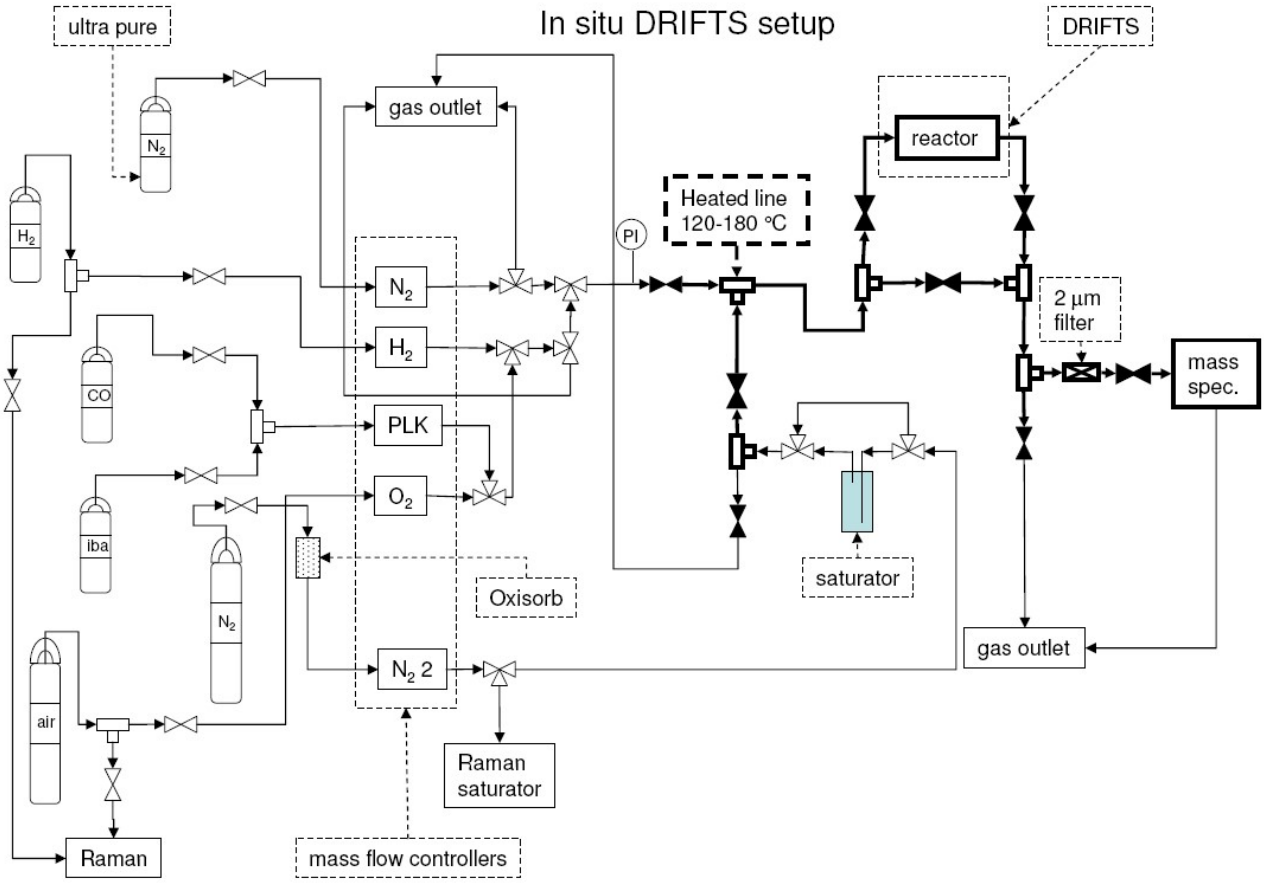
The Use of DRIFTS-MS Equipment

Updated 6.7.2021, Tiia Viinikainen

Important to all users: These instructions are made <u>to remind you</u> to carry out all the steps in the experiment, NOT to teach you how to use the equipment. The responsible person is always needed to first teach you how to use DRIFTS-MS.

1 Flow Chart


In situ DRIFTS setup
ultra pure
gas outlet
DRIFTS
reactor
Heated line
120-180 °C
PI
2 µm
filter
mass
spec.
N_2
H_2
CO
iba
air
PLK
O_2
N_2 2
Oxisorb
saturator
gas outlet
Raman
saturator
Raman
mass flow controllers

2 Alignment

Alignment is done once a month OR before starting a set of experiments, if DRIFTS has not been used for a while. **The alignment is done by the responsible person of the equipment. Please contact the responsible person in advance to carry out the alignment before starting your experiments.**

- Disconnect GAS IN and GAS OUT –lines from the external connections (no need to disconnect the cooling water lines) or leave the lines connected and be extra careful
- Close the gas line valves to prevent the collection of moisture into the pipelines
- Remove the sample array from the chamber:
    - Disconnect the white air tube coming to the back of the array by pressing the metal part on the left side of the tube
    - Lift the array holding on to the black base plate at the front and the back (mirror doors closed). If lines are connected, be extra careful in this.
- Close the chamber and let it flush for one to two hours (you can also leave the chamber to get flushed over night)
- Open OMNIC software
- Select: Collect -> Experiment Setup... -> Bench: change Autogain -> Gain 1
    - Write down the maximum peak value into the laboratory notebook (on the computer table)
- Select: Diagnostic -> Align…
- The alignment can be done multiple times, if needed
- Wait until alignment is complete and press OK
    - Write down the new maximum value
- Run diagnostics Collect -> Advanced diagnostics
    - Do Performance test: Signal to Noise, write down the results
    - Check the working of the components on their own tabs, write down the values of voltage etc.
- Set the sample array back into the chamber (mirror doors closed)
- Connect the white air tube back to the back of the array. When you hear the click you know that the tube is in place.
- Let the chamber flush before starting measurements
- Change the gain back to Autogain (Collect -> Experiment Setup -> Bench -> Gain 1 -> Autogain

3 Recording the Background

- Set the mirror in the sample holder with clean plastic gloves. The mirror should be taken out of its protective pocket with your fingers so that they only touch the sides of the mirror. The mirror is stored in the black suitcase (on the computer table), in a box with text: **7004-014 Alignmt mirror**. When the mirror is in place, close the mirror doors.
- Open OMNIC software
- Select: Collect -> Experiment Setup -> Collect.
  Change the following entries:
  - No of Scans 200
  - Final Format: % Reflectance
  - Save automatically

  Background Handling:
  - Collect background after 100 minutes
- Adjusting the height of the sample:
  - Select: (Collect -> Experiment Setup ->) Bench
  - Adjust the height by the turning the screw in the front of the IR cell. (When raising the screw, you need to help the spring to rise with your fingers)
  - If the location of the peak isn't constant or the spectrum looks otherwise peculiar, select: (Collect -> Experiment Setup ->) Diagnostic: Reset Bench. If this does not help, try to Check Desiccant: (Collect -> Experiment Setup -> Diagnostics). Press OK when the check is complete.
  - When you've found the maximum, write the value down into the laboratory notebook (on the computer table) and press OK
- Place the reactor dome in the sample array and fasten it with four screws – one in every other hole. Tighten the opposing screws in turn.
- Close the mirror doors and adjust the sample height as before and record the maximum value
- Close the chamber
  - If the maximum value changes at this point, the lines are not properly placed. Ask the responsible person of the equipment for help.
- Make sure that GAS IN and GAS OUT lines are connected and the valves are open.
  - There is also a bypass valve below the valves right before and after the reactor – make sure it's closed
- Open the argon bottle and turn on the mass flow controller
- Set argon flow at 20 ml/min and after a short while (so that the flow stabilizes) turn the flow into to the spectrometer (OUT -> IN). Observe the pressure gauge to prevent pressure rise in the equipment due to blockages
- Increase the argon flow to 45.4 ml/min (equals to 70 ml/min)
- Allow the equipment to flush for at least 30 minutes
- Decrease the argon flow to 36 ml/min (equals to 50 ml/min) before recording spectra
- Record the background spectrum:
  - Click the Col Bkg key in OMINC
  - Write down the peak value during the recording
  - Choose: Yes to add the spectrum to the window
  - Compare the spectrum to previous ones
    - If the shape is strange check the reactor windows for impurities and clean with a cotton stick slightly moisturised with ethanol (Aa)

    - Save the background with the name Bkg"yymmdd".spa (e.g. Bkg090121.spa) in MyDocuments/omnic/Backgrounds folder
- Decrease the argon flow to zero and turn the flow away from the spectrometer (IN -> OUT)
- Open the chamber and remove the reactor dome and the mirror. Touch the mirror only on the sides with clean plastic gloves. Place the mirror back to its box in the black suitcase
- Replace the reactor dome O-rings (two small ones in the cooling line) and one larger O-ring between the dome top and the sample holder base). New ones from the black case)
- Make the following changes before measuring sample spectra:
    - Select: Collect -> Experiment Setup -> Collect
      Change the following entries:
        - No of Scans 100
        - (Final Format: % Reflectance)
        - Background Handling:
        - Use specified background file:
          Select Browse… and find the background you have just saved (They should be located at C:\My Documents\Omnic\Spectra\Bkg"date".SPA)
        - Press OK
    - Open a new window: Window -> New Window (Ctrl + N)

Keep the chamber closed as much as possible to prevent moisture collecting in the equipment.

4 Loading the Sample

- Write details of your sample into the laboratory notebook (on the computer table)
- Use a lab coat, protective gloves and a respirator when you handle a sample. Samples may be harmful.
- Load the powdered sample into the sample holder using a spattle
- Level the surface of the sample using the flat end of the spattle
    - Avoid knocking on the sample holder and pressing on the sample
    - If some of the sample goes outside of the reactor, wipe it carefully away into a brush that you clean on a piece of hand towel

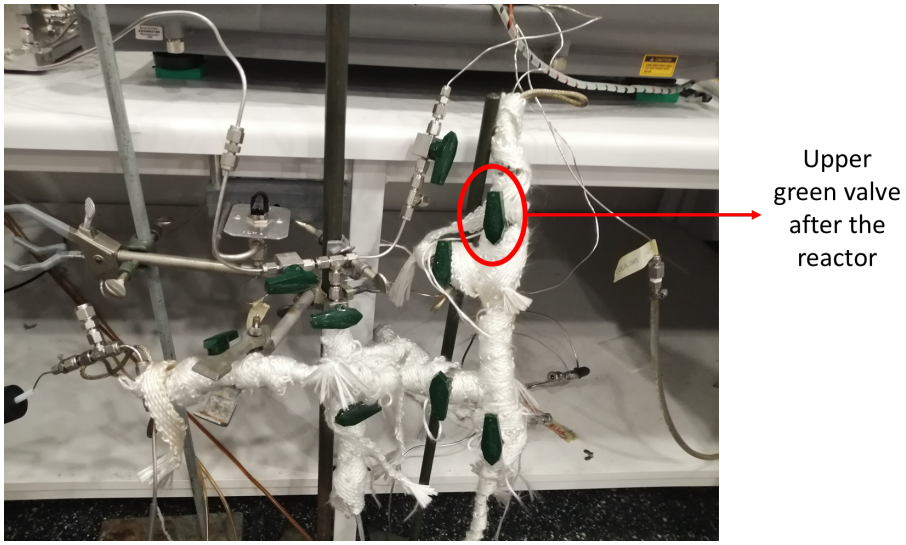
  (Take off the gloves you used while handling the sample)
- Close the mirror doors
- Adjust the height of the sample:
    - Select: (Collect -> Experiment Setup ->) Bench
    - Adjust the height by the turning the screw in the front of the cell. (When raising the screw, you need to help the spring to rise with your fingers)
    - When you've found the maximum write the value down into the laboratory notebook (on the computer table) and press OK
- Place the reactor dome into the sample array and tighten all eight screws – opposing ones in turn – to properly seal the reactor
- Close the mirror doors
- Adjust the sample height and write down the peak value as above
- Close the chamber
- If you do the actual sample measurements in the following day, don't close the program. Only turn off the screen (this way all the changes you've made are preserved)
- Test the seals of the cooling water spiral connections to the reactor dome

- Turn the cooling water line on (on the wall on the right side of computers) and turn the water flow in to the cooling spiral (turn cooling line valve below the equipment VEI/ILMA to VESI slowly), you can use your fingers to check for droplets
- After a few minutes observe if droplets of water have formed at the base of the reactor dome where the cooling spiral is connected to the base of the sample holder
- If droplets have formed:
  - Empty the cooling spiral
  - Open the reactor dome
  - Dry out the water
  - Replace the reactor dome and tighten the screws with more care before repeating the test
- If water is not leaking, turn the cooling line valve (VESI/ILMA) to the middle position (pointing left), open the pressurized air line above the equipment (blue valve) and turn the cooling line valve from VESI to ILMA. Let the line dry for 5 minutes. Then turn VESI/ILMA to middle position (pointing left). Close the pressurized air line above the equipment (blue valve) and close the cooling water line (on the wall on the right side of computers)

- Perform a pressure test

5 Pressure Test

- Set the argon flow to 10 ml/min
- Turn the flow into the reactor
- Check the pressure gauge to make sure there are no blockages
- Close the **upper green valve** after the reactor (see picture below)



- When the pressure has risen 0,1 bar, turn the gas flow out of the reactor
- Decrease the argon flow to zero
- Turn off the mass flow controller unit
- Close the argon bottle and reducer valve (leave the middle valve in its position, that controllers the outgoing pressure!)
- Check at least after 15 min that the pressure has stayed in the reactor
- The minimum time for a pressure test is approx. 0.5-1 hours
- Release the pressure by opening the **upper green valve** after the reactor (see the picture above)

If you do the actual experiment on the following day, do not close OMNIC software (only turn the screen of the computer off). This way all the changes you have done, are preserved.

Plan the timing of your experiment in detail. The only way to make comparable experiments is to use the same amount of time in each step as was done in previous experiment. Use the attached Excel-file (DRIFTS_example_experiments.xlsx) to plan your timing. If needed, the responsible person of the equipment can help to plan the timetable for your experiment.

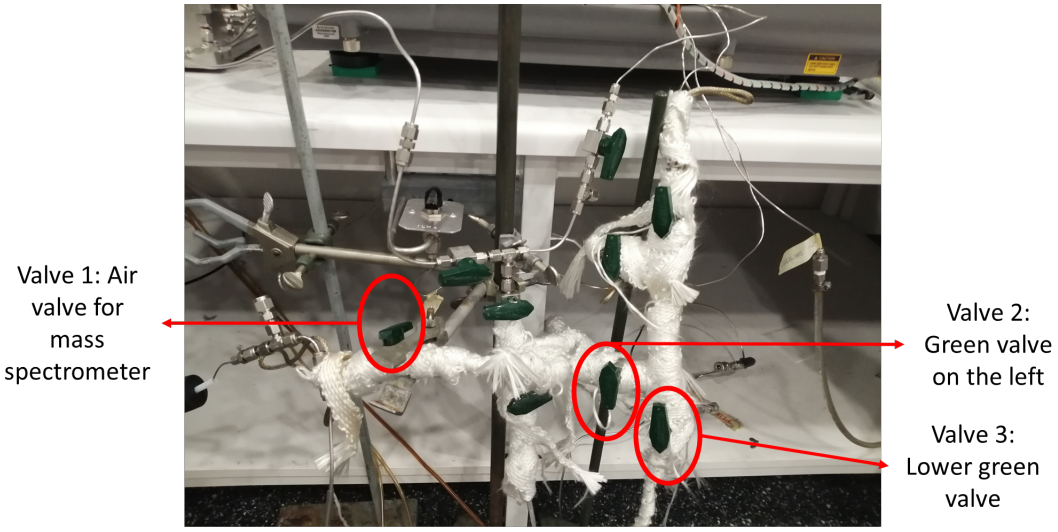
6 Starting the experiment

- Open the argon bottle (and air bottle if needed) and the valve in the reducer (make also sure that the valves in the lines are open), NEVER adjust the middle valve in the reducer (defines the outgoing pressure)
- Turn on the cooling water:
    - Open the water valve fully (on the wall on the right side of the computers)
    - Turn the cooling line to VESI
- Turn on the mass flow controller unit
- Turn on the heating unit (from the back) and switch HEATER from DISABLE to ENABLE
- Make sure that the SV (set value) temperature is 30 °C in the heater
- Set the gas flow according to your calcination/heating requirements
    - Calcination: Ar = 18 ml/min (= 25 ml/min) and O2 = 25 ml/min (-> 10% O_2/inert)
    - Heating in inert: Ar = 36 ml/min (= 50 ml/min)
    - Turn the flow(s) into the reactor (OUT -> IN), first inert and then O_2 (if used)
    - Check the pressure gauge to make sure there are no blockages
- Record the spectrum at 30 °C (100 scans) with the name "catalyst"_"gas"_"temp"u by selecting "Col Smp" from the buttons on the top row
    - "catalyst" is the name of your sample
    - "gas" is the gas or gas mixture used, e.g. Ar or air
    - "temp" is the temperature, e.g. 030
    - u means up (other symbols: d down, f flush etc.)
    - Example: Rh/ZrO2_air030u
    - The connection between the computer and spectrometer might sometimes be off (there is no blinking light in the spectrometer at "Scan"), some tricks to help with this
        - If you see that the light is not blinking select: Collect -> Experiment Setup, when the light starts to blink, press OK
        - If the spectrum measurement says "Timed-out", close the measurement window (the lower X in the top right corner) and start the measurement again
        - If the Background is lost (there is no "Bkg Age" text in the bottom of the window and/or the y axis shows "Sigle Beam", stop the measurement and try again
    - Write down each spectrum with its measuring time into the laboratory notebook (on the computer table). Write down also the maximum peak value during recording each spectrum (shown below the spectrum while recording).

Starting mass spectrometer

- Start mass spectrum around 45 minutes before it is needed (at least 30 min is needed to stabilize the signals)
- If mass spectrometer is completely shut down (no lights in the panel)

    - Turn power on from the back of the mass spectrometer -> POWER button becomes green
    - Start the pumps by pressing PUMP button -> READY button flashes green until turbo pump has reached its speed, wait until READY button is green
- Turn on the heater in the spectrometer (120,2 °C) (the HEATER button)
- When the set temperature has been reached, turn the flow coming from the reactor to the mass spectrometer:
    - Close the **air valve** (valve 1 in the picture below) for mass spectrometer
    - Open the **green valve after the reactor on the left** (to mass spectrometer, valve 2 in the picture below)
    - Close the **lower green valve** (3 in the picture below) after the reactor



- Open the program QUADSTAR 422
    - Open the magnetic valve of the mass spectrometer
        - Measure->Manual->DI/DO, double click on the valve button, then press OK
        - The valve is open, when then the ball turns green and you hear TWO clicks
    - Press Ctrl + S (only when you're sure that the valve is open – the pressure wave caused by the opening of the valve can brake the filament if it's on)
        - Tick Emission
        - Press OK
- Select: MID -> Versus time
    - Check that Emission is ticked, press OK. A new window opens
    - Choose co2hyd.mip as the method (modify the measured signals, if needed -> see Software manual Quadstar 422 for help)
- Select: File -> Save cycle data
    - Save according to the date yymmdd.mdc
    - Check that the Number of cycles is a very high number (e.g. 100 000)
    - Press: Save Measure-data + info

7 Heating/cooling (at any point of the experiment)

- Raise temperature up to the desired temperature:
    - For calcination and pre-treatment: Increase the temperature 15 °C at a time and let it settle until PV ~ SV–4 °C (process value is 4 degrees below the set value)

    - For adsorption/desorption/reaction: Increase the temperature 5 °C (or 10 °C) at a time and let it settle until PV ~ SV–2 °C (process value is 2 degrees below the set value)
    - When you reach 300 °C turn the RANGE switch from LOW to HIGH, at this point the temperature will rise automatically for a while – let it settle a little and continue to increase it gradually
    - Record spectra at suitable temperatures
        - During calcination/pre-treatment e.g. at 200 and 400 °C with the names as above
        - During adsorption/desorption/reaction at every 25 °C (or 50 °C)
    - Record spectrum at the final temperature
    - Record spectra during calcination/pre-treatment at final temperature at times 60 and 115 minutes (or at other suitable times), add minutes to the name of the spectrum, e.g. Rh/ZrO2_air600u60
- Cooling down to the desired temperature
    - Decrease the temperature 25 °C at a time and let it settle until PV ~ SV–4 °C (process value is 4 degrees below the set value)
    - At 300 ºC switch the RANGE button from HIGH to LOW (after the switch the temperature will go down for a while automatically – let it settle a bit and then keep on reducing it gradually)
    - Record spectra at suitable temperatures, e.g. at 400, 200 and 30 ºC

8 Changing gases (at any point of the experiment)

- Flush the line for the new gas for about an hour before it is needed
    - Empty the line of the previous gas (applies only to PLK line):
        - Keep all valves closed in the PLK line
        - Switch valve O2-PLK -> PLK, valve O2/PLK-H2 to O2/PLK and H2/O2/PLK -> OUT. This can be done only if O_2 or H_2 lines are not currently in use (O_2 or H_2 is not fed to the reactor)
        - Set the flow in the PLK line to 80 ml/min and let it drop back to 0
        - Set the flow to 0
    - Turn the gas alarm on
        - The gas alarm should be placed so that it's located above the gas bottle, if the gas is lighter than air (e.g. hydrogen) and below the mass flow controllers, if the gas is heavier than air (e.g. isobutene)
    - Next open the gas bottle you're about to use
        - Open the bottle
        - Open the valve in the line
        - NEVER adjust the middle valve in the reducer (defines the outgoing pressure)
    - Flush the line
        - Switch valves to feed the desired gas (valves O2-PLK and O2/PLK-H2) and make sure that the valve H2/O2/PLK -> OUT
        - First set the flow to 20 ml/min
        - After a moment, increase the flow to 60 ml/min
- When the line has been flushed, set the flow to the desired value (calculated beforehand, using the correction factors for different gases)
- Record spectrum (30 scans) 2 minutes before changing gas mixture into the reactor
- Decrease argon flow first to the desired value (if new gas is mixed with argon)

- Change gas / gas mixture of the feed:
    - If argon is not used, first turn argon out from the reactor (N2 -> OUT)
    - Then turn valve H2/O2/PLK -> IN (at even minute)
    - Record the spectra according to time 1/min (30 scans, named according to time, i.e. as software suggests) for 5 or 10 minutes (first spectrum at the same minute as flow was turned into the reactor)
    - After first 5 (or 10) spectra, record spectrum (100 scans) and name it as usual
    - Follow the changes by recording spectra every 5 minutes for 30 min (last spectrum at 25 min)

9 Oxidation/cleaning of the reactor at the end

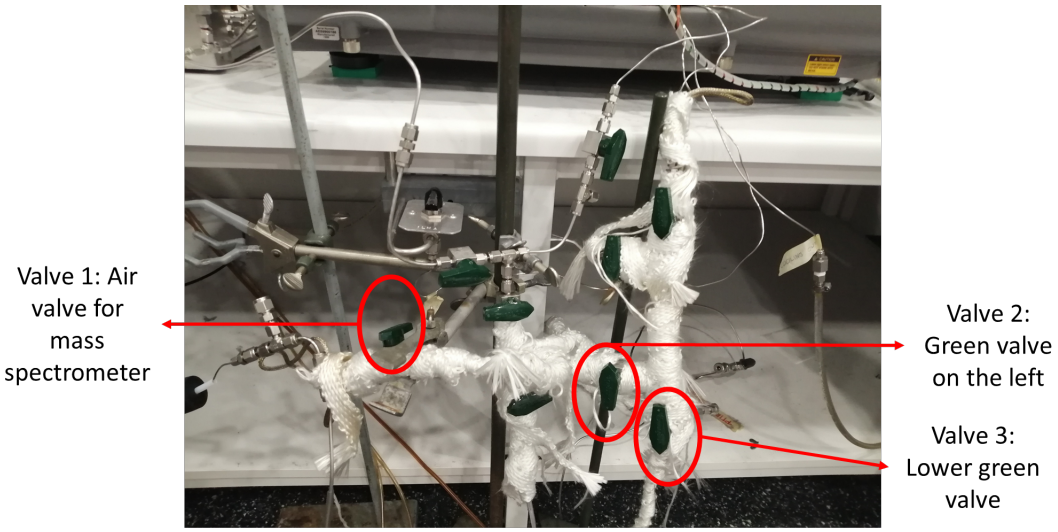
- It is highly recommended that after adsorption/reaction experiment, the sample and the reactor is flushed with oxygen at the highest temperature used in the experiment (to oxidize possible carbonaceous residues from the system)
    - The oxidation is done in two cycles, each of which lasts 10 minutes
    - When you change the flows, always decrease the other one before you increase the other!
    - During the oxidation you record spectra with 30 scans and save them according to the clock 1/min
    - The flows during the cycles:
        - Ar = 29.4 ml/min ja O2 = 5 ml/min for 10 min (-> 2% O2/inert)
        - Ar = 16.2 ml/min ja O2 = 25 ml/min for min (-> 10% O2/inert)

10 Ending the experiment

- After cooling down to 30 ºC and recording a spectrum

Stopping mass spectrometer

- Select: Operation -> Halt
- Close the window: the program asks: ”Stop saving?” answer Yes
- Turn the gas flow from the reactor away from the mass spectrometer
    - Open the **lower green valve** (valve 3 in the picture below) after the reactor
    - Close the **green valve after the reactor on the left** (to mass spectrometer, valve 2 in the picture below)
    - Open the **air valve** (valve 1 in the picture below) for mass spectrometer



- Turn of emission:
  - Press Ctrl + S
  - Remove the cross from Emission
  - Press OK
- Close the magnetic valve to the spectrometer
  - Measure -> Manual -> DI/DO, double click on the valve button and press OK
  - The valve is closed when the ball turns grey and you hear a click
  - Close the magnetic valve only when the filament has been turned off (emission)
- Close the program
- Turn off the heating (press the HEATER button)

With DRIFTS

- Turn the argon flow (and oxygen flow) away from the reactor (IN -> OUT)
- Decrease the gas flows to zero
- Switch HEATER unit from ENABLE to DISABLE and turn the heater unit off (back of the unit)
- Turn the cooling line valve below the equipment from VESI to ILMA and open the pressurized air line above the equipment (blue valve) to dry the line for 5 minutes
- Close the main valve for the cooling water and let the cooling line drain
- Turn off the mass flow controller unit
- Turn the pressurized air off from the valve above the equipment and turn VESI/ILMA to middle position (closed – points left). Close the pressurized air line above the equipment (blue valve)
- Close the gas bottles (bottle valve and line valve, NEVER adjust the middle one in the reducer)
- Switch off the gas detectors and start recharging if needed

11 Removing the Sample

- Get a piece of tissue, the waste jar and a brush, also remember your lab coat, protective gloves and the respirator
- Open the chamber and the mirror doors
- Remove the screws from the reactor dome (8 – first loosen the screws opposite each other) and move the dome top aside
- Remove the two black screws holding the sample holder base to the sample array

- Lift the sample holder base out of its casing and carefully tip the sample into the waste jar. Be careful not to hit the sample holder anywhere. Use the brush to aid emptying the sample holder.
- Check the reactor dome top seals (two small ones in the cooling line and one larger between the dome top and the sample holder base base). Replace if needed (from the black case)
- Replace the sample holder base in the sample array
- Close the mirror doors
- Close the waste jar and clean the brush on the tissue
- Screw the two black screws back in to the sample array
- Close the chamber

12 Saving the DRIFTS Spectra

On the computer of the spectrometer:

- Save each spectrum as a spectral file in the folder:
  My Documents\Omnic\Spectra\"yymmdd_Catalyst" (create new folder first)
  - Edit -> Select All
  - File -> Save as…
  - Set Filename to Title and Save
  - Repeat until all spectra has been saved
- Create a new folder for Kubelka-Munk data into
  My Documents\Omnic\Spectra\"yymmdd_Catalyst"\Kubelka-Munk
  - Convert data into Kubelka-Munk
  - Edit -> Select All
  - Process: Other Conversions -> Kubelka-Munk
  - File -> Save as…
  - Set Filename to title and Save
  - Repeat until all spectra has been saved
- Save all Kubelka-Munk spectra as CSV-files:
  - Edit -> Select All
  - File -> Save as
  - Change filetype into CSV
  - Set Filename to title and Save
  - Repeat until all spectra has been saved
- Collect data files with a USB-stick

On your own computer:

- Open CSV-files in Excel (or Matlab)

13 Collecting of MS data

On the computer of the MS:

- Open the program QUADSTAR 422
  - Select: Dispsav -> Process -> Cycles… -> File name: select according to date yymmdd.mdc, press OK
- Save the results of the mass spectrometer in ASCII format: File -> Convert to ASCII (name comes automatically)
- Close the window and turn off the screen (keep the MS computer on)
- Open and convert ASCII-files in Excel

14 Examples of experiments / parts of experiments

14.1 Calcination and reduction

- Open the argon bottle (and air bottle if needed) and the valve in the reducer (make also sure that the valves in the lines are open), NEVER adjust the middle valve in the reducer (defines the outgoing pressure)
- Turn on the cooling water:
    - Open the water valve fully (on the wall on the right side of the computers)
    - Turn the cooling line to VESI
- Turn on the mass flow controller unit
- Turn on the heating unit (from the back) and switch HEATER from DISABLE to ENABLE
- Make sure that the SV (set value) temperature is 30 °C in the heater
- Set the gas flow according to your calcination/heating requirements
    - Calcination: Ar = 18 ml/min (= 25 ml/min) and O2 = 25 ml/min (-> 10% O_2/inert)
    - Heating in inert: Ar = 36 ml/min (= 50 ml/min)
    - Turn the flow(s) into the reactor (OUT -> IN), first inert and then O_2 (if used)
    - Check the pressure gauge to make sure there are no blockages
- Record the spectrum at 30 °C (100 scans) with the name "catalyst"_"gas"_"temp"u by selecting "Col Smp" from the buttons on the top row
    - "catalyst" is the name of your sample
    - "gas" is the gas or gas mixture used, e.g. Ar or air
    - "temp" is the temperature, e.g. 030
    - u means up (other symbols: d down, f flush etc.)
    - Example: Rh/ZrO2_air030u
    - The connection between the computer and spectrometer might sometimes be off (there is no blinking light in the spectrometer at "Scan"), some tricks to help with this
        - If you see that the light is not blinking select: Collect -> Experiment Setup, when the light starts to blink, press OK
        - If the spectrum measurement says "Timed-out", close the measurement window (the lower X in the top right corner) and start the measurement again
        - If the Background is lost (there is no "Bkg Age" text in the bottom of the window and/or the y axis shows "Sigle Beam", stop the measurement and try again
    - Write down each spectrum with its measuring time into the laboratory notebook (on the computer table). Write down also the maximum peak value during recording each spectrum (shown below the spectrum while recording).
- Raising the temperature up to 600 °C (or any other pre-treatment temperature):
    - Increase the temperature 15 °C at a time and let it settle until PV ~ SV–4 °C (process value is 4 degrees below the set value)
    - When you reach 300 °C turn the RANGE switch from LOW to HIGH, at this point the temperature will rise automatically for a while – let it settle a little and continue to increase it gradually
        - Record spectra at suitable temperatures, e.g. at 200 and 400 °C with the names as above
    - Record spectrum at the final temperature
    - Record spectra during calcination/pre-treatment at final temperature at times 60 and 115 minutes (or at other suitable times), add minutes to the name of the spectrum, e.g. Rh/ZrO2_air600u60

- Reduction: Flush the reactor with nitrogen for an hour:
    - Turn the oxygen flow out of the reactor (IN -> OUT)
    - Increase the nitrogen flow up to 50 ml/min
    - Record spectra at times 0 and 55 minutes with names “catalyst”_N2580u”time”
    - Decrease the oxygen flow to 0
- Start measuring with the mass spectrometer, when you’ve flushed the system with nitrogen for 45 minutes according to Chapter 6
- Record a spectrum when the system has been flushed with nitrogen for 55 minutes (100 scans) with the name “Catalyst”_N2_600u55
- Record a spectrum when the system has been flushed with nitrogen for 58 minutes (30 scans) with the name according to time
- Flush H2 line about an hour before it is needed
    - Turn the gas alarm on
        - The gas alarm should be placed so that it’s located above the gas bottle, if the gas is lighter than air (e.g. hydrogen) and below the mass flow controllers, if the gas is heavier than air (e.g. isobutene)
    - Open H2 bottle
        - Open the bottle
        - Open the valve in the line
        - Never adjust the middle valve in the reducer (defines the outgoing pressure)
    - Flush the line
        - Switch valves to feed the desired gas: O2/PLK-H2 to H2 and make sure that the valve H2/O2/PLK -> OUT
        - First set the flow to 20 ml/min
        - After a moment, increase the flow to 60 ml/min
- Set the flow of reducing gas (with hydrogen and carbon monoxide the value is 2.5 ml/min)
- Decrease the argon flow into 34.2 ml/min (= 47.5 ml/min)
- Turn the flow of the reducing gas into the reactor (OUT -> IN) (start timing – the reduction cycle lasts e.g. for 15 minutes)
- Record the spectra according to the clock 1/min (30 scans). The two last spectra are collected with 100 scans (when reduction has continued for e.g. 9 and 12 minutes)
- When the reduction has lasted the desired time, turn the flow of H2 out of the reactor (IN -> OUT)
- Increase the argon flow back to 32.6 ml/min (= 50 ml/min), flush the sample for half an hour
- During the argon flush, record spectra at suitable times, e.g. at 5, 10 and 25 minutes (100 scans) according to the clock
- Record a spectrum, when the nitrogen flushing has lasted e.g. 28 minutes (30 scans) according to the clock

14.2 Adsorption – desorption experiment (TPD)

- Perform calcination/pre-treatment according to Chapters 6 and 7 or calcination and reduction according to Chapter 14.1
- Flush the PLK line with the gas you wanted to adsorb on the catalyst according to Chapter 8
- Set the flow of the adsorbing gas to wanted (using the correction factors for different gases)
- Decrease the argon flow to appropriate value (using the using the correction factors for different gases and calculating that the actual total flow is 50 ml/min)
- Turn the adsorbing gas to the reactor (at even minute)

- Record 5 spectra according to the clock 1/minute (30 scans) and record the 6th spectrum (100 scans) when the adsorbing gas has been inserted for 5 minutes, continue for e.g. 30 minutes by taking spectrum at every 5 minutes
- Turn the adsorbing gas away from the reactor, when the desired time is up and increase the argon flow to 32.6 ml/min (= 50 ml/min) and decrease the adsorbing gas flow to 0
- Record 5 spectra according to the time 1/minute (30 scans) and record the 6th (100 scans) when the flushing has continued for 5 minutes.
- Record spectra also at 10, 15, 20 and 25 minutes (100 scans) according to time
- Increase temperature in steps of 25 ºC or 50 ºC to measure the spectrum according to Chapter 7

14.3 Adsorption (while heating) experiment (TPA)

- Perform calcination/pre-treatment according to Chapters 6 and 7 or calcination and reduction according to Chapter 14.1
- Flush the PLK line with the gas you wanted to adsorb on the catalyst according to Chapter 8
- Set the flow of the adsorbing gas to wanted (using the correction factors for different gases)
- Decrease the argon flow to appropriate value (using the using the correction factors for different gases and calculating that the actual total flow is 50 ml/min)
- Turn the adsorbing gas to the reactor (at even minute)
- Record 5 spectra according to the clock 1/minute (30 scans) and record the 6th spectrum (100 scans) when the adsorbing gas has been inserted for 5 minutes, continue for e.g. 30 minutes by taking spectrum at every 5 minutes
- Turn the adsorbing gas away from the reactor, when the desired time is up and increase the argon flow to 32.6 ml/min (= 50 ml/min)
- Record 5 spectra according to the time 1/minute (30 scans) and record the 6th (100 scans) when the flushing has continued for 5 minutes, continue for e.g. 30 minutes by taking spectrum at every 5 minutes
- Insert the adsorbing gas to the reactor in cycles while increasing temperature:
    - At every 100 ºC flush the sample with nitrogen for 5 minutes
    - Record a spectrum immediately (100 scans) “catalyst”_”gas”030u
    - First cycle:
        - Heat the reactor 5 ºC at a time to 50 ºC and record a spectrum
        - Record spectra every 4 minutes and 25 ºC
        - At 100 ºC record a spectrum
        - Turn the adsorbing gas away from the reactor after recording and flush for 5 minutes
        - After flushing for 5 minutes record the spectrum “catalyst”_”gas”100f5 (f=flush)
        - Turn the adsorbing gas back to the reactor and continue heating
    - Repeat until you’ve recorded a spectrum in 500 ºC
    - At 300 ºC turn the RANGE switch from LOW to HIGH. The temperature rises automatically for a while after this
    - Turn the adsorbing gas away from the reactor and record the flushing spectrum as before
    - Continue the flushing for 30 minutes (spectra at 5, 10, 15, 20 and 25 minutes)
    - Empty the PLK line and flush it with oxygen
    - The experiment continues with oxidation according to Chapter 9 and cooling according to Chapter 7

15 Using a Saturator (NOT updated 2021)

- A saturator enables e.g. water, ethanol or toluene (liquids) to be used as adsorbents
- The idea is that you bubble nitrogen through the liquid in the saturator
    - the N_2 flow needs to be optimized for each liquid, however use a maximum of 50 ml/min
    - For some liquids you need to heat the saturator to increase the liquids volatility
- When using a saturator you need to use line heating (temperature ca. 120 ºC) to keep the substance from condensing in the lines
- Performing adsorption using a saturator:
- Perform the calcination and possible prereduction as shown above and cool to the desired temperature
- Bubble nitrogen through the saturator at 50 ml/min (N2 2 on the Raman mass flow controller) and direct the gasses to the outtake
- Open a new window and record the last spectrum of the nitrogen flushing
- Set the flows of the nitrogen going through saturator and the ultra pure nitrogen to a total of 50 ml/min
- Turn the gas coming from the saturator to the reactor (on an even minute)
- Record 5 spectra according to the clock 1/minute (30 scans) and record the 6^{th} spectrum (100 scans) when the adsorption gas has been inserted for 5 minutes
- If needed continue the adsorption and record spectra every 5 minutes (100 scans)
- Turn the adsorption gas away from the reactor away from the reactor and increase the flow of the ultra pure nitrogen to 50 ml/min (on an even minute)
- Record 5 spectra according to the clock 1/minute (30 scans) and record the 6^{th} (100 scans) when the flushing has continued for 5 minutes
- Record spectra also at 10, 15, 20 and 25 minutes (100 scans) according to the clock
- Flush the saturator line with nitrogen (to the outtake). Turn the 3 way valves to bypass the saturator
- Flush the PLK line with oxygen during the nitrogen flushing
    - Increase the oxygen flow to 50 ml/min
    - Turn the PLK/O_2 valve O_2 -> PLK
- Open a new window for the TPD spectra
- Perform the TPD as above
- The experiment continues with oxidation and cooling as above