

GC-MS Service:

Please note:

Before submitting samples for routine GC-EI-MS analysis, a GC run of your sample is required.

Please download/printout the sample submission form from <http://www.oc.tu-bs.de/massenantrag.doc> (filename: Massenantrag.doc) and fill in as much information as possible (in particular: solvent, temperature program, chemical formulas and masses, educts, reagents and expected products). An example of a filled in sample submission form is attached to the end of this document.

Here are some more general rules and hints to speed up sample processing:

- clear samples (no floaties inside)
- use dry organic solvents with **low** evaporation points like acetone, dichloromethane etc.
- Samples need to be free of mineral acids (HCl, H₂SO₄ etc.) or bases (NaOH etc.)

- Samples need to be salt free

Please perform a micro scale work-up for crude reaction mixtures, which includes extraction of the reaction mixture with water and then drying the organic phase with Na₂SO₄ or MgSO₄ prior to submission)

- **Concentration of submitted samples: ca. 100 ng/μl (= 0.1mg/ml)**
- **Please label your sample vials with a unique code using water proof pen or adhesive label**

Please run a GC-FID from your sample before submitting it for GC-MS analysis using following (or similar) conditions:

1μl injection; split ratio 10:1, injection port temperature 250-280°C, detector temperature 300°C. Also keep in mind to run the temperature program at least up to 300°C and hold it there for several minutes (this is true for DB1/DB5 and analog GC columns. If you use a different column please follow the instructions of the manufacturer for the temperature limits).

The GC run on your instrument is mandatory before you can submit your sample for GC-MS. This procedure helps to minimize down times of the GC-MS instrument.

To estimate the concentration in your sample or the performance of your GC device in general, please check it by comparing it to our **standard reference mix**.

The **standard reference mix** contains 100 ng/µl of each, decane, methylpalmitate, eicosane and octacosane.

If you do not have this standard mix already in your lab you can get an aliquot from the MS facilities (Room 053).

Figure 1 shows the GC separation of the standard mix. If our instrument is in good condition, you should get a similar chromatogram.

This will help to estimate the concentration in your sample by comparing the integrated peak **areas** of your compounds with those areas that you get with the standard mix on your GC.

We recommend, using the standard reference mix to control the performance of your GC from time to time, as well, by comparing the actual performance with earlier runs.

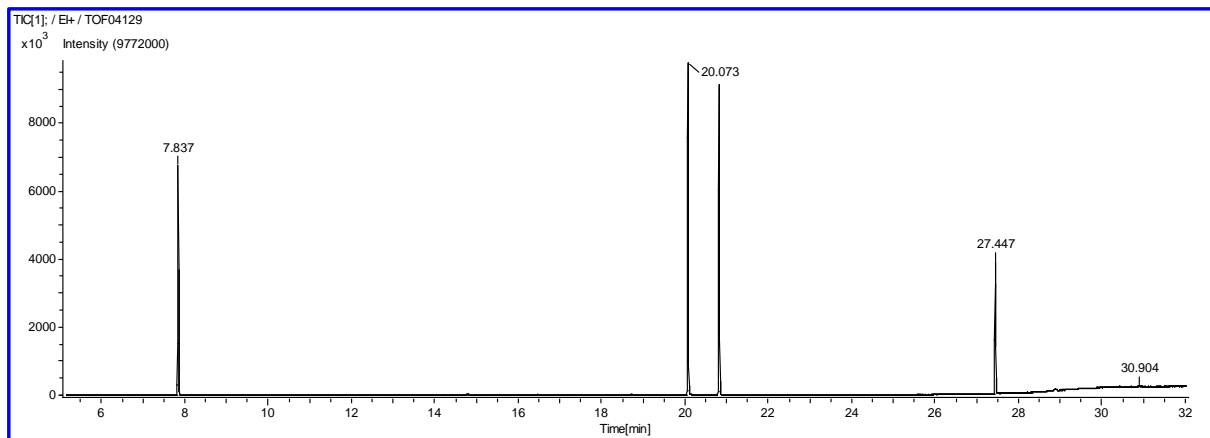


Fig. 1: Exemplified GC separation of the standard reference mix.
Conditions: Split 1: 10, Inj. 270°C, Temp.prog.: 50 °C (3min) - 10°C/min - 310°C (3 min),
column: ZB5MS (30m x 0.25 mm ID x 0.25 µm ft)

If you have any questions concerning GC-MS feel free to contact me.

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Tel.: 5385

V. 10.08.2016

A N T R A G zum Massenspektrum-Nr.

Massenspektrometer Finnigan MAT 100-SS

Finnigan MAT 45-5

EXAMPLE

Arbeitsgruppe Dr. Q

Hersteller der Substanz Miss Moneypenny Tel. 007

Substanzcode Reaktion 3

Summenformel C₁₁H₉O₅N₁ Massenzahl 235

Präzisionsmasse, theor. 235.04807

Präzisionsmasse, exp. _____ ± ppm Res.: _____ (10% Tal-Def.)

Schmelzpunkt/Zersetzungspunkt 132 °C

Siedepunkt/Sublimationspunkt n.d. °C/mm Pa

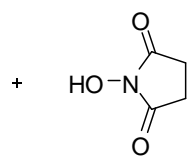
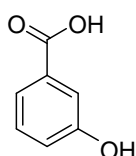
Empfindlichkeit: Wärme/Licht/H₂O/O₂

FAB-Matrix _____

Verunreinigungen Phthalate aus Lösungsmittel

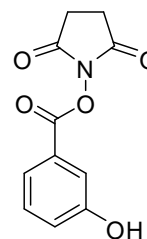
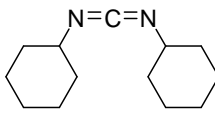
Reaktionsweg und Strukturvorschlag

Chemical Formula: C₇H₆O₃
Exact Mass: 138.03169



Chemical Formula: C₄H₅NO₃
Exact Mass: 115.02694

Chemical Formula: C₁₃H₂₂N₂
Exact Mass: 206.17830



Chemical Formula: C₁₁H₉NO₅
Exact Mass: 235.04807

Solvent: CH₂Cl₂

Temp. Prog.: 70°C-3min-10°C/min-300°C-3min

Please send the data in file format to: moneypenny@mi6.uk

CI/DCI	FAB	GC-MS	EI
pos.	pos.	▲	RES
neg.	neg.		

Matrix

NH₃

Endtemp.