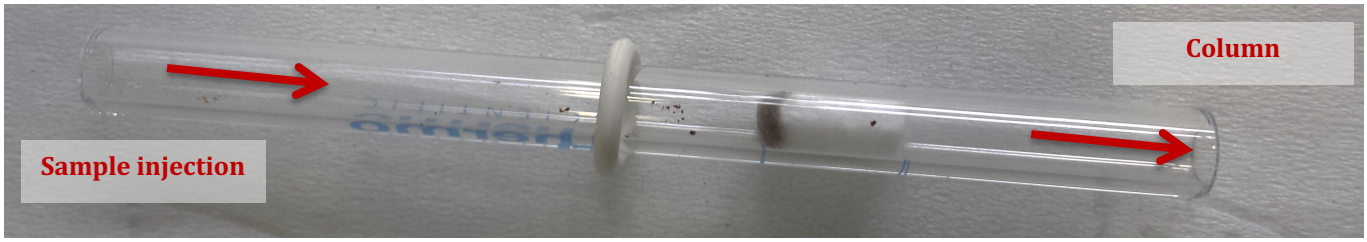
Instruction Manual ISQ GC-MS (Thermo)

SAMPLE PREPARATION

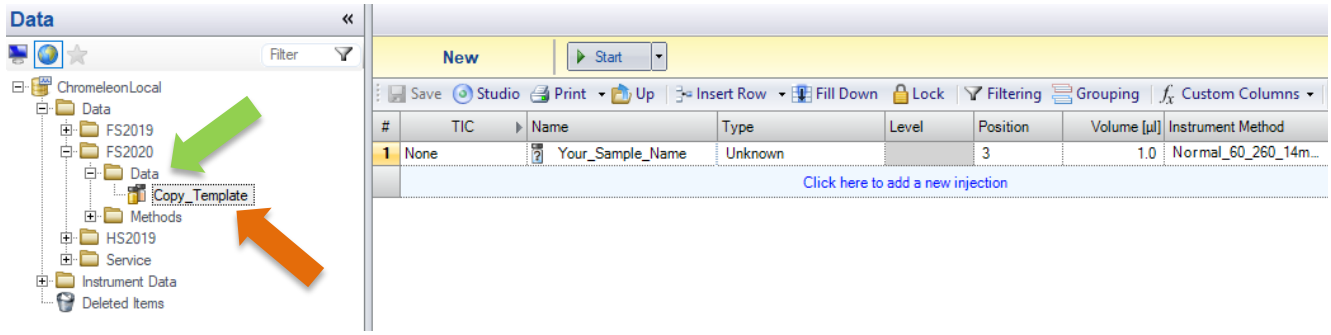
1. Dissolve the compound **(< 100 µg mL^{-1})** in (preferred) diethyl ether, ethyl acetate, and hexane or (workable) acetonitrile, dichloromethane, and methanol.
   Attention, **solvent reactivity** (e.g. amines in presence of acetone) must be considered.
2. Your sample must be **completely dissolved** and the solution must be **monophasic**! Short **filtration** over silica gel in a glass pipette required if the solution contains salts or precipitate.
3. Fill the 1.8 mL vial with **at least 0.5 mL sample solution** & seal the vial with the crimper.
4. Label your vial.
5. Place the vial in the auto-sampler carousel **according to the vial position in the sample list**.

In general, GC-MS is only suitable for volatile, apolar substances. If you inject nonvolatile compounds the liner will get dirty (black ring) and doesno longer provide an inert enviroment.

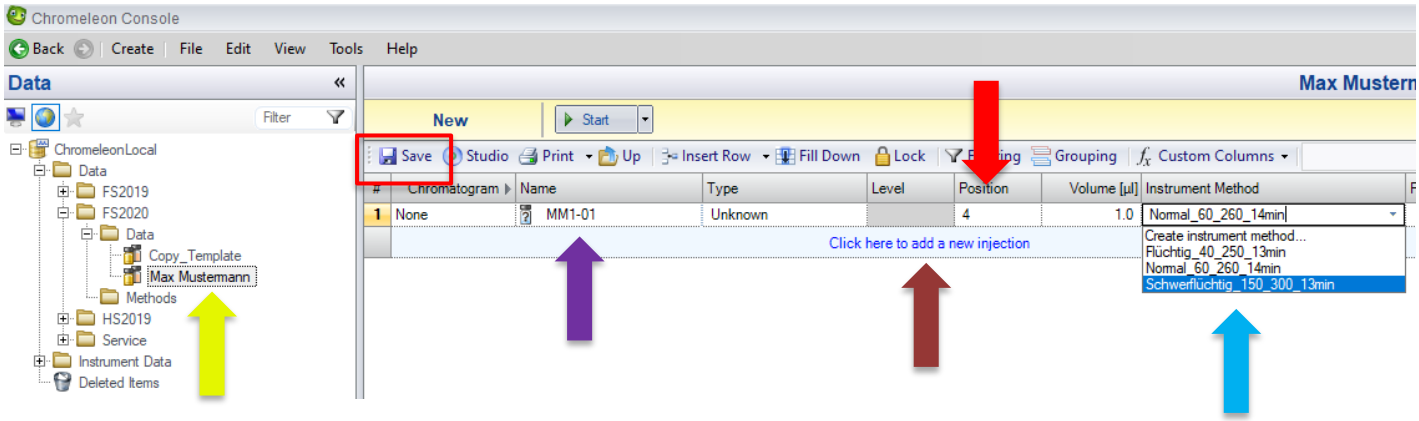


MEASURING SAMPLE(S)

1. Open "***Chromeleon 7***" with the link on the desktop (if not already open).
2. If you do not have a folder yet, click right ***"Copy_Template"*** from *"**Data**"* (orange arrow) and press copy. (If you do have a folder, proceed with step 5.)



3. Right click *"**Data**"* (green arrow) and press paste.
4. Rename "Copy_Template_Copy" to your name "studentxyz". (yellow arrow)



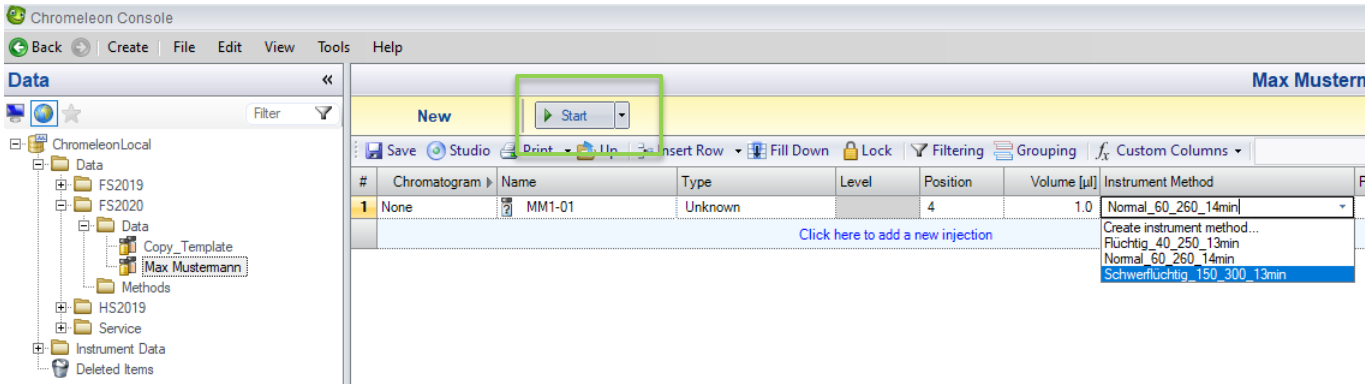
L. Bigler, Tel 54286, laurent.bigler@chem.uzh.ch

5. Go into your Data folder and set up your sample for injection:
    a. ***File Name***: your initials + substance code (e.g. sb1-25). It should be a unique name. (violet arrow)
    b. ***Instrument Method:*** (blue arrow)
        '*Fluechtig_40_250_13min*'
        '*Normal_60_300_16min*'
        '*Schwerfluechtig_150_300_13min*'
    c. ***Vial* position:** Double-check that your vial is in the indicated position!!! See number at autosampler! (red arrow)
    d. ***Injection Volume***: always 1 µL
    e. ***Processing method***: always Qualitative, do not forget to choose it!!!

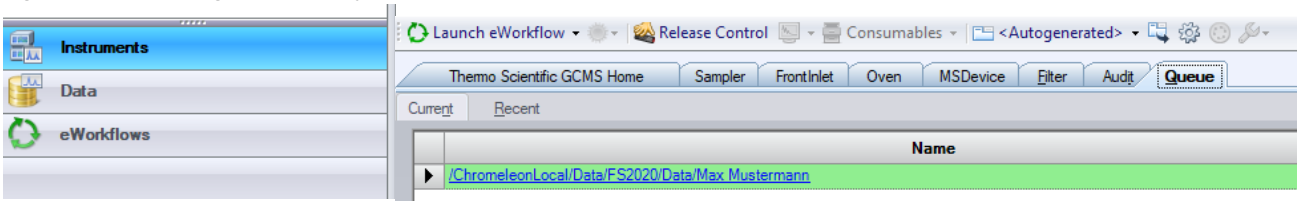
6. To add a new sample, click ***"Click here add a new injection"*** (brown arrow) and adjust the sample information as in step 5.

7. **Important**! Press ***"Save"***, when you are finished with adjusting your sequence! (red box)

8. Select the sample row(s) to analyze and press "***Start***" / "***Resume***". (green box)



9. Your sample will be attached to the measurement queue. To check how many measurements are in front of your sample, click on ***"Instruments"*** in the bottom left corner and choose ***"Queue"*** on the top right corner. To go back to your sample sequence, click on ***"Data"***.



10. Wait until your sample is measured.

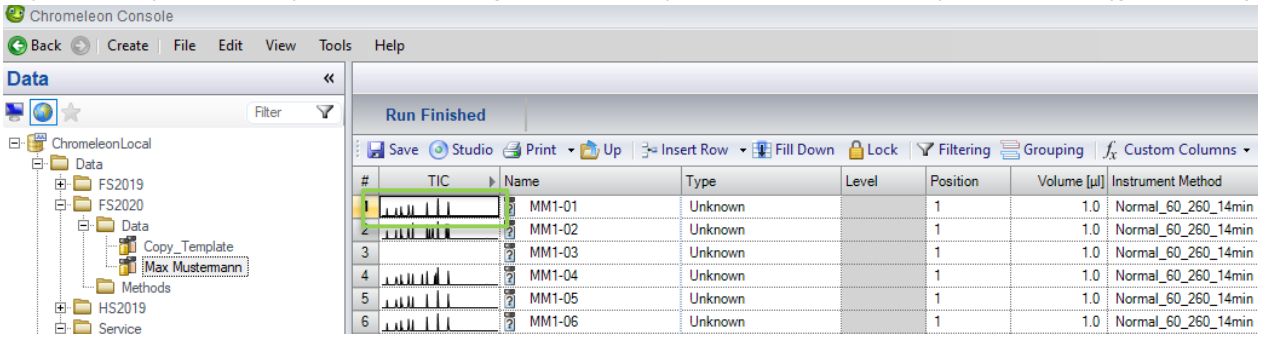
11. If any problems or errors occur during the measurement, please inform the teaching assistant.

L. Bigler, Tel 54286, laurent.bigler@chem.uzh.ch

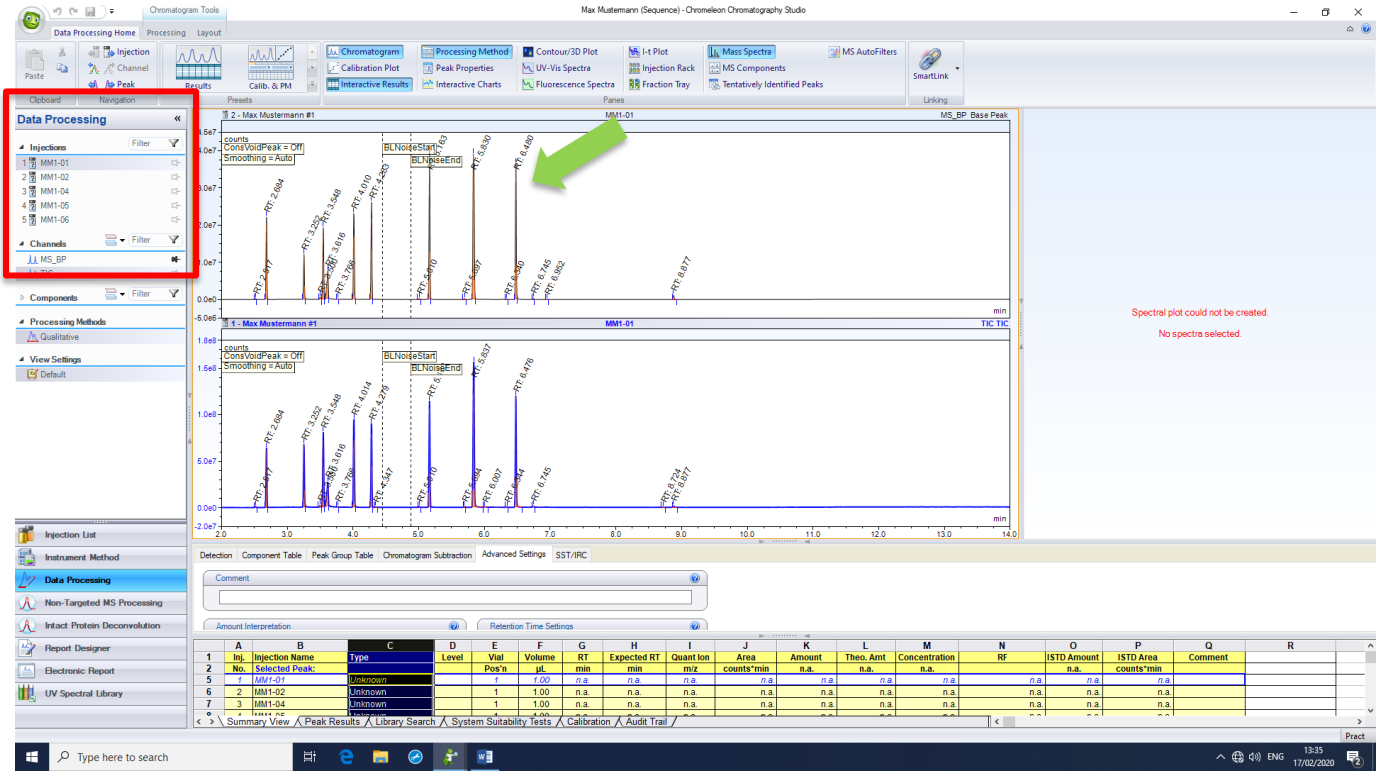
DATA PROCESSING

1. Once the analysis is performed, go back to your data folder and find your measured sample.

2. Open your analysis data by double clicking on the newly appeared **"TIC"** of your sample (green box).



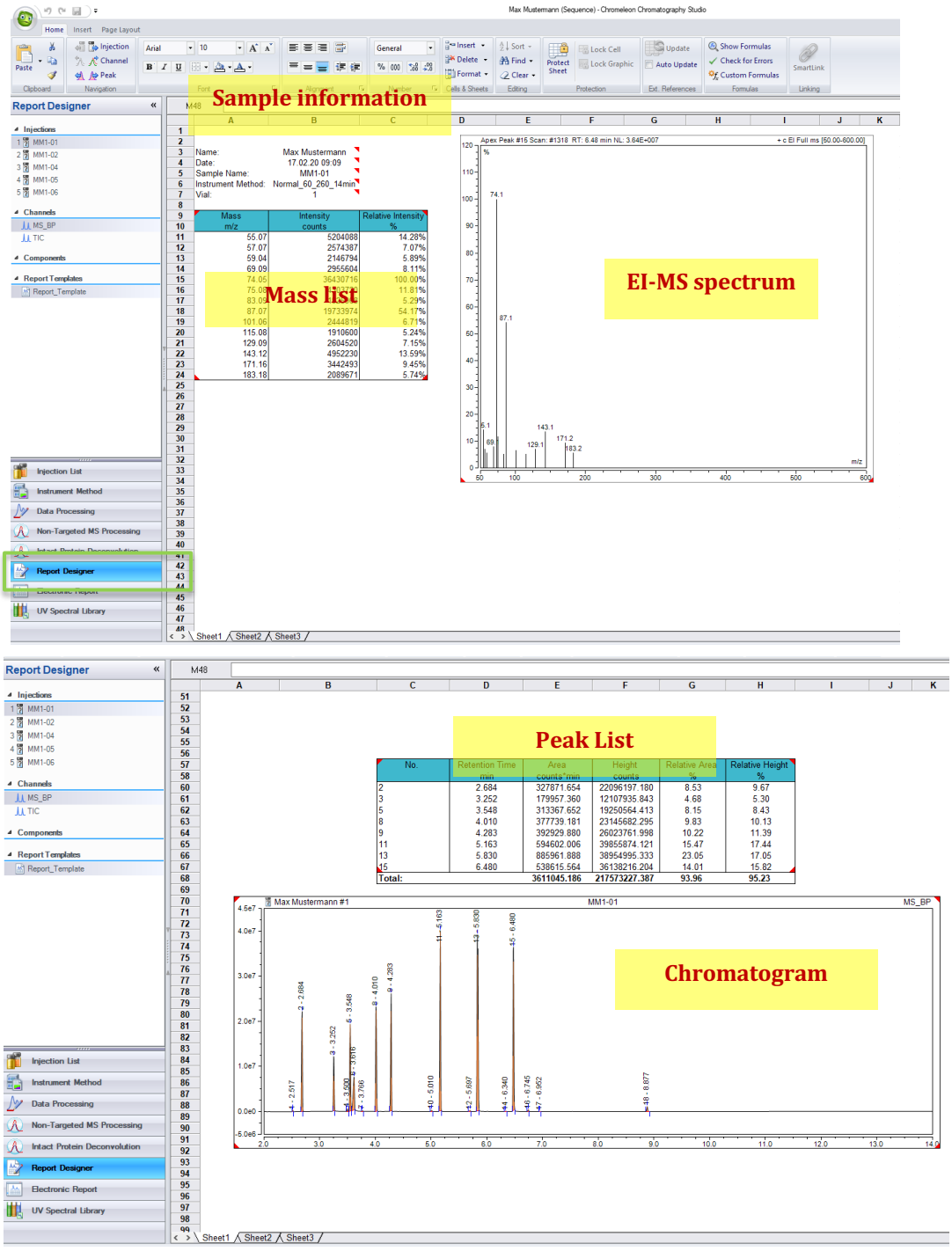
3. A window similar to this should open:



4. Select the sample you want to process. (red window)

5. Double-click on the peak of your compound in the **UPPER** window (basepeak chromatogram) to get a mass spectrum of the peak. (green arrow)

6. Click "***Report designer***". (green box) The automatically opened default layout "Report Template" includes all sample information, an EI-MS spectrum and its corresponding mass list as well as a chromatogram with an integrated peak list. (detection threshold >5%)



7. Create a pdf-file. In the print preview window, you can check whether the scaling is good (***Chromeleon-Icon → Print → Print preview)***. Depending on your mass / peak list, you may have to move your tables/pictures. Save your pdf-files under 'C:\Thermo\data\Praktikum_FS2021\studentXYZ' in your individual folder by exporting them as pdf under ***Chromeleon-Icon → Export***. The pdf-files can be finally transferred to a memory stick.

Technical Support

1. Teaching assistant responsible for GC/MS (Joe Woods or Leonardo Palaferri);
2. Teaching assistant of the day;
3. MS Service (lab 12E70 / Tel 044 635 42 85)