



Member of ORLEN Unipetrol Group



# RESEAERCH & DEVELOPMEN





# **ABOUT** POLYMER INSTITUTE BRNO

ORLEN Unipetrol RPA s.r.o. - POLYMER INSTITUTE BRNO, o.z. (PIB) is a polymer service provider and technology partner with more than 60 years of tradition in Research & Development.

### **HISTORY**

POLYMER INSTITUTE BRNO, formerly known as the Research Institute of Macromolecular Chemistry (RIMC), has long been the base of applied research in the field of polymers. The institute has been shaped by more than 60 years of evolution. For many years, the RIMC was a state-sponsored organisation, and its interests were very broad, although always directed at areas more industrial than academic. However, the institute has a long history of close cooperation with a number of universities and academic institutions at home and abroad. With a long tradition in Ziealer-Natta catalyst research and polymer stabilisation, the institute's position became very clear, particularly after building the ORLEN Unipetrol Litvínov polyolefin production plants in the early seventies. Since then, the institute's activities have mainly focused on polyolefin catalyst research, development of new resin grades, post-reactor modification of polymers (reactive compounding, polymer alloys, polymer reinforcement, blends, etc.), new formulations of additives (stabilisers, colour concentrates, antistatics, etc.) and troubleshooting.

In 1994, the POLYMER INSTITUTE BRNO became a limited liability company with the status of an independent contract research organisation in which German company Consil VBmbH and Czech petrochemical company Chemopetrol, a.s. owned a capital share. In 2004, Unipetrol, a.s. acquired the entire share from both owners, and the institute thus became a daughter company of the biggest petrochemical complex in the country. After the privatization of Unipetrol, a merger was set up with Unipetrol/Orlen Group. Now, after the merger (December 31, 2015), PIB is a part of ORLEN Unipetrol RPA as ORLEN Unipetrol RPA s.r.o. – POLYMER INSTITUTE BRNO, o.z.

# **RESEARCH & DEVELOPMENT** POLYMER SYNTHESES

### **POLYMER SYNTHESES**

The polymer syntheses research in PIB has nearly 50 years of tradition and involves highly qualified personnel and special laboratory equipment constructed through its expertise acquired over decades of experience. All polymerisation components are treated a pure, inert atmosphere. Synthesis is done in proprietary polymerisation reactors equipped with fast and accurate systems for processing, controlling and data collection. The experimental synthesis conditions can be varied, and much effort is placed on customising them to industrial customers' needs. Some reactors are equipped with electrostatic charge sensors to monitor the static charge created in the polymer bed during the polymerisation reaction.

The experimental equipment allows us to study the effect of various additives on the polymerisation kinetics and the catalyst system's performance, including in-situ stabilisation, nucleation, electrostatic charge eliminating agents and catalyst poisons. The synthesized polymers are further tested for details of their properties, such as inner microstructure, polymer particle morphology and mechanical features.

The polyolefin research teams predominantly focus on ethene and propene polymerisation in the presence of heterogeneous Ziegler-Natta, chromium and metallocene catalyst systems.



# POLYMERISATION EQUIPMENT

### **STAINLESS-STEEL COMPUTER CONTROLLED BATCH REACTORS:**

VOLUME	w	1.8 I	2.5 I	4 1	50 I
Monomer & Co- monomer continuously charged*	<b>propene</b> ethene butene	<b>ethene</b> propene butene pentene hexene	<b>ethene</b> propene butene pentene hexene	ethene propene butene pentene hexene	ethene propene butene pentene hexene
H2 continuously charged	NO	YES	YES	YES	YES
Static charge monitoring	NO	YES	NO	NO	NO
Max. pressure	2.0 MPa	4.0 MPa	3.0 MPa	4.0 MPa	2.5 MPa
Max. temperature	45 °C	120 °C	100 °C	120 °C	100 °C
Yield	(0.5–1.0) g	(0.1–0.5) kg	(0.1–0.7) kg	(0.1– 1.0) kg	(0.5–12) kg
Polymerisation mode	stopped-flow	gas, bulk HC slurry	gas HC slurry	gas, bulk HC slurry	gas
Number of reactors	1	6	2	2	1

\* connection of the reactor to a GC apparatus ensures the replenishment of consumed comonomers and hydrogen by suitable mass flow controllers





# POLYETHYLENE **SYNTHESIS**

### **POLYETHYLENE SYNTHESIS**

The PE research aroup has long-term experience with the synthesis of various types of catalyst systems, i.e., ZN and Cr-based. The group traditionally supports the production unit in Litvínov with modified or developmental catalysts which innovate the produced polymer grades.

### **DEVELOPMENT OF CATALYTIC SYSTEMS FOR PE PREPARATION**

Because ORLEN Unipetrol RPA is licensed to produce its own catalyst for production units, a significant part of research is dedicated to PE and the development and modification of new catalyst systems. By modifying catalyst systems created at the PIB laboratories, **ORLEN Unipetrol RPA** innovates its product portfolio.

Because commercial units use our supported catalyst systems, much of our research work is on catalysts which anchor to the surfaces of carriers, i.e., mainly silica gels. The support used in these systems is a part of the catalyst which contributes to final polymerisation performance, for example, altering the kinetic profile or incorporating comonomer or transfer reactions. From that point of view, several techniques which characterize carrier properties have been developed over time.

We focus on support for commercial Ti-, Cr-, or Zr-based catalyst systems. Research of model systems based on these transition metal salts provide highly valuable information which can be applied to optimize of commercial catalysts or develop of new catalyst systems.

# **POLYETHYLENE** SYNTHESIS

The typical methods used in the process of catalyst development or characterization of silica gel properties are as follows:

- Catalyst synthesis and its handling is performed under strictly inert conditions involving the use of high vacuum lines or glove boxes together with highly purified nitrogen stands for standard operation procedures.
- A lab-scale quartz activator (approx. 15 g) and pilot-scale equipment (up to 5 kg) are used for silica or Cr-based catalyst activation processes.
- Characterization of silica gels involves a complex method which consists of several steps to attain the required quality: (i) chemical analysis, (ii) analysis related to a specific surface area (S.A.), particle size distribution (PSD) Malvern, pore volume (P.V.), DTA, SEM and TEM microscopy, and (iii) analysis of -OH group content and their distribution using IR, NIR and DRIFT techniques.

All these studies focus on obtaining a better understanding of the relationship between catalyst composition and catalyst polymerisation performance and aim to innovate polymer properties. The development of two proprietary Cr- and Ti-based catalyst systems resulted from systematic research in this field. The Cr-based catalyst (covered by patents – CZ 288056 B6 and US 6.569.966 B1) has been used in industrial production of film and pipe grades.

PIB can perform catalyst synthesis at the customer's request.

Catalyst synthesis represents one part of PIB research and is completed with an evaluation of catalyst polymerisation performance and final polymer properties.



### DEVELOPMENT AND OPTIMIZATION OF POLYMERISATION PROCESSES IN THE LABORATORY

The typical polymerisation line consists of a set-up with a reactor connected online to a GC which allow the consumed gaseous components to be continuously replenished. This system permits polymerisation tests under a constant mixture composition.

The reactor design allows simulation of the commercial fluidized bed process which requires a seed bed. Analysis of the polymer formed afterwards is thus easier and precise.

Efficient purification system for all gases (ethylene, comonomer, nitrogen and hydrogen) allows even chromium oxide or metallocene catalysts to be tested without the addition of a scavenger. The polymerisation run starts just after all parameters (temperature, composition of polymerisation mixture) are set at the required levels in the same manner as a commercial polymerisation process.

These test conditions allow commercial and developmental catalysts to be compared under real conditions. Kinetic profiles can also be studied, and the formation of agglomerates, deposits on the reactor walls or even the formation of by-products (oligomerisation or hydrogenation) can be observed. These can be easily compared with the structure of the obtained polymer, and a comprehensive view of the tested catalyst system can be produced.

# **POLYETHYLENE** SYNTHESIS

### **INNOVATION OF POLYMER PROPERTIES**

Analysis of the polymer structure is essential in understanding the relationship between the catalyst performance and physico-mechanical properties of the resulting polymer. The techniques applied for polymer structure assessment are as follows:

- Analysis of structural parameters using GPC, FTIR, DSC.
- TREF temperature rising elution fractionation (solution method).
- SIS/DSC stepwise isothermal segregation (melt fractionation) techniques.

The common mechanical properties of polymers are evaluated using standard methods (such as tensile or flexural tests, ESCR) and many other methods related to certain application areas, for example:

- Plain Stress Impact PSI an alternative technique used widely to estimate the polymer resistance towards Rapid Crack Propagation (RCP simulates the standard S4 test).
- Full Notch Creep Tests (FNCT test) technique for evaluating the material resistance against Slow Crack Growth (SCG); this technique is typically used for pipe and blow moulding grades.

### **OTHER ACTIVITIES**

- Development of cost effective chromium-based catalyst systems.
- Titanium-based catalyst systems for the production of syndiospecific polystyrene.
- Study of the substituent effect on a CGC catalyst complex in the formation of ethylene styrene interpolymers.
- > Development of laboratory techniques for the simulation of CSTR cascade mode polymerisation processes.
- > Study of the effect of the silica support activation process and the effect of the concentration and arrangement of hydroxyl groups on silica surfaces.
- Evaluation of hydroxyl group content based on volumetric analysis via the interaction of silica support with organoaluminium compounds.





# **POLYPROPYLENE** SYNTHESIS

### **POLYPROPYLENE SYNTHESIS**

The process involves homo- and copolymerisation (random and sequential) of propene with other 1-alkenes, specifically in gas phase or liquid propene or hydrocarbon slurry conditions. The polymer synthesis is continuously monitored during the process, and the data obtained are used to assess the polymerisation kinetics.

# The synthesised polymers are studied from the point of view of their structural and mechanical properties:

Melt flow rate, soluble fractions, extractable fractions, density, particle size distribution, bulk density, porosity, pourability and polymer particle morphology

 Detailed structure characterisation using DSC, TGA, 13C-NMR, pulsed 1H-NMR, GPC, FTIR, S SA/DSC, SIS/DSC, TREF, VOC and other methods
 Tensile strength, toughness, hardness, heat resistance

# **POLYPROPYLENE** SYNTHESIS

### **PROJECT EXAMPLES**

- > Development of new or modified PP grades (homopolymers, random and sequential copolymers)
- Examination of the effect of specific impurities ("polymerisation poisons") in raw materials on the kinetics of the polymerisation process and the polymer properties; analytical evaluation of raw materials used for industrial olefin polymerisation
- Fine purification of raw monomers to polymerisation grade purity (down to a level below 10 ppb of the most critical impurities)
- > Development of catalyst systems based on commercial catalysts to fit specific industrial processes
- Electrostatic charge monitoring and control for specific polymerisation systems
- Development of in-situ polymer additive processes
- > Cooperation with major world producers of catalysts for olefin polymerisation
- > Theoretical study of polymerisation kinetics
- Determination of the concentration of active centres
- > Assessment of polymerisation kinetics suitable for catalyst system optimization under the desired industrial conditions





# **SPECIAL POLYMERISATION PROCEDURES**

### POLYMERISATION KINETICS EVALUATION "SECOND CATALYST DOSE"

Accurate data collection during polymerisation and comprehensive evaluation of kinetics is the basis of assessing polymerisation kinetics in industrial processes. For this purpose "the second catalyst dose" is added to the polymer bed at polymerisation temperature and pressure.

## MOLECULAR MODELLING OF ZIEGLER-NATTA CATALYST INTERACTIONS

We can perform molecular modelling such as conformational search and energy evaluation of Ziealer-Natta catalysts interactions with various polymerisation components, including internal and external donors, cocatalysts, catalyst poisons, etc. From the results, the correlations between theoretical and experimental data can be constructed and used to predict of new structures and also visualize trends in catalyst performance and the resulting polymer properties.

### **HOMO-POLYMERISATION SOFTWARE**

Software enables the modelling of the two-step homo-polymerisation laboratory process and the resulting polymer properties of homopolymer grades. The enclosed figure shows an example of this type of calculation.

- Polymerisation kinetics patterns and corresponding polymerisation yields
- Molecular weight distribution (MWD) curves
- Values of molecular weights Mn, Mw, Mz and polydispersity index Mw/Mn
- MFR of polymer produced during particular homopolymerisation steps and MFR of the final material

(input data: polymerisation temperature, hydrogen concentration and duration of both polymerisation steps)

# **SPECIAL POLYMERISATION** PROCEDURES

### MODELLING OF INSTANTANEOUS POLYMERISATION PROCESSES STOPPED-FLOW FACILITY FOR 1-ALKENE POLYMERISATION

The high pressure stopped-flow apparatus was constructed for the synthesis of special polypropene-blockpoly( propene-co-ethene) copolymers and yields up to 1.0 g. Polymerisation is performed in liquified propene monomer. Stopped-flow polymerisation is performed in a thin glass tube of 0.4 – 2.0 ml volume. Heterogeneous Ziegler-Natta catalyst dispersed in liquified propene is activated by triethylaluminium in a special mixing zone, then the activated catalyst flows through the first polymerisation zone, where the propene homopolymer component is synthetized. Ethene comonomer is added to the second zone and poly(propene-co-ethene) is produced, finally reaching a vessel with a quenching agent. This technique allows polymerisation experiments with a shorter-than-average (several tenths of a second)lifetime of growth in the polymer chains to be conducted and the production of real polypropene-block-poly(propene-co-ethene) copolymers.

The successful preparation of block copolymers provides new insight into the nature of active sites formed on heterogeneous Ziegler-Natta catalysts and also allows the investigation of the role of block copolymers in heterogeneous materials which consist of a crystalline matrix (polypropene) and inclusions of amorphous poly(ethene-co-propene) rubber (impact resistant sequential copolymers produced in industrial two-reactor technologies).





# MATERIALS ENGINEERING AND POLYMER PROPERTIES

### MATERIALS ENGINEERING AND POLYMER PROPERTIES

The team is active in the research and development of thermoplastic materials and monitors new trends in the field of plastics processing, compounding and polymer applications. Their main areas of interest are reinforced plastics, especially polyolefins, reactive compounding, special polymer blends, thermoplastic elastomers, organosilane crosslinkable polymers, adhesives, electroconductive compounds, flame retardant polymers and many others.

The modified materials and products can be subjected to various physicomechanical tests, flammability tests, thermal analyses, microscopy or x-ray analysis.

These activities are typically applied in the development of materials designed for automotive, electro-engineering, construction, cabling, pressure pipe, agricultural, and mining purposes, and many others.

# **MATERIALS ENGINEERING** AND POLYMER PROPERTIES

### LABORATORY OF PLASTICS PROCESSING AND RHEOLOGY

Expertise in the structure of polymers and their rheological behaviour. Compounding and conversion are an excellent base for the following activities:

- Grafting reactions (maleic anhydride, organosilane, glycidyl methacrylate, etc.)
- Polymer blending and subsequent dynamic crosslinking
- Compatibilisation of polymer blends
- Extruder polymerisation
- Adhesives
- Controlled rheology PP
- Oxo- and bio-degradable polymers
- Modification of effects
- Flame retardant plastics (halogenated and halogen free)



### MECHANICAL TESTING LABORATORY

#### Standard test methods:

- Tensile properties
- Flexural properties
- Compression tests
- Tear and peel tests
- Hardness
- Impact tests Charpy, Izod
- Falling dart tests
- Vicat/HDT tests
- Density
- Coefficient of linear thermal expansion

#### Special testing methods:

- Full-notch creep test (FNCT)
- Crack opening creep test (PENT)
- Time to failure of plastic pipes (hydrostatic)
- Resistance to rapid crack propagation (S4)
- Creep tests
- Antistatic properties
- Dynamic mechanical analysis (DMA)
- Morphology and fractography



# **STABILISATION AND DEGRADATION** OF POLYMERS

### **STABILISATION AND DEGRADATION OF POLYMERS**

The laboratory studies the UV, heat and long term thermal stability of polymers, kinetics of degradation and behaviour of additives in polymers and develops stabilising formulations. Processing stability is evaluated using the multiple extrusion technique (polypropylene) or by measuring the torque necessary for extrusion or kneading of the molten polymer (HDPE).

Thermooxidative stability (accelerated tests) is examined in open test tubes or in an oven with forced air circulation at temperatures of 50-150 °C depending on the type of examination and polymer. To a limited extent, the oxygen absorption method may be used or the OIT can be measured.

UV stability is assessed in accelerated ageing tests or by exposure to natural weathering conditions at the PIB weathering station with the option of terrestrial light intensity monitoring.

The chemical and physical behaviour of additives in polymers may be studied by measuring the diffusion kinetics, exudation rate and solubility of additives in the polymer matrix and by following the chemical changes of the additives in the course of stabilisation process. The nucleation, antistatic and optical brightening effects and mutual interactions of particular additives in the package and the phenomena associated with water carry over during the production of oriented slit-tapes can also be assessed.

The group has expertise in the development of stabiliser formulations for polyolefins and additive masterbatches and also extensive experience in production plant trouble-shooting.

### CHEMICAL ANALYSIS AND POLYMER STRUCTURE ANALYSES

The laboratory provides testing services, trouble-shooting, research and quality control for a broad range of clients, markets and industries.

### Instrumentation equipment enables the examination of:

- Polymer molecular structures
- The content of non-polymeric components and their character, for example fillers, stabilisers and other additives, impurities, contaminants, residues (e.g., traces of polymerisation catalyst residues, auxiliary materials, solvents)
- Input material purity (monomers, auxiliary materials and additives)
- Identification of polymers or unknown compounds present in the system



### LABORATORY OF SEPARATION METHODS

Expertise in polymer structures and the rheological behaviour of polymers. Compounding and converting Chromatographic methods (GC/MS, GPC, HPLC) are used for:

- Identification and determination of stabilisers and other additives in plastics (PP, PE, ABS, polystyrene, polycarbonates, PVC) and their transformation products
- Analysis of deposits formed during plastics processing
- Identification of bad-smelling volatile organic compounds using the head-space technique (static and dynamic) in combination with closed loop stripping analysis, and head-space analysis with gas chromatography and mass spectroscopy
- Determination of trace impurities in monomers (CO, CO<sub>2</sub>, AsH<sub>3</sub>, PH<sub>3</sub>, H<sub>2</sub>S, COS, mercaptans, ethylenoxide, alcohols, ketones)
- Determination of residual monomers and other volatile compounds in plastics using the headspace technique with gas chromatography and mass spectroscopy
- Analysis of oils, waxes, solvents and emulsifiers
- Analysis of paint thinners
- Gas analysis
- Water analysis



### LABORATORY OF ELEMENTAL ANALYSIS AND ELECTROCHEMISTRY

#### The following can be performed at this laboratory:

Determination of water content using the Karl Fischer procedure (including coulometric determination) in liquid and solid samples
 Determination of carbon-black and ash content in plastics
 Determination of active oxygen (peroxide level)
 Determination of F, Cl and Br in plastics
 Determination of conductivity and pH

### LABORATORY OF SOLUTION METHODS

- Determination of average relative molecular weights and distribution curves of HDPE, PP and their copolymers by using gel permeation chromatography (GPC)
- Determination of viscosity number and limiting viscosity number of polymers and copolymers in a given solvent and at a certain temperature according to ISO 1628 and ISO 307
- GPC determination of branched PE: connection of the GPC apparatus with the viscometer detector enables determination of the branching coefficient, branching factor and the distribution of branches across a range of measured molecular weights
- Determination of structure heterogeneities of PE and PP using the TREF method in analytical and preparative modes

### LABORATORY OF SPECTRAL METHODS

Infrared spectroscopy is used for qualitative studies such as:

- Identification of polymer materials, stabilisers and additives
- General identification of substances and determination of purity
- FTIR spectroscopy
- Identification of small objects in materials using IR microscopy
  Continuum from size 15 × 15 μm

#### For quantitative studies:

- Determination of composition of copolymers, additive content in polymers, monitoring degradative changes in polymers
- Degree of branching, degree of crystallinity of semicrystalline polymers, dichroic ratio in oriented polymers

#### Polymer UV-VIS-NIR spectroscopy is applied mainly for:

- Determination of organic solvent purity
- Photometric control of stabiliser dosage in PP and PE
- Determination of -OH groups in silica supports
- Determination of polystyrene content in styrene (ASTM D2121)
- Determination of p-tert-butylcatechol in styrene (ASTM D4590)
- Spectrophotometric determination of some metals

#### NMR spectroscopy – examples of use:

- Structure identification, purity determination, degree of deuteration
- > Determination of isomerisation degree of polymer chains
- Branching of polymers, detection, identification and quantification of branches
- Structural defects of macromolecules, determination of tacticity
- Analysis of propylene/ethylene or other 1-olefin copolymers, determination of number average molecular weight by means of quantitative end group analysis

#### Atomic absorption spectroscopy (AAS)

AAS is used for the qualitative and quantitative determination of metals. Possible applications: analysis of catalytic systems, traces of catalyst residues in polymers, content of fillers, determination of metals in fillers etc.

# Atomic absorption spectroscopy with electrothermal analysis (ETA-AAS)

Electrothermal analysis AAS in connection with microwave decomposition is able to determine traces of metals at ppb levels.

Possible application: determination of impurities in monomers.





### LABORATORY OF SPECIAL METHODS FOR EVALUATING MATERIALS

Thermal analysis (DSC, TGA) is used especially for:

- > Determination of melting and crystallisation temperature
- Determination of structure heterogeneity of PE (PP) by SIS(SSA)/DSC
- Determination of the degree of crystallinity in polymers and composites
- Measurement of the glass transition temperature
- Analysis of the blends of polymers according to their characteristic melting temperatures
- Study of the kinetics of crystallisation
- Measurement at low temperatures (to –90 °C)
- Determination of the thermooxidative stability of polyolefins
- Measurement of the thermal stability and mass changes in the temperature range 25 – 1000 °C
- > Determination of the organic and inorganic content in composites

- Polymer chain segment distribution and short chainbranching in copolymers, composition and crystallisability distribution
- Apart from GPC, isothermal crystallisation (measured by DSC) and NMR, fractional crystallisation methods either from solutions or polymer melts are used, for example:
- Temperature raising elution fractionation (TREF), composition distribution, crystallisability distribution
- Successive multistep isothermal crystallisation (SIC)
- Successive self-nucleation annealing (SSA, SNA)
- Combination of methods, e.g. TREF-SIC, TREF-SNA

#### Microscopy

This method involves both optical microscopy for the examination of samples in transmitted and reflected light and electron microscopy (fitted with SEM/EDX probe) applied for the morphological studies of polymers, composite materials, fillers and other solid materials.

Typical examples of the application of these methods are:

- Determination of particle size and shape
- > Dispersion of inorganic fillers and elastomers in composites
- Qualitative and semiquantitative analysis of elements (EDX, Z = 11+ or Z = 5+) in the ash of polymers and composites and in screenpack deposits and other solid substances

#### X-ray analysis (XRD)

This method is employed for both qualitative and quantitative analysis of samples, determination of crystallinity, the size of crystalline domains and the degree of orientation (difractor SIEMENS D-500). Evaluation of powdered materials

- Particle size determination (laser analyser, 0.02 2000 μ m)
- > Determination of the whiteness of powders and composite materials

#### Flammability testing

- ▶ LOI determination according to ISO 4589, ČSN 640756
- UL-94 procedure (HB, V-0, V-1, V-2)
- Glow wire test (DIN IEC 695, ČSN 345615)
- Combustible building materials (DIN 4102-1/B2)
- Determination of the burning behaviour of automotive interior materials (ISO 3795)

#### Automotive test methods

- Component contamination according to ISO 16232
- Emission properties of materials (PV 3341, PV 3900, PV3015, PV3925)
- Gas fading
- Flammability testing



# ACCREDITED SERVICES

The Czech Accreditation Institute has granted PIB a Certificate of Accreditation as an accredited testing site for mechanical, analytical and special properties of materials. A list of valid Accreditation Certificates and Accredited Tests is located directly on the website of Czech Accreditation Institute (https://www.cai.cz/?subjekt=1380-unipetrol-rpa-s-r-o-polymer-institute-brno-odstepny-zavod&lang=en).



# RESEARCH & DEVELOPMENT

# **AND CONSULTANT SERVICES**

Even during reliable processes of polymer manufacturing and conversions, problems can occur and expert troubleshooting is needed for rapid and efficient solutions.

We offer a unique multidisciplinary approach to problem solving, starting from chemical analysis of raw material purity and polymerisation catalyst activity, examination of the chemical engineering principles in production units, and finally evaluation of the processing conditions and additive package performance. We can suggest and in most cases supply suitable polymer raw materials and additive and colour concentrates according to customer's specific needs. The experience of our staff and the analytical and testing facilities at the institute have an important role in our services.

# ORLEN UNIPETROL RPA POLYMER INSTITUTE BRNO CONTACTS

+420 513 207 911

pib@orlenpolymer.cz

ORLEN Unipetrol RPA s.r.o. – POLYMER INSTITUTE BRNO, o.z. Tkalcovská 36/2 602 00 BRNO

Czech Republic

Identification Number: 27597075 Tax Identification Number for VAT: CZ699000139 (group registration) Tax Identification Number: CZ27597075

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