

Precise analysis of PFAS and micropollutants – how the West Pomeranian University of Technology in Szczecin supports the industry in wastewater monitoring

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Faculty
of Chemical Technology
and Engineering



West Pomeranian
University of Technology
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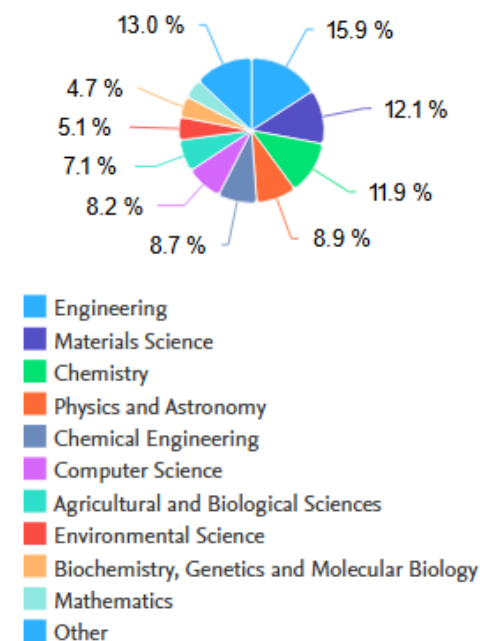
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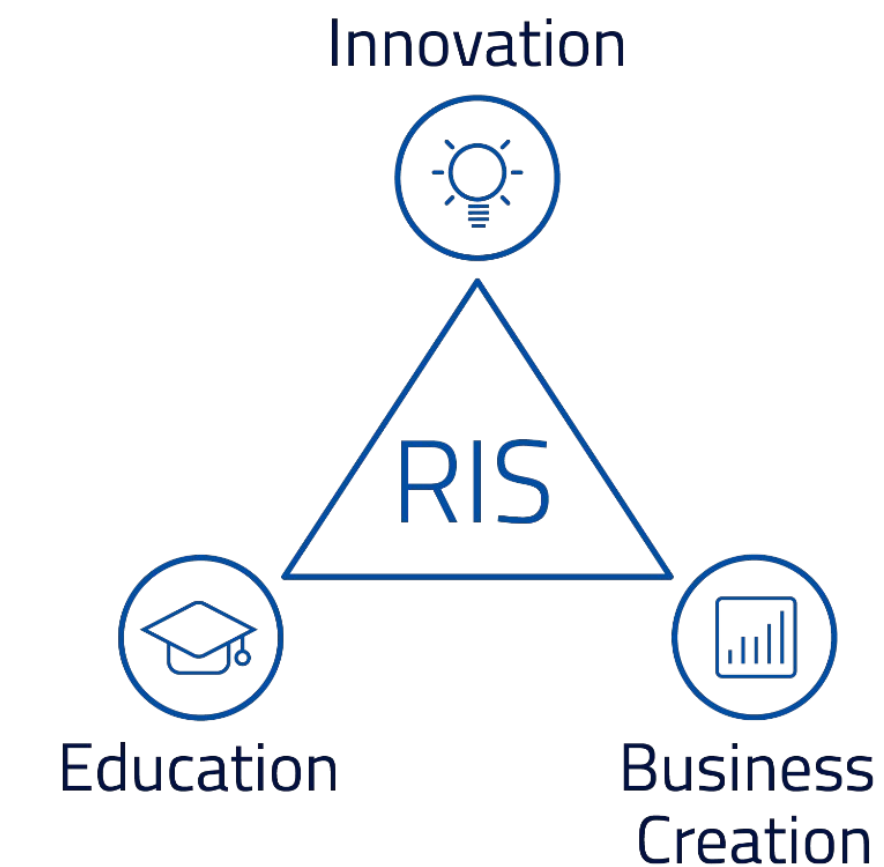
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Subject area	Documents
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Agricultural and Biological Sciences	1,924
Environmental Science	1,386
Biochemistry, Genetics and Molecular Biology	1,266
Mathematics	1,168

Subject trends



Our strategy is based on the so-called "*knowledge triangle*" that highlights the need for an integrated approach to research, innovation and teaching.



Republic
of Poland

European Union
European Regional
Development Fund



Information about WPUT



Center for Advanced Materials and Manufacturing Process
Engineering

The Intelligent Development Operational Programme 2014-2020 (IDOP)

Priority axis 4.2 “Development of modern research infrastructure for the science sector”

Agreement No.: POIR.04.02.00-00-D0018/20-00

Center for Advanced Materials and Manufacturing Process Engineering (CZMIPW)



**Republic
of Poland**

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European Regional
Development Fund



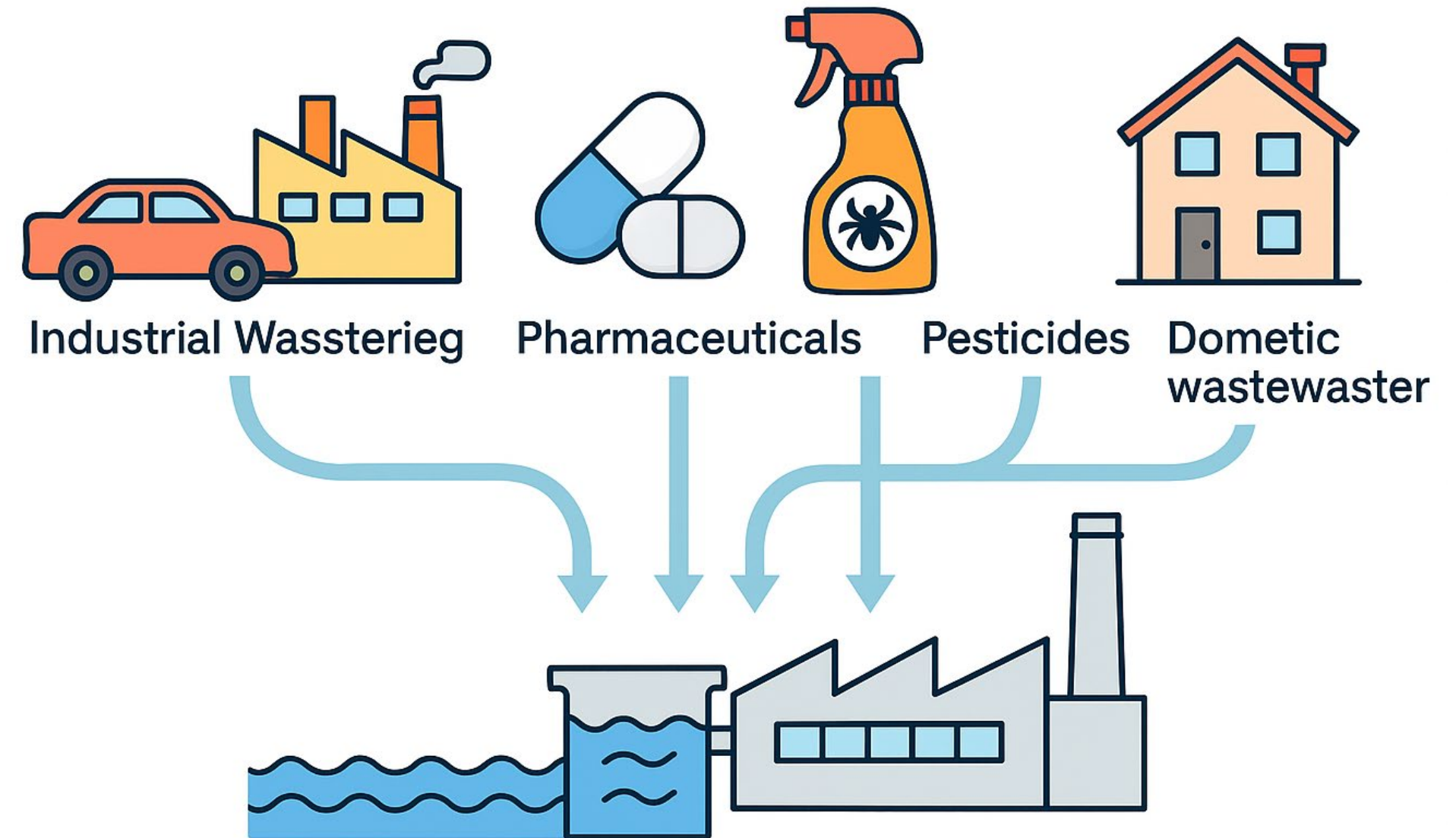
Challenges of PFAS and Micropollutants in Wastewater

- **PFAS (Per- and polyfluoroalkyl substances):**

- Persistence in the environment (can last hundreds of years in soil and water).
- Widespread use (waterproof clothing, cleaning agents, paints, chemical industry).
- Health issues associated with their presence: carcinogenicity, liver damage, hormonal disruption.

- **Micropollutants:**

- Presence of pharmaceuticals, pesticides, microplastics in wastewater.
- Increasing awareness and regulatory efforts.



The Problem of PFAS and Micropollutants in Wastewater

- **Water contamination:**

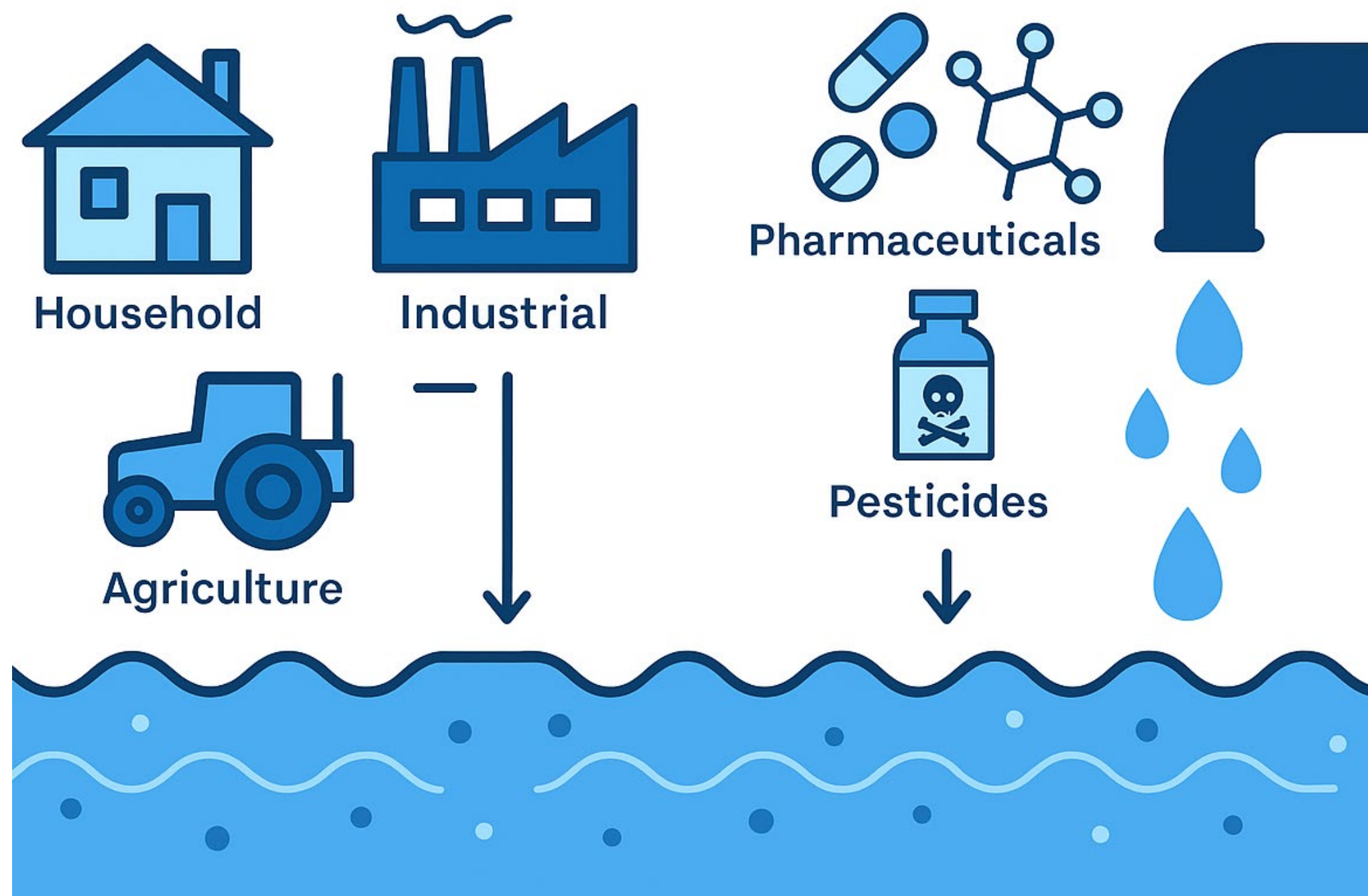
Micropollutants such as PFAS, pharmaceuticals, microplastics enter surface waters through industrial, domestic wastewater, and agriculture.

- **Scale of the issue:**

Wastewater is the main source of micropollutants that can infiltrate groundwater.

- **Health effects:**

Decline in drinking water quality, diseases related to toxic substances.





Proper Sampling for PFAS Testing – Key Considerations

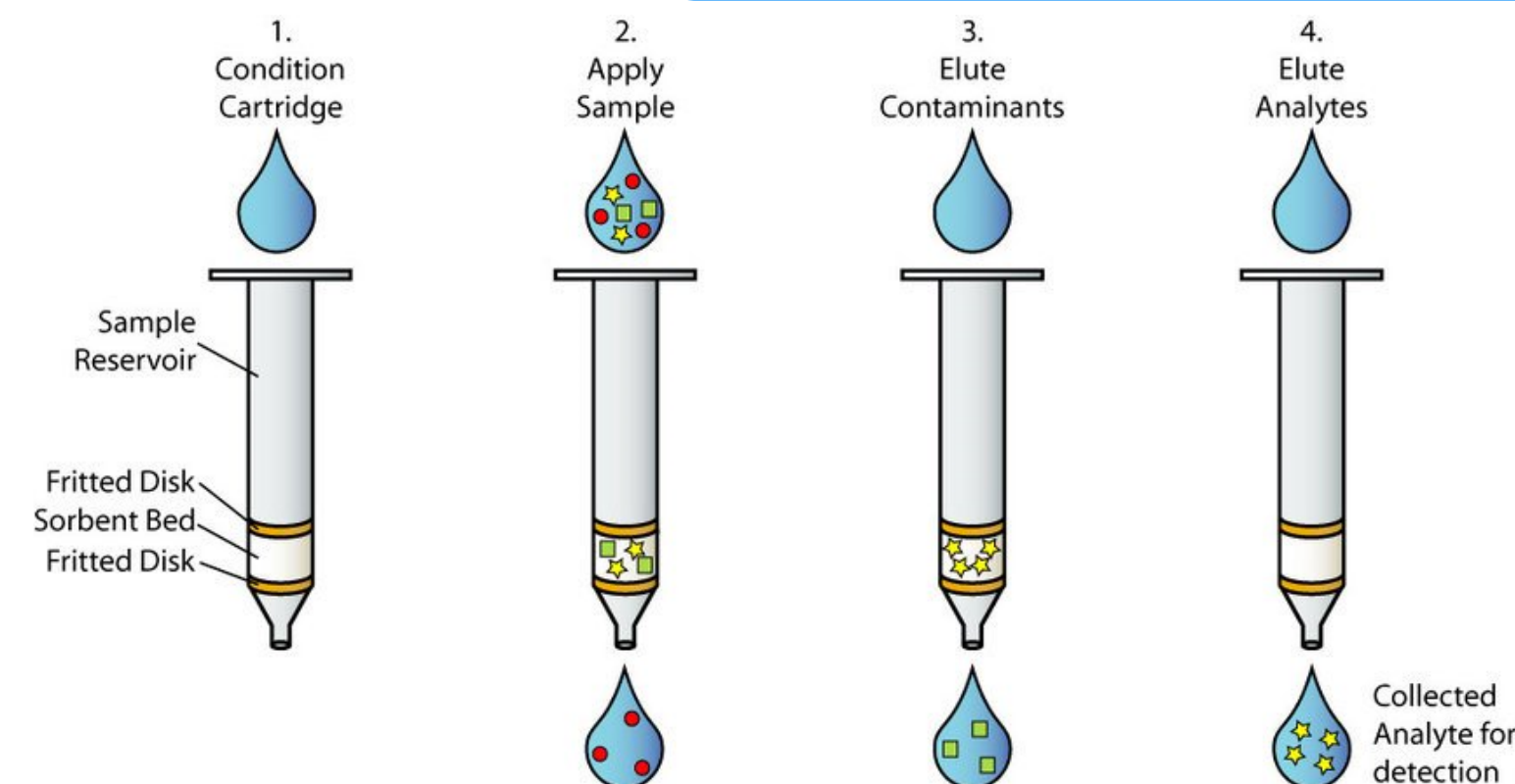
Approved materials that are considered safe and PFAS-free

include:

- Stainless steel
- HDPE (High-Density Polyethylene)
- Vinyl
- Silicone
- Polypropylene (PP)
- Natural ice produced from verified PFAS-free water
- LDPE (except for LDPE verified as PFAS-free)
- Glass (except for glass verified as PFAS-free; but unsuitable for storage)
- Teflon and its derivatives
- Materials containing any fluoropolymer
- Clothing, shoes, and materials containing Gore-Tex or other water- or stain-resistant synthetic materials
- Clothing washed with fabric softeners
- Personal hygiene products
- Aluminum foil
- Food containers and packaged food

Sample preparation

- **Volume of sample: 100 ml**
- **SPE cartridges:** BEKOLut PFAS WAX/GCB, 200 mg/50 mg (WAX/GBC)/6 ml
- ISO method ISO/DIS 21675
- SPE Procedure:
 - Column Conditioning:
 - ✓ Rinse with: 4 mL 0.3% NH_3 in methanol → 4 mL methanol → 5 mL water
 - Sample Loading - apply samples at 4 mL/min flow rate
 - Wash: 2 x 4 mL water
 - Elute: 2 x 4 mL 0.3% NH_3 in methanol
 - Dry Down: evaporate to dryness
 - Reconstitute: to 1 mL with LC mobile phase



Evaporate to dryness with
nitrogen using a TurboVap®
(1.5 bar at 40°C for 1 hr)





Sample analysis – HPLC-MS/MS

Equipment

Vanquish Flex UHPLC system combined with Orbitrap Exploris 120

Determination of micropollutants

LC conditions:

- Column: Hypersil GOLD™ aQ 100 x 2.1 mm, 1.9 μm
- Column temp. 25 °C
- Flow rate: 0.3 mL/min
- Solvent A: Water containing 0.1% acetic acid
- Solvent B: Methanol containing 0.1% acetic acid
- Gradient:

POSITIVE IONS

Time	%Solvent B
0	0
1	0
5	100
6.5	100
6.6	0
8.5	0

NEGATIVE IONS

Time (min)	%SolventB
0	10
1	10
3.5	100
6.5	100
6.6	10
8.5	10

Optimized MS parameters:

Spray voltage: positive: 3 kV; negative: 2 kV
 Sheath gas: 60 arb
 Auxiliary gas: 15 arb
 Sweep gas: 1 abs
 Ion transfer tube temp.: 300°C
 Vaporized temperature: 375 °C
 Cycle time: 0.35 s
 Q1/Q3 resolution: 0.7 Da
 CID gas: 2 mTorr





Sample analysis – HPLC-MS/MS

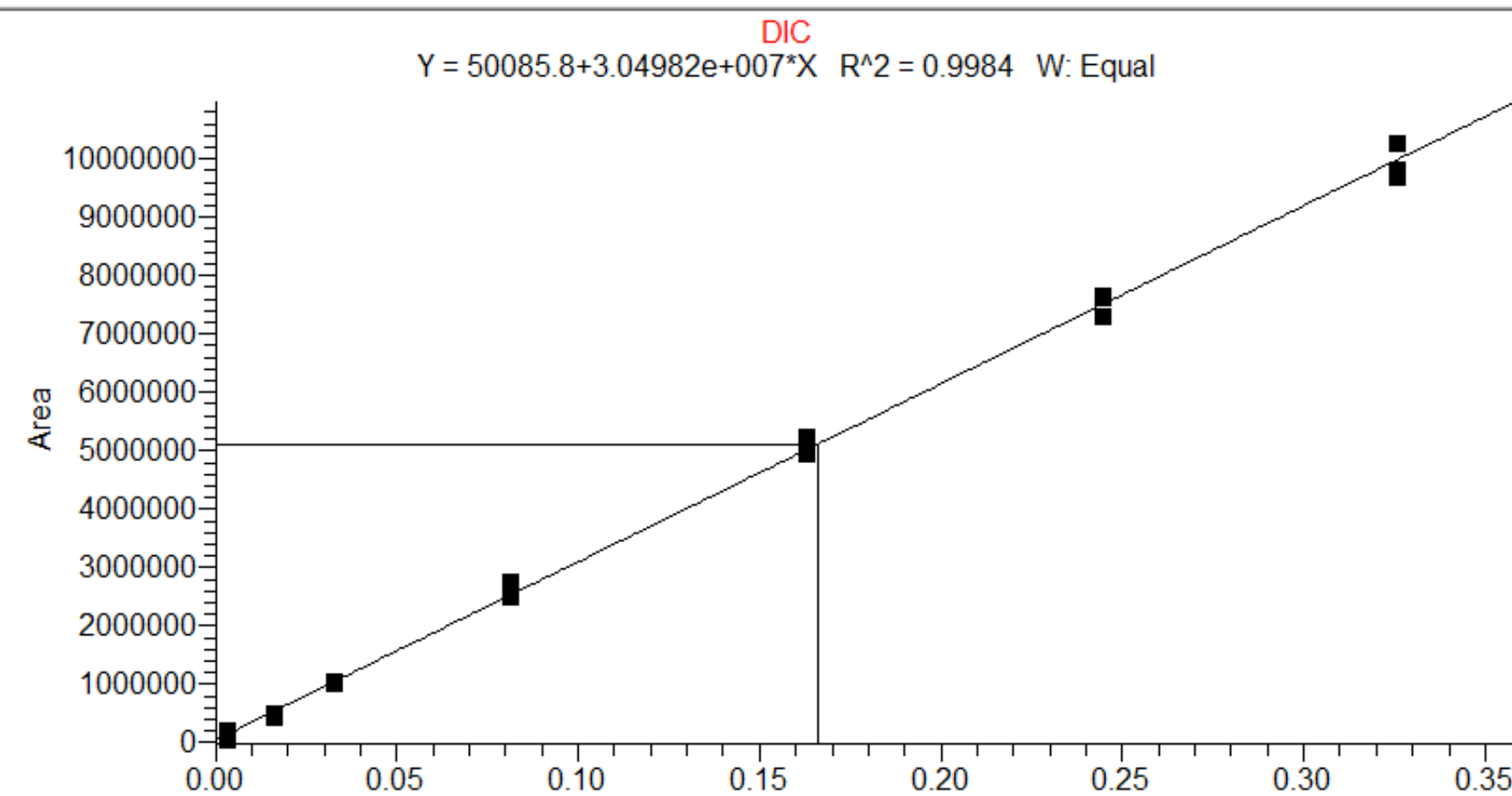
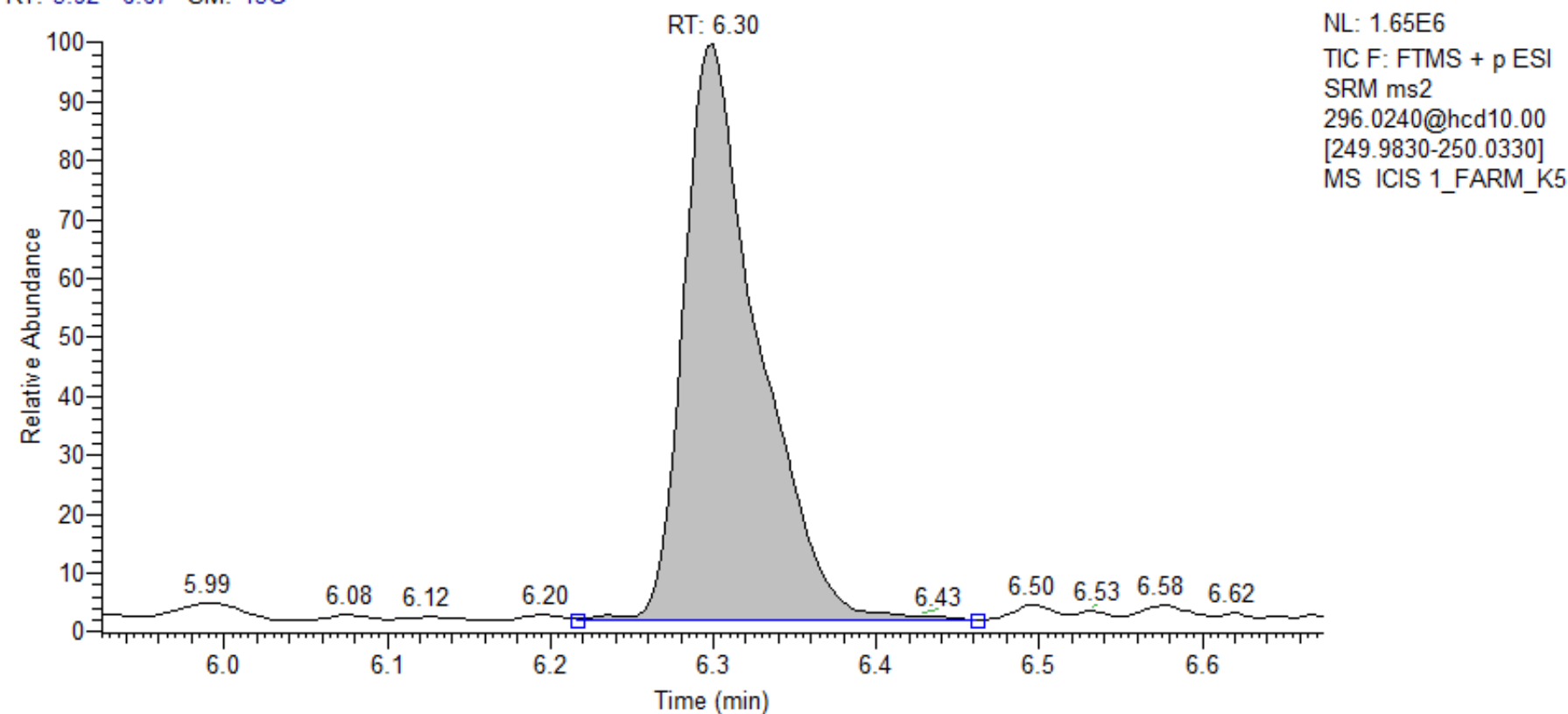
Determination of micropollutants

Example results and calibration curve for diclofenac

1_FARM_K5 (Method Settings)

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RT: 5.92 - 6.67 SM: 15G



Sample analysis – HPLC-MS/MS

Determination of PFAS (Per- and polyfluoroalkyl substances)

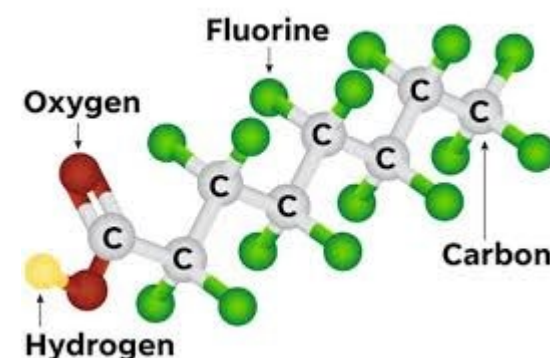
LC conditions:

- Isolator column: BDS Hypersil™ C18 50 x 2.1 mm, 5 µm
- Analytical column: CHROMSHELL®C18 Polar HPLC column, 150 x 3.0 mm, 2.6 µm
- Column temp. 45 °C
- Flow rate: 0.5 mL/min
- Solvent A: Water containing 2 mM ammonium acetate, 2% methanol, and 0.1% acetic acid
- Solvent B: Methanol containing 2 mM ammonium acetate, 2% water, and 0.1% acetic acid
- Gradient:

Time (min)	% Solvent B
0	0
1	30
6	45
13	80
14	95
17	95
18	0
21	0

Optimized MS parameters

- HESI source: Negative ionization mode
- Spray voltage: 2.5 kV
- Sheath gas: 50 arb
- Auxiliary gas: 10 arb
- Ion transfer tube temp.: 325°C
- Vaporized temperature: 300 °C
- Cycle time for transitions in negative SRM mode: 0.3 s
- Q1 resolution: 0.7 Da
- Q3 resolution: 1.2 Da
- CID gas: 2 mTorr



EXAMPLES OF APPLICATIONS OF THE DEVICE

The **Orbitrap Exploris 120** mass spectrometer is widely used in various fields, such as **environmental** and **food safety**, **biochemical** and **pharmaceutical analysis**:

SCREENING AND
CONFIRMATION OF
DRUGS

ENVIRONMENTAL
POLLUTION ANALYSIS

PESTICIDE RESIDUES
ANALYSIS

ANALYSIS OF DRUG
RESIDUES

PHARMACEUTICAL
APPLICATIONS

CHARACTERIZATION AND
IDENTIFICATION OF THE
SUBSTANCE





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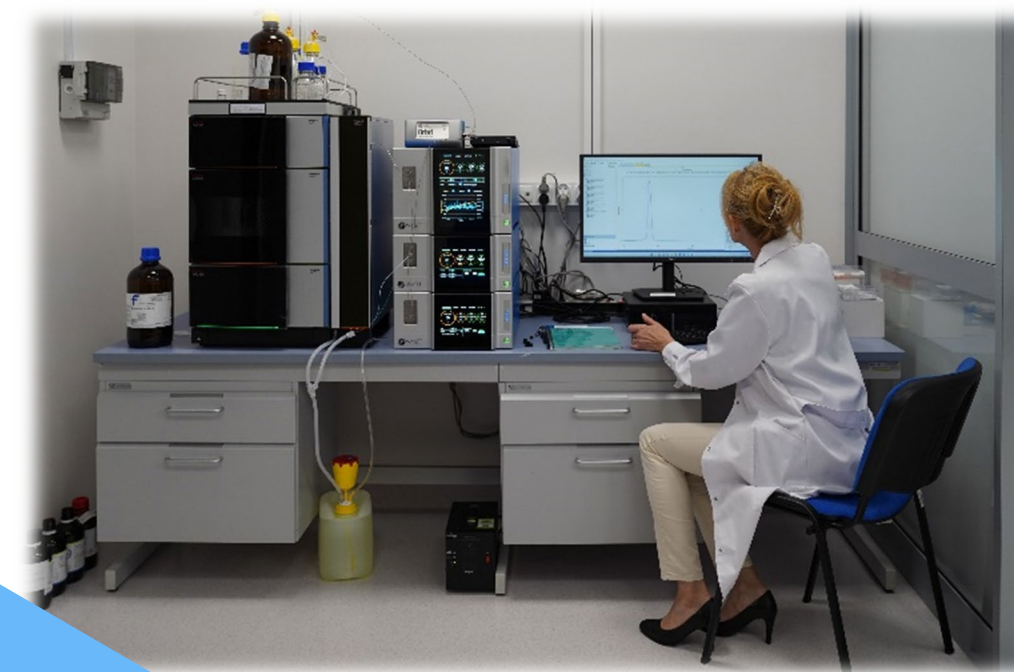
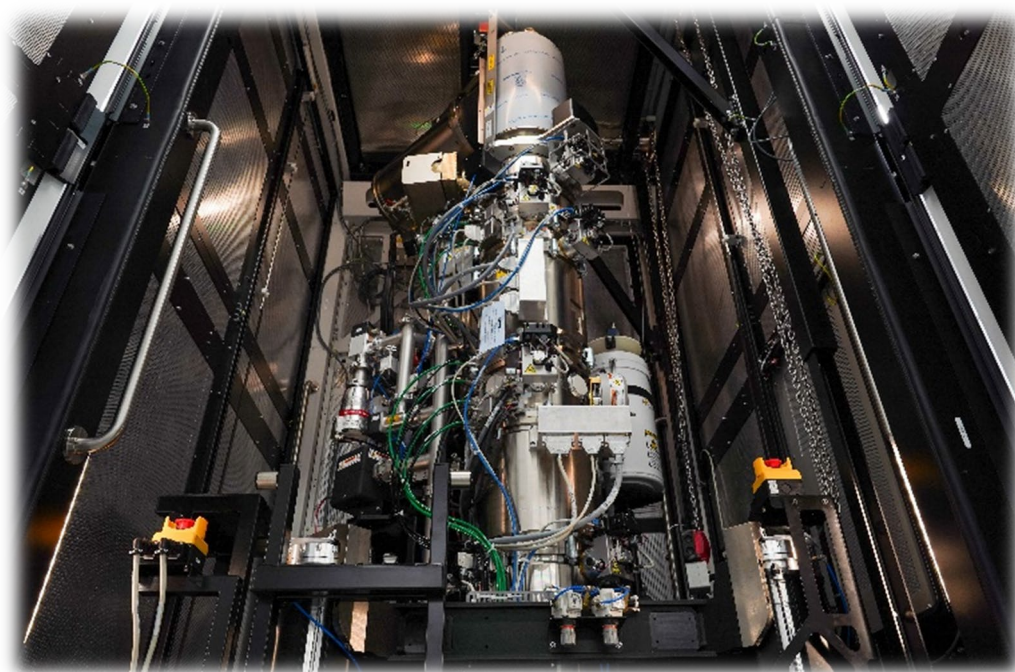
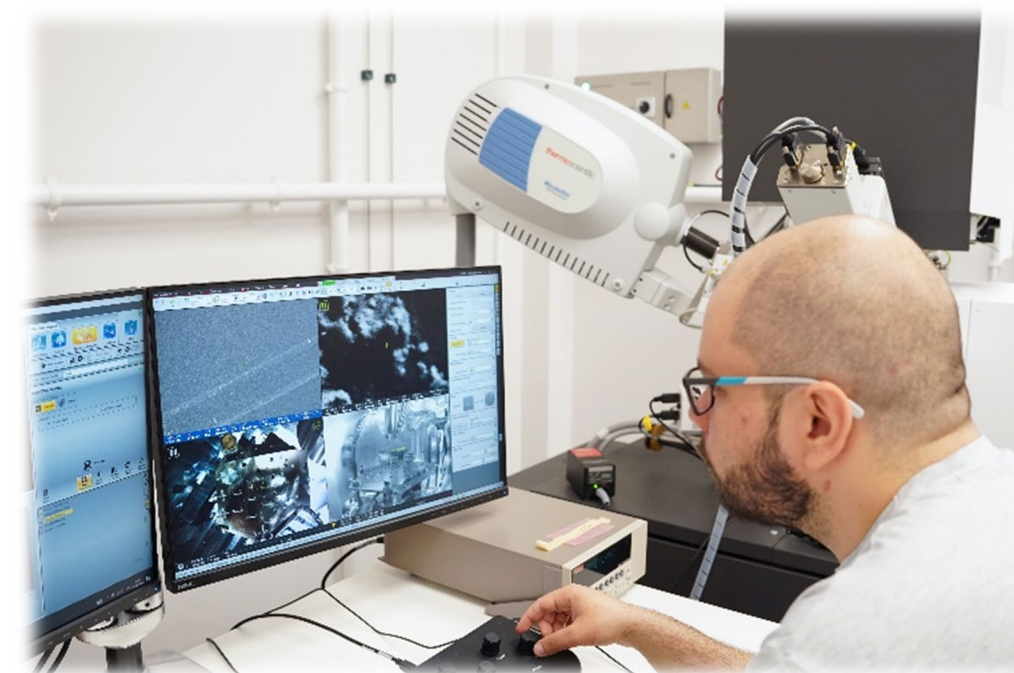


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Center for Advanced Materials and Manufacturing Process Engineering

Center:

- 8 labs
- 18 set-ups
- 67 components of the research infrastructure at the WPUT



European
Funds
Smart Growth



Republic
of Poland

European Union
European Regional
Development Fund





Faculty
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Manufacturing Process Engineering



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Center for Advanced Materials and Manufacturing Process Engineering

Q-TOF High
Resolution
Mass
Spectrometer
(HRMS-Q-
TOF)

MALDI-
TOF/TOF
Spectrometer

GPC/SEC-
MALS Gel
Permeation
Chromatograph

Nuclear
Magnetic
Resonance
Spectrometer
(NMR)

Electron
Paramagnetic
Resonance
Spectrometer
(EPR)

High
Resolution
Transmission
Electron
Microscope
(HRTEM)

Scanning Electron
Microscope with
Energy Dispersive
and Wavelength-
Dispersive X-Ray
Detectors
(SEM/EDX/WDX)

Wavelength
Dispersive
Sequential X-
Ray
Fluorescence
Spectrometer
(WDXRF)

Confocal
Microscope

Flow
Cytometer

Computed
Microtomograph
(m-CT)

Quartz
Crystal
Microbalance
(QCM)

Porometer
capillary

Laser
scanning
microscope
(LSM)

Cone
calorimeter

ICP-MS
inductively
coupled
plasma mass
spectrometer

Ion
chromatograph

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