Submission of Samples

1. ESI analyses

➔ Samples for ESI direct introduction are analyzed by accurate mass spectrometry only. Please note for sample submission:

- Please prepare your samples for ESI-Service in an HPLC vial without insert(!) (ND10). We recommend the vials from Neolab (also can be purchased from the stores):

Article	Description	Price
7-0728	neochrom® Screw caps PP, ND10 w. hole, natural rubber redorange / TEF transparent Thickness 1.3 mm, 60° shore A 100 pcs/pack	17.70 € (Brutto 21.06 €)
EC-1308	neochrom® Threaded vials ND10, clear glass, 1.5 mL, 100 pcs/pack	12.40 € (Brutto 14.76 €)

Picture from Neolab.

If required, the vials can be cleaned and re-used with a new septum on your own risk.

- Vials containing 0.1 µmol sample are submitted as solids to be dissolved in 1 mL methanol. Alternatively, you may submit vials containing 100 µM solutions in 1 mL methanol or acetonitrile (water till 80% acceptable). Higher concentrations do not only spoil your own analysis result by overloading, but what is more possibly affect the following samples with cross-contamination and finally damage the instrument. Your sample solution must be clear and particle-free and won't be analyzed else; if required, use a membrane filter for clearance of the solution. If you have only small amounts of your sample, please care for a minimal volume of 300 µL solution in the sample vial.
- If your sample cannot be dissolved in the solvents named above, you can use 50 µL dichloromethane, chloroform, DMSO, DMF or THF and top up with methanol or acetonitrile to 1 mL. Please USE THESE SOLVENTS ONLY IF INDISPENSIBLE; they damage our instruments on the long term. DO NOT USE CONCENTRATED ACIDS OR BASES. Please note any deviation from the recommended standard procedure on your sample sheet.
- Please name your sample according to this scheme: [name_AK]_[Your name]_[short sample ID no special signs].
- Download the most recent version of the sample sheet from our website and fill in the required information.
- Place your sample in the sample rack on the shelf in front of room 173a in the Technikum/Analytikum and deposit the sample sheet in the designated box.

2. EI analyses

- Samples are submitted as solids in small tubes or closed glass capillaries (can be obtained from us) together with the obligatory sample information sheet.
- High resolution analyses can be carried out up to *m/z* 900 only with a window of 50 *m/z*. This window is selected with respect to the expected molecular mass if not requested different. Until further notice, HR analysis will be carried out only if the prior low resolution full scan confirms the presence of the target peak.

Please provide hydrolysis-sensitive, air-sensitive or samples requiring cooling in a suitable sample vessel; we will prepare these samples before analysis.

For all GC-MS, LC-MS and MALDI-MS analyses please contact Dr. Birkemeyer: birkemeyer@chemie.uni-leipzig.de, tel.: 36092.

Submission of results

All analysis results will be deposited on the server ftp://spekserv.chemie.uni-leipzig.de/MS-Spektren in a folder named with the analysis date. The required pass word will be provided from your research group.

For all ESI-MS analyses, you can download the raw file containing the analysis file of your sample and evaluate with MestReNova. The pdf report contains a list of the signals ("peaks") detected in the sample and the corresponding noise-corrected m/z lists. You will find it as "Report.pdf" in your measurement file. In the front range (0-2 min) you should find your substance and in the range of 2.5-3.5 min the calibration peak. Since the report is generated automatically, all separated signals present in the sample are shown as "compounds".

For all EI-MS analyses, you will be provided with a result sheet in pdf format.

Please contact us immediately, at the latest within 3-4 days, if your spectrum does not show the expected peak with the corresponding tolerance (5 ppm for HR, 1 amu for LR), so that we can possibly still achieve a positive result in manual operation by adjusting the measurement parameters.

If you have any questions or suggestions, please send us a message (ms_service_fakchemie@uni-leipzig.de) or contact us directly (Susan Billig Tel.: 36077 or Claudia Birkemeyer Tel.: 36092).

Data evaluation

For data evaluation you can use the software MestReNova (Mestrelab) or ACD Labs Spectrus processor.

For MestReNova, download and install the software (http://research.uni-leipzig.de/nmr/MNOVA/); a one-year license is available from Dr. Icker: maik.icker@uni-leipzig.de. Installation and the instruction pages are only available in the campus net. The raw data files are .d files. You'll need to install the 32-bit version of Bruker CompassXtract library first and the provided license before you can open the files. In the document „Data evaluation with MestReNova" deposited on the server you'll find instructions how to open and evaluate the data files (also at the Homepage and the NMR homepage).

Furthermore, the Spectrus Processor from ACD Labs as an alternative evaluation has been available since December 2021; information on this is also available from Dr. Icker.

How to find my mass in MestReNova

The measurements are first displayed in MestReNova as TIC (Total Ion Chromatogram). The chromatogram in the report, however, is a so-called BPC (Base Peak Chromatogram), therefore different intensity curves (chromatogram) may result. Below is a short summary of how you can quickly find your signal with Mnova. (Please also read the instructions for sample measurement and data evaluation deposited on our server).

ESI MS analysis will mostly produce ions that correspond to adducts of the target compound with protons [M+1], sodium [M+23] or potassium ions [M+39] or other positive ions in your sample. If more than one of these ions or mixtures of them are attached to the target compound, it will have a morefold charge and you will be observing the resulting *m/z*.

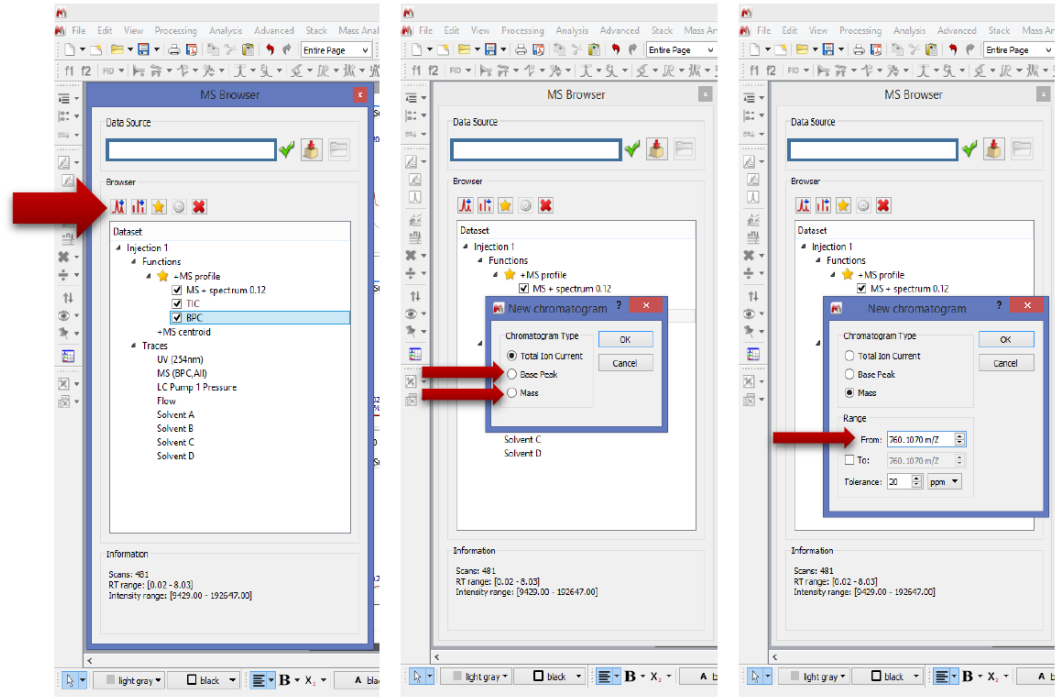
Acid H may be also replaced by alkali ions in solution: [M+2 Na-H]. In this case, no change in charge is observed.

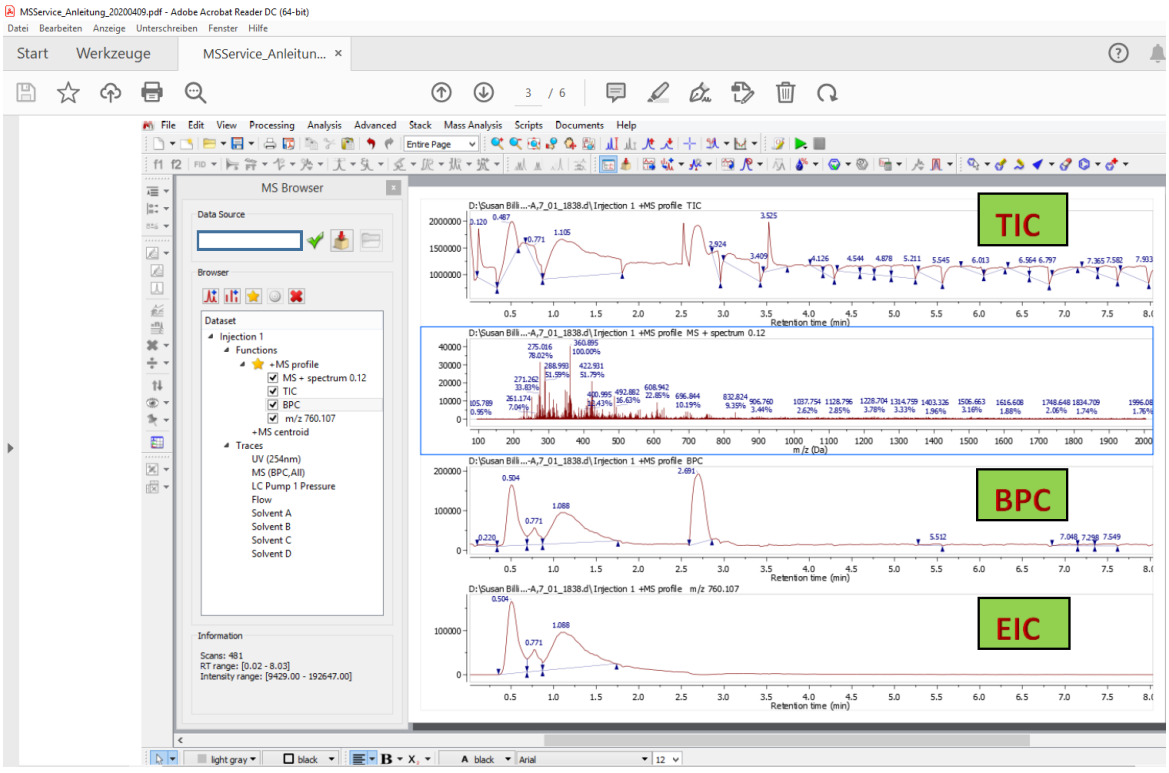
In the negative mode, deprotonation or other dissociation products of your target compound or adducts with negative ions in solution such as chloride can be observed.

In addition to that, you may have your target molecule associated with neutral solvent molecules or any other additives present in the sample solution. The isotopic pattern of your signal can give valuable hints on the identity of the adducts.

However, should you wish help with the interpretation of your spectra, please feel free to contact us: Ramona Oehme (36095), Susan Billig (36077), Claudia Birkemeyer (36092).

Click on "open new chromatogram" in the MS browser, make your selection (enter base peak or the mass of the ion you are looking for with the corresponding deviation) and confirm.





Documentation of the instrumental parameters

In the following, you'll find a documentation of the instrumental parameters you might need for documentation of your analyses. If you open the .d file and then the folder .m in the windows explorer, you'll find the „microTOFAcquisition.method" for the Bruker micrOTOF or „microTOFQImpactAcquisition.method" for the Bruker Impact II or the „EsquireAcquisition.method" for the Bruker Esquire 3000+.

1. ESI Analyses

Instrument: micrOTOF

ESI-TOF micrOTOF (Bruker Daltonik, Bremen, Germany), with Agilent 1100 HPLC (Agilent Technologies, isocratic pump and autosampler), and otofControl 3.4 and HyStar 3.2-LC/MS. (accurate analyses)

Instrumental parameters:

		Manuel injection	**Autosampler**
Source	Nebulizer	0.3 bar (4.4 psi)	1.8 bar (26.1 psi)
	Dry Gas Flow	4 L/min	6 L/min
	Dry Gas Temperature	200°C	220°C
Injection	Syringe pump	200 µL/h	---
	Injection volume	---	5-25 µL
	Flow	---	0.1 mL/min
	Eluent	---	ACN 0.1% FA
Calibrant		Tuning Mix Low or 50 µM Arginine in 80% ACN	

Analyzer:

	small *m/z*	**middle *m/z***	**large *m/z***	**small *m/z***	**middle *m/z***	**large *m/z***
Mode	**positive**			**negative**		
Mass Range	50-1500 *m/z*	100-2000 *m/z*	1000-3000 *m/z*	50-1500 *m/z*	100-2000 *m/z*	1000-3000 *m/z*
Capillary Exit	100 V	150 V	200 V	-70 V	-100 V	-180 V
Skimmer 1	50 V	50 V	50 V	-50 v	-50 V	-50 V
Hexapole 1	23 V	23 V	23 V	-23 V	-23 V	-23 V
Hexapole RF	70 Vpp	350 Vpp	800 Vpp	85 Vpp	200 Vpp	800 Vpp
Skimmer 2	23V	23 V	23 V	-25 V	-25,1 V	-25 V
Lens 1 Transfer	70 µs	88 µs	88 µs	75 µs	70 µs	88 µs
Lens 1 Pre pulse Storage	8 µs	10 µs	11 µs	8 µs	10 µs	15 µs

Instrument: Impact II

ESI-qTOF Impact II (Bruker Daltonik) with Dionex Ultimate 3000 UHPLC (ThermoFischer) and otofControl 4.0 and HyStar 3.2-LC/MS. (accurate analyses)

Instrumental parameters:

		Manuel injection	**Autosampler**
Source	Nebulizer	0.3 bar (4.4 psi)	1.8 bar (26.1 psi)
	Dry Gas Flow	4 L/min	8 L/min
	Dry Gas Temperature	200°C	220°C
Injection	Syringe pump	200 µL/h	---
	Injection volume	---	1-25 µL
	Flow	---	0.1 mL/min
	Eluent	---	ACN 0.1% FA
Calibrant		Tuning Mix Low or 100 µM Arginine in 80% ACN	

Analyzer:

	small *m/z*	**large *m/z***	**small *m/z***	**large *m/z***
Mode	**positiv**		**negativ**	
Mass Range	50-1300 *m/z*	700-3000 *m/z*	50-1300 *m/z*	700-3000 *m/z*
Funnel 1 RF	150 Vpp	400 Vpp	150 Vpp	300 Vpp
Funnel 2 RF	200 Vpp	600 Vpp	200 Vpp	400 Vpp
Hexapole RF	150 Vpp	800 Vpp	50 Vpp	600 Vpp
Ion Energy	4 eV	4 eV	4 eV	4 eV
Low Mass	100 *m/z*	400 *m/z*	100 *m/z*	400 *m/z*
Collision Energy	7 eV	10 eV	7 eV	7 eV
Collision RF	300 Vpp	1600 Vpp	800 Vpp	1600 Vpp
Transfer Time	100 µs	160 µs	100 µs	100 µs
Pre Pulse Storage	5 µs	15 µs	5 µs	5 µs

Instrument: Esquire 3000+

ESI-Ion trap Esquire 3000+ (Bruker Daltonik GmbH) with 1100 HPLC (Cohesive Technologies, Agilent) and esquireControl 5.3 and Agilent ChemStation Rev.B.01.03. (low resolution analyses)

Instrumental parameters:

		Manuel injection	**Autosampler**
Source	Nebulizer	11 psi	40 psi
	Dry Gas Flow	5 L/min	10 L/min
	Dry Gas Temperature	300°C	300°C
Injection	Syringe pump	200 µL/h	---
	Injection volume	---	25 µL
	Flow	---	0.1 mL/min
	Eluent	---	ACN 0.1% FA
Calibrant		Tuning Mix	

Analyzer:

	small *m/z*	**middle *m/z***	**middle *m/z***	**large *m/z***
Mode	**positive and negative**			
Mass Range	50-750 *m/z*	100-1500 *m/z*	500-1500 *m/z*	700-3000 *m/z*
Target Masse	200 *m/z*	500 *m/z*	1000 *m/z*	2000 *m/z*
Optimize	Wide	Wide	Wide	Wide

2. EI analyses

Instrument: MAT 8230

EI double-focusing B-E sector field (Finnigen MAT) with MASPEC II.

Ionization source:

Temperature:	200°C
Vacuum:	2 x 10^{-7} mbar
Electron enery:	70 eV
Emission:	0.5 mA
Acceleration Voltage:	3 kV

Analyzer:

Scan rate:	5 sec/decade (20 sec for HR)
Scan range:	0-2100 amu
Interscan delay	1 sec

Instrument: Finnigen MAT 95XP

EI double-focusing B-E sector field (Firma Thermo Electron Corporation) with Xcalibur ™ 1.4.

Ionization source:

Temperature:	240°C
Vacuum:	9 x 10^{-6} mbar
Electron energy:	70 eV
Emission:	1 mA
Acceleration voltage:	3.5 kV

Analyzer:

Scan rate:	0.5 sec/decade (5 sec for HR)
Scan range:	See result file
Interscan delay	0.2 sec

3. LC- and GC-MS, MALDI

Parameter may vary considerably; you will receive a separate description for the parameters along with your results.

For all further questions, please contact us: Ramona Oehme for EI and ESI direct introduction (tel. 36095), Susan Billig for hyphenated analyses and server/software issues (tel. 36077) or Frau Birkemeyer for data interpretation and on organizational issues (senior executive, tel. 36092).