)
- Changzhou University (Changzhou, China)

Customers

- Ministry for Science and education of the Russian Federation
- CJSC "RPC "Micran"
- FSBO "Forensic expert institution of federal fire-fighting service "Testing fire-fighting laboratory" for Tomsk region"
- LLC "Sintegal"
- JC "SCTB "Katalizator"
- LLC "Novochem"
- CJSC "Aldomed"



EDUCATIONAL ACTIVITY

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erm and graduate papers are prepared by undergraduate and master students of chemical department of TSU annually. In 2014-2015 27 students and 15 master students are involved.

In 2014-2015 21 post-graduate students and one post-doc have been carrying out research under supervision of LCR staff.

LCR staff delivers lectures to students of chemical department, e.g. "Physical chemistry", "Scientific foundations of catalyst preparation", "Physical and chemical fundamentals of material surfaces", "Physical chemistry of surfaces of nanocomposite systems", "Resource-saving technologies", "Colloidal chemistry", "Adsorption and catalytic studies of solids", "Acid-base catalysis", etc.

Refresher courses, individual internships, seminars for university staff and specialists of industrial companies are carried out on the basis of the LCR. Duly issued identity document is given to specialists passed their final exams.

Topics for refresher courses:

- * Determination of specific surface area and porous structure of solids using low-temperature gas adsorption (72 h)
- * Study of functional properties and reactivity of surfaces of solids using non-isothermal kinetics (72 h)
- * Fundamentals of heterogeneous catalysis: synthesis, physicalchemical properties, application of catalysts (72 h)
- * Analysis of gaseous and liquid mixtures using gas and liquid chromatography (72 h)

MORE INFORMATION ON EDUCATIONAL PROGRAMS CAN BE FOUND ON www.lcr.tsu.ru



from Russian and CIS countries take our programs annually



CONFERENCES



The LCR organizes conferences:

- * International scientific school-conference for young-scientists "Catalysis: from science to industry", http://catconf.tsu.ru
- * International conference of students and young scientists "Prospects of fundamental sciences development", http://science-persp.tpu.ru
- * All-Russian competition of student presentations "Functional materials: development, investigation, application"



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CONTACTS WELCOME TO COLLABORATION WITH LCRI



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