Usage Policy of BOKU Core Facility Analysis of Lignocellulosics (ALICE)

Usage Conditions and Pricing Policy

Available Service Modes at ALICE

- **Trained user mode**: Independent use of instrumentation after appropriate training by ALICE staff.
- **Full-service mode**: Execution of experiments and, where applicable, data analysis and report generation by experienced ALICE staff.

The appropriate service mode (or a combination thereof) is determined jointly by ALICE staff and the user based on the specific requirements of the planned experiment (e.g., technical complexity, number of samples), staff availability, and financial considerations.

ALICE operates on a first-come, first-served basis, regardless of the user's institutional affiliation or service mode. Exceptions for time-sensitive experiments (e.g., manuscript resubmissions) may be granted upon prior approval.

Internal Users (BOKU)

Service fees for internal users are determined according to the legal and financial framework of the project under which the work is conducted. Before starting any experiment, the Principal Investigator (PI) must provide:

- Project name
- Funding agency
- Account number for invoicing (6-digit cost center or 10-digit internal order number)

Based on this information, the PI will receive the applicable internal pricing details. ALICE reserves the right to **revise service fees** and pricing at any time, including for ongoing projects.

External Users (Non-BOKU)

External users receive a customized quotation after an initial project discussion. The offer, sent via email, must be confirmed in writing (typically by email) before any experimental work begins. Pricing updates issued by ALICE also apply to ongoing projects unless otherwise agreed upon in writing.

Instrument Usage

List of Instrumentation

Chromatography and Molar Mass Analysis

- SEC/MALS(488nm)/RI/FL in DMAc/LiCl
- SEC/MALS(785nm)/RI/DAD in DMSO/LiBr
- SEC/DAD Thermo Dionex UltiMate 3000 in aqueous-based solvents with optional MALS(685nm or 785nm) and/or RI detection
- Asymmetric flow field flow fractionation Waters|Wyatt AF4-Eclipse®/MALS(685nm)/RI
- Viscometer Waters|Wyatt Viscostar® for online and batch analysis
- Dynamic light scattering for online and batch R_h measurements Waters|Wyatt Dynapro® Nanostar® DLS
- Ultra-performance liquid chromatography Waters Aquity® APC/RI/PDA

Analytics (LC)

- 2D-LC Agilent Infinity II with two-dimensional HIC (hydrophobic interaction chromatography) and SEC separation
- Ion chromatography Metrohm
- UHPLC/PDA Waters Acquity® UPC^2 with supercritical CO_2 as the main eluent
- UPLC/PDA Waters Acquity® I-Class

Analytics (GC)

- GC-FID (1)
- GC-FID (2)
- GC-MS-ARC-FID (1) Headspace sampler
- GC-MS-ARC-FID (1) PAL-RTC sampler
- GC-MS-ARC-FID (2) Combi PAL
- GC-MS-ARC-FID (2) Pyrolysis

Analytics (MS)

- Quadrupole Time-of-Flight high-resolution mass spectrometer Waters qTof Xevo® G2

Analytics (spectroscopy)

- FTIR Brucker Invenio X
- Solid State NMR Brucker 400 MHz

Sample preparation & processing

- Accelerated Solvent Extractor Thermo Dionex™ ASE350
- Climate chamber
- Planetary ball mill Pulverisette 5

- Retsch Cryomill
- Supercritical carbon dioxide scCO$_2$ extraction unit
- Sieving machine

List of Services

Cellulose Analysis

- Molar mass distribution of celluloses by SEC/MALS/RI
- Carbonyl group profiles of celluloses ("CCOA method")
- Carboxyl group profiles of celluloses ("FDAM method")
- Oxidised reducing end groups quantification ("NED method")
- Fibre cross-sectional profiles of molar masses and functional groups
- Cellulose crystallinity and allomorphs by solid-state NMR spectroscopy
- Hemicellulose content and monosaccharide composition (Methanolysis)
- Hemicellulose content and monosaccharide composition (Total Hydrolysis)
- Degree of substitution in cellulose derivatives
- Ageing studies of cellulosic materials

Lignin Analysis

- Isolation and purification of technical lignins
    - Lignin isolation by precipitation
    - Ultrafiltration of lignin and adsorption methods
    - Lignin isolation by accelerated solvent extraction (ASE)
- Basic lignin characterization
    - Solids content by means of freeze-drying and/or vacuum oven drying
    - Extractives in lignin by GC-MS/FID or UPLC/qToF-MS
    - Thermal analysis techniques: thermogravimetry (TG), differential scanning calorimetry (DSC) and simultaneous thermal analysis (STA)
    - Lignin content in biorefinery process liquors: Klason lignin; Acetyl bromide lignin determination
- Functional group analysis of lignins
    - Methoxy, ethoxy and other alkoxy groups by headspace-isotope dilution GC-MS
    - Hydroxyl groups by ^{31}P NMR
    - Hydroxyl groups by ^{1}H NMR after peracetylation
    - Hydroxyl groups by methylation headspace isotope-dilution GC-MS
    - Sulfonic acid groups by means of conductometric titration
    - Sulfonic acid groups by elemental analysis
- Linkage analysis
    - Quantitative ^{13}C NMR
    - 2D NMR experiments for structure analysis of lignins
    - all relevant other NMR experiments: ^{1}H, COSY, TOCSY, ROESY, HSQC, HMBC, HSQC-TOCSY for liquid NMR; CP/MAS NMR for the solid state NMR
- IR-based Chemometrics for high-throughput function group analysis

- Molar mass distribution
    - Size exclusion chromatography SEC/RI in DMSO or NH_4OH with calibration
    - Size exclusion chromatography SEC/DAD/MALS(785nm)/RI in DMSO
    - Ultra-performance liquid chromatography APC/DAD/RI
    - Asymmetric Flow Field-Flow Fractionation AF4/MALS/RI
- Simultaneous molar mass and lignin content analysis in black liquors based on SEC/DAD in NH_4OH
- Molar mass dependent functional groups profiling by 2D-LC approach (HIC/SEC)
- Lignin nanoparticles (LNPs) analysis
    - Size distribution by AF4/DLS/RI
    - Offline dynamic light scattering analysis (DLS)
- Carbohydrate components in lignin
    - Methanolysis
    - Total Hydrolysis

Separation techniques and extractions

- Extraction of lipophilic compounds by $scCO_2$ or ASE
- Analysis of lipophilic extractives by hyphenated gas chromatography (GC-MS/FID)
- Separation and analysis of extractives by UPC2 / QToF-MS

Training

Use of ALICE instrumentation is permitted **only after successful training** by ALICE staff. **Important: Users are strictly prohibited from training other users.** Violation of this rule will result in loss of access for both the unauthorized trainer and trainee. To schedule training, please contact ALICE staff.

If a user has not used a specific instrument for over one year, a refresher or retraining session may be required. Training includes an introduction to instrument operation, data handling, and facility safety regulations.

Failure to complete training successfully or to comply with facility rules (especially safety-related) may result in suspension or permanent revocation of instrument access.

Instrument Booking and Cancellation

Most ALICE instruments are available to trained users during regular working hours (Mon–Fri, 9:00–17:00). Use outside these hours requires prior approval. BOKU-internal users may access certain instruments 24/7 only after completion of training that includes instruction on emergency procedures.

All instrument use and training must be booked via Stratocore (PPMS):

- **Internal users**: Log in with BOKU credentials to request an account.
- **External users**: Email your full name, contact details, PI's full name, and billing address to alice@boku.ac.at to set up a user account.

BOKU

Core Facility
Analysis of
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> Violation of booking rules or instrument use without prior booking or notification of ALICE staff will result in the immediate loss of access to the facility.

Cancellations within 24 hours of a scheduled session must be made in person, by phone, or via email. Unused bookings that are not cancelled in time will be fully charged. Instruments may not be booked for more than seven consecutive days, except in justified cases (e.g., manuscript revisions or short-term visits by external collaborators).

If instruments become unavailable (e.g., due to maintenance or repair), users will be informed as early as possible. ALICE reserves the right to cancel bookings at short notice for operational reasons.

Users must handle all equipment responsibly, follow all instructions from ALICE staff, and immediately report any issues.

> **In cases of user-induced damage (e.g., breaking an autosampler needle due to incorrect sample holder placement), the user may be held responsible for the cost of repairs.**
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> **Such cases will be evaluated individually.**

Sample Quality

All materials submitted to the Core Facility Analysis of Lignocellulosics (ALICE) must be of appropriate quality. Users are responsible for assessing **impurities and sample homogeneity** and accurately determining the sample concentration prior to analysis. ALICE does not accept responsibility for unsatisfactory results arising from poor sample quality or errors in the provided material.

If additional characterization is required to perform the requested service, this may incur additional costs. For guidance or assistance in assessing sample quality, users are encouraged to contact ALICE staff prior to submission.

Data Transfer and Storage

All ALICE computers are equipped with specific software for data analysis. Raw data generated on ALICE instruments are stored locally on the respective control PCs. It is the user's sole responsibility to transfer and securely store their data after completing experiments. A copy of the generated files must remain on the facility PC as evidence of usage (e.g., created sequences, log files, instrument output). Users are not permitted to delete any files from the control computers. **Accessing, viewing, or copying data belonging to other users or ALICE staff is strictly prohibited.**

Data can be transferred via network platforms such as drive.boku.ac.at, files.boku.ac.at, BOKU Box, or other secure services. If assistance with data transfer or access is required, please contact ALICE staff. Users may only use USB devices provided by ALICE for data transfer.

A user PC for data processing is available to ALICE users (Mon–Fri, 9:00–17:00) on a first-come, first-served basis. No reservation is required.

Publications

Users who publish results obtained using ALICE instrumentation or services must report the publication via PPMS.

If assistance is required for data presentation or methodology description, users are encouraged to contact ALICE staff. In accordance with good scientific practice, ALICE staff members who have made substantial intellectual contributions to a project or publication should be offered co-authorship.

Acknowledgment Guidelines

All publications, oral presentations, or posters that include data or services from ALICE must acknowledge the facility. Depending on the usage mode and staff involvement, one of the following templates should be used:

- **Trained user mode** (e.g., SEC): "The SEC/MALS equipment was kindly provided by the BOKU Core Facility Analysis of Lignocellulosics."
- **Full-service mode** (e.g., analysis conducted by ALICE staff): "We thank [staff name] for conducting the SEC/MALS experiments. The equipment was kindly provided by the BOKU Core Facility Analysis of Lignocellulosics."
- **Core Facility staff listed as co-author:** "The SEC/MALS equipment was kindly provided by the BOKU Core Facility Analysis of Lignocellulosics."

Contact

For inquiries, service requests, or training appointments, please contact: alice@boku.ac.at

Feedback and Evaluation

ALICE values user feedback and strives for continuous improvement. Users are encouraged to share their experiences, suggestions, or concerns directly with the ALICE team at any time.

Additionally, users will be invited annually to provide anonymous feedback through an online survey. Constructive input helps improve the facility's services and user experience.