

## Mémoire, Partim B

**Auteur** : Henin, Adèle

**Promoteur(s)** : Focant, Jean-Francois; Stefanuto, Pierre-Hugues

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# **Annexes**

## Annex 1: Standardized Operational Procedure: Exhaled breath sampling

- Prepare and verify the material
- Verify the flow of the pump with the flowmeter  
The flow must be calibrated slightly under 200 mL/min, at 185 mL/min for example, in continuous flow meter. Refer to the [manual instruction](#) to calibrate the flow of the pump
- Unlock the sampling bag's valve and blow in the bag. Once it is full, lock the sampling bag's valve
- Connect the decapsulated TD tube to the pump and the TD tube to the bag  
The arrow on the TD tube stand for the way of sampling, it must go from the bag to the tube.
- Unlock the valve and push on the pump on the same time to concentrate the VOCs contained in the bag onto the TD tube
- Push off the pump when the time of sampling<sup>9</sup> is over.  
*Note: 1 minute=100 on the pump*
- Disconnect the pump, the tube and the bags and put back the golden caps on the tube.  
*The TD tube can be stored for 3 month and keep the sample integrity*

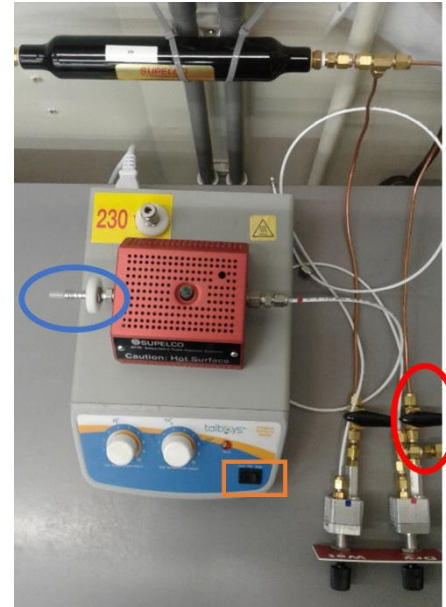


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<sup>9</sup> Volume to sample / Flow of the pump

## Annex 2: Standardized Operational Procedure of ATIS Experiment

- Put the ATIS instrument on thanks to the switch (orange box). The temperature is normally settled around 120°C
- Connect the gas bottle to the ATIS instrument by the carbon filter
- Unscrew the piece circled in blue.
- Place the flowmeter in front of this outlet
- Open the gas bottle and the gas valve circle in red to open the flow through the ATIS instrument
- Set the nitrogen flow to 100 mL/min with the flow meter
- When the flow is settled, close the gas valve and gas bottle but keep the regulator valve settle.
- Screw back the little piece
- Verify the temperature of the instrument with a thermometer, placing it in the hole indicated by the green arrow. When the temperature reaches around 110°C, the instrument is ready
- Open the gas bottle
- Fill a sampling bag with exhaled breath at 80% of the capacity. *You can use paper clip to fold the bag side and get 4/5 of the initial bags surface*
- Connect the bag to the ATIS instrument indicated by the yellow arrow
- Prepare the volume of standard in a fixed needle *Generally, 2  $\mu$ L of a solution concentrated between 30 to 50ppm of standards mix of the exhaled breath*
- Open the bag's valve
- Inject in the ATIS' chamber, indicated by the blue arrow the prepared volume of standard and turn on simultaneously the gas flow
- Start the chronometer, for 1 minute if it is a 1 L bag
- Once the time is over, close the gas flow and lock the valve of the bag
- The bag can now be concentrated onto a TD tube, according to its SOP



## Annex 3: Standardized Operational Procedure of exhaled breath sampling materials reconditioning

### A. TD tubes reconditioning

- Verify that the conditioner is connected to the nitrogen gas bottle
- Place the TD tubes without the caps into the tube conditioner support. The arrow of the TD tubes must be opposite to the flow direction of the tube conditioner
- Open the Nitrogen gas bottle and set the outlet pressure around 50 bar
- Verify the pressure coming out of the tubes with the flow meter, settled in N<sub>2</sub> mode

The flow must be comprised between 50 and 100 mL/min

- Insert the tube conditioner support in the oven manifold
- Set the reconditioning cycle program according to the parameters of the tube

*For exhaled breath sampling, we use generally Tenax<sup>®</sup>*

*TA/Carbopack<sup>™</sup> B or Tenax<sup>®</sup> GR/Carbopack<sup>™</sup> B TD tubes. The parameters of the reconditioning cycle are 300°C for 3 hours.*

*The top screen (green) is used for temperature set the bottom set (blue) is for the time.*

- Use the arrow buttons to modify the parameter, press on “P” buttons to validate your new program, and then, start the reconditioning cycle by pushing on “U” button of the timer screen
- Once the cycle is over, the timer turns back to 00.00 and the effective temperature decrease gradually (green characters). Remove the tube conditioner support from the oven manifold and let the TD tubes cold down, under the nitrogen flow
- When the TD tube are at room or acceptable temperature, recaps them with the golden caps and with the help of the tightening caps’ tools
- Close the Nitrogen gas bottle
- The tubes can be store approximately 3 months before using them

You can also follow the procedure on the following links: [Markes' use instructions video](#)



## B. Sampling bags UV treatment

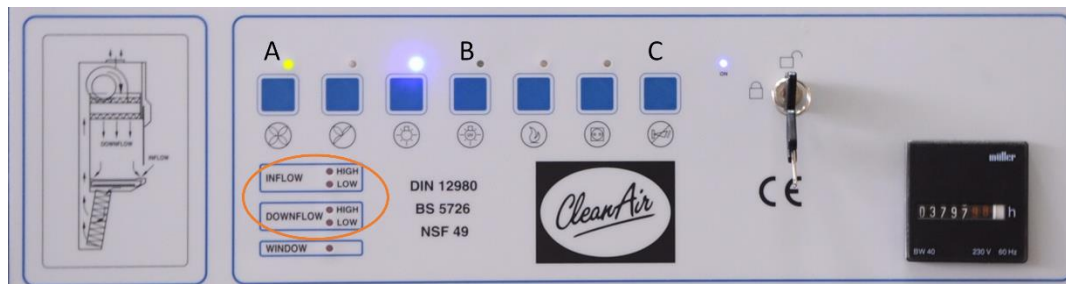
*This step must be done after the exhaled breath sampling and before the bags reconditioning step*

*You must wear a lab coat and lab gloves*

### *Specific hood procedure*

Here is a specific procedure to repeat at each time you want to manipulate under the hood

- Open the ventilation hood by pushing on A button
- Open the windowpane to the indicating line on the side walls  
Turn off the alarm by pressing on C button if it rings
- The hood is ready when the indicators (circled in orange) are switched of
- Wash your gloves as you wash your hand with ethanol 70% spray



- Once the specific hood procedure is over, spray each face of the bags with ethanol 70% and place them into the hood
- Close the hood's windowpane when the set of bags is correctly placed in the hood  
The hood alarm would trigger
- Press on the A button to deactivate the ventilation, the alarm will stop too
- Begin the UV cycle by pressing on the B button.  
The alarm sound will activate itself several times, push on the C alarm button to deactivate it  
A UV cycle takes 30 minutes
- Once the UV cycle is over, repeat the specific hood procedure and turn each bag over
- Close the hood's windowpane, deactivate the ventilation (button A), and begin the second UV cycle
- When the two faces of the bags are treated, push on B button, open the hood according the specific hood procedure and take back the bags
- Do not forget to close the hood's windowpane and pushing on the A button one last time
- You can dry the bags with paper towel before the next part of the bags reconditioning step

### C. Sampling bags reconditioning

- Set the temperature of the oven to 50°C
- Press on the A button and used the arrow buttons (B, C and D) to set temperature to 50°C.

Press a second time on the A button to validate the new temperature

*The top screen indicates the actual temperature of the oven and the bottom screen indicates the settle temperature*

- Make space in the oven to have enough place for the set of bags

*When you open the oven door, switch off the fan (yellow box)*

*Let at least an oven shelf over the oven ventilation to not obstruct the air circulation*

- During the adaptation temperature, fill the bags at least at 80% of the full capacity with pure nitrogen of the tube conditioner<sup>10</sup> gas bottle

- Verify that the bag valve is closed

- Once the oven temperature is reached, put the bags in the oven for 30 minutes

- Get the first bags out of the oven and flush it

- Repeat this step one bag by ones

*It is important to flush them as soon as the oven exit to directly eliminate the VOCs and the vapor water, that could recondense.*

- Flush and fill at 80% of the full capacity three times with Nitrogen each bag

- Flush all the bags and lock the bag's valve

- Clean the outside of the bags' valves with Ethanol 70% on a paper towel

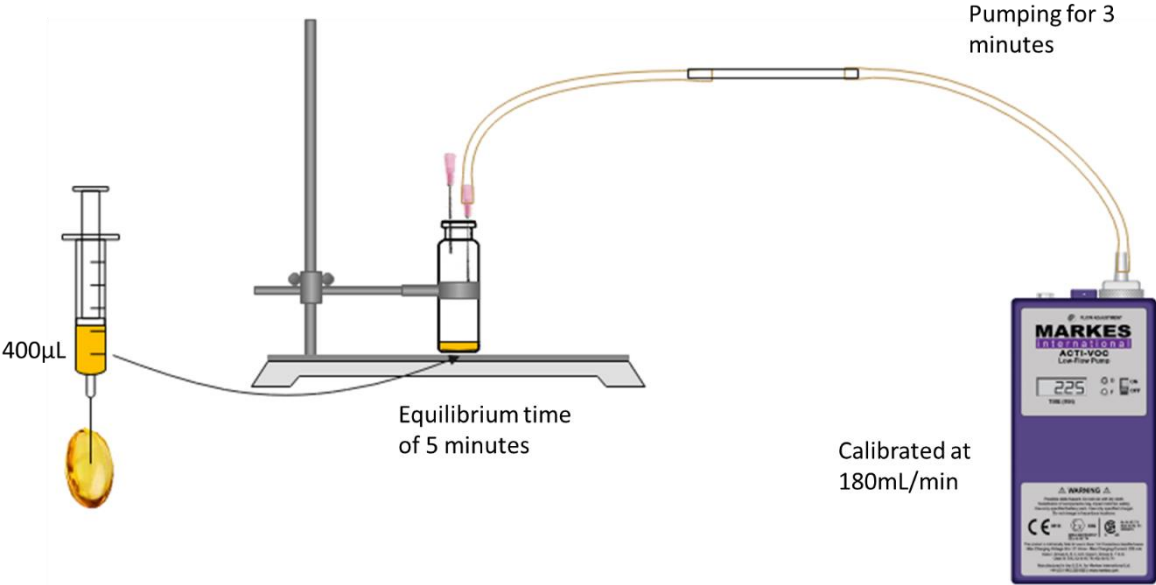
- Do not forget to add a cross on the bags label to control the thermal cycle number



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<sup>10</sup> If the gas bottle is connected to the tube conditioner, disconnect it with a spanner and add a plastic tube for the junction

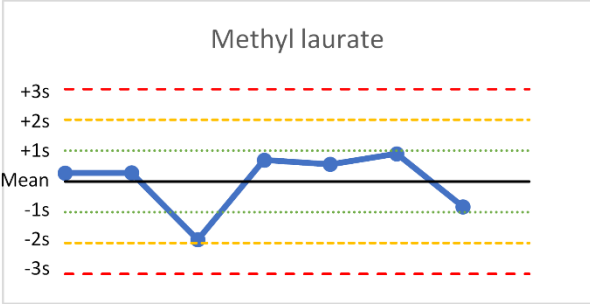
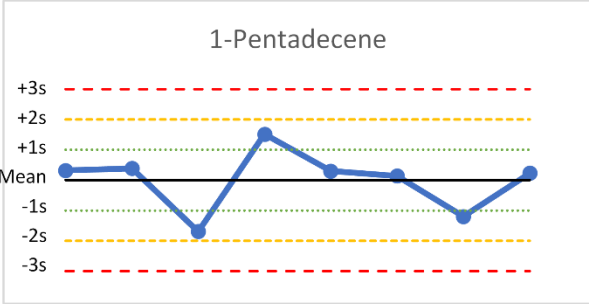
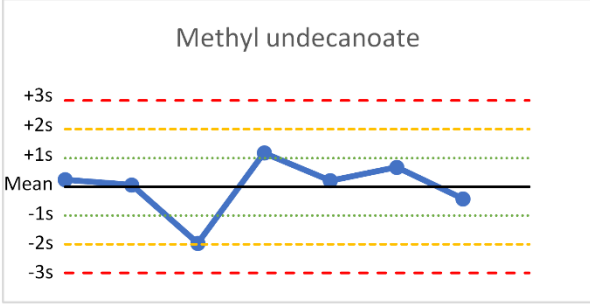
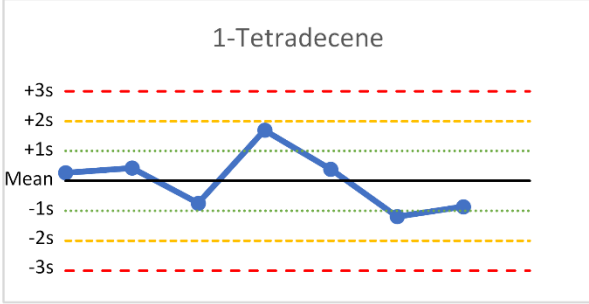
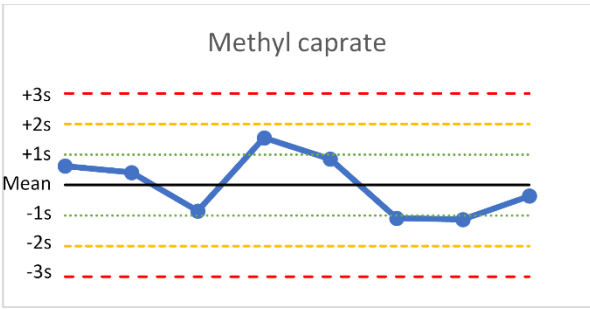
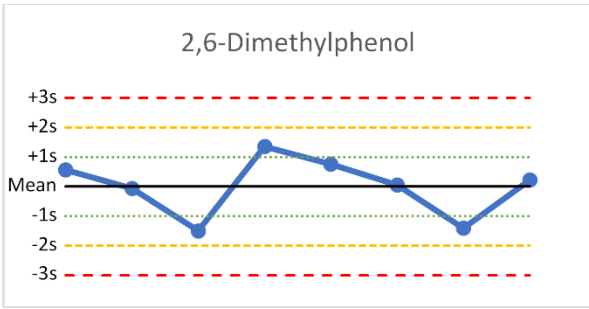
Annex 4: Peppermint oil analysis





## Annex 5: QC charts of the TD-GC×GC-HRTOFMS





Annex 6: Washout trend of all the volunteers tested for the study

