The Heidelberg Integrated $^{14}CO_2$ Sampler

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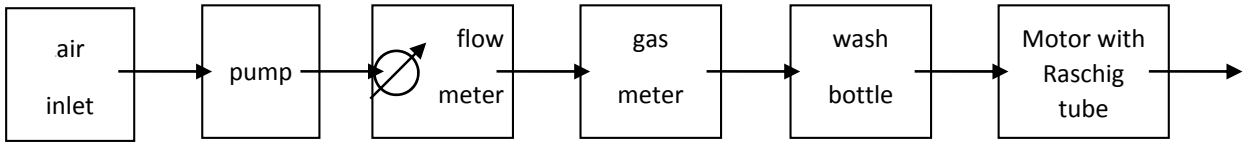
1. Description of the sampling technique and operation mode

With the $^{14}CO_2$ sampler atmospheric CO_2 is being collected over about two weeks to be analysed for its $^{14}C/^{12}C$ ratio. The CO_2 is absorbed in sodium hydroxide solution (NaOH), which is sent to the Central Radiocarbon Laboratory for analysis.

This is the schematic set-up of the sampler:



Atmospheric air is being pumped from the air inlet through a flow meter, where one can adjust the airflow with a needle valve. For a conventional ^{14}C analysis, CO_2 from about 25 m^3 air is necessary. Therefore, the mean flow rate for a sample integration time of two weeks must be about 75 l/h. After the flow meter, the air passes a gas meter where the amount of the already passed air is monitored. Then the air goes through a wash bottle to get humidified. This prevents the NaOH solution to get too concentrated. Now the air flows through the glass Raschig tube where the CO_2 is absorbed by the NaOH in the tube. The tube is slowly rotated with an evaporator drive to renew the absorbing NaOH film on the Raschig rings. The motor acts as a vacuum rotary feedthrough.

2. Installation of the sampler and start of first sampling

The above described parts of the sampler have to be arranged as shown in figure 1. The metal plate with the pump must be screwed on the four rubber buffers (figure 2). Also, the flow meter has to be fixed at its small pedestal with the two screws (figure 3). The gas meter is put on its holding and fixed with the elastic bands (figure 4). The wash bottle stands in the ring-shaped bottle fitting (figure 5).

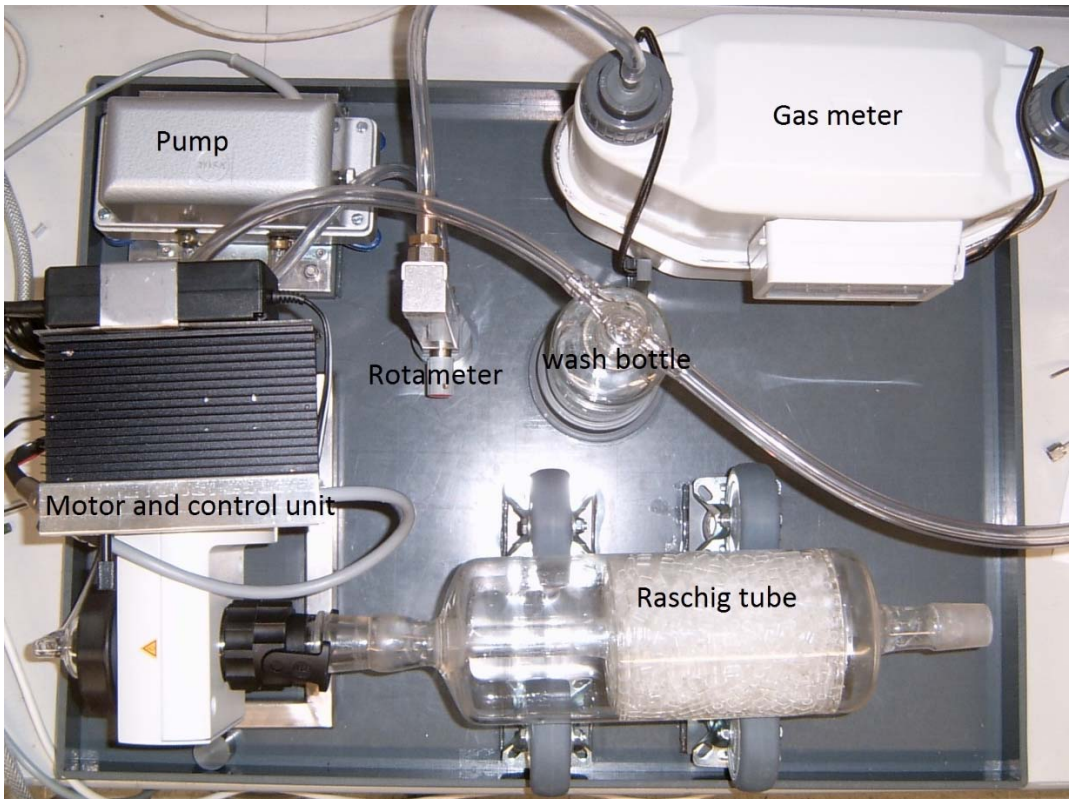


Figure 1: View on the $^{14}CO_2$ sampling system

Figure 2: Installation of the pump

Figure 3: Flow meter (back)

Figure 4: Gas meter

Figure 5: Wash bottle

Now stick up the black attachments from the motor on the hollow glass shaft as shown in figure 6 and screw it together. Put the hollow shaft (see figure 7) into the motor, then from the other side the gasket ("Drive side" must show to the motor), the glass adapter for the flexible hose and the metal spring. Finally, fix all parts with the remaining black plastic ring. For easy assembling one can lock the motor by the "lock"-button.

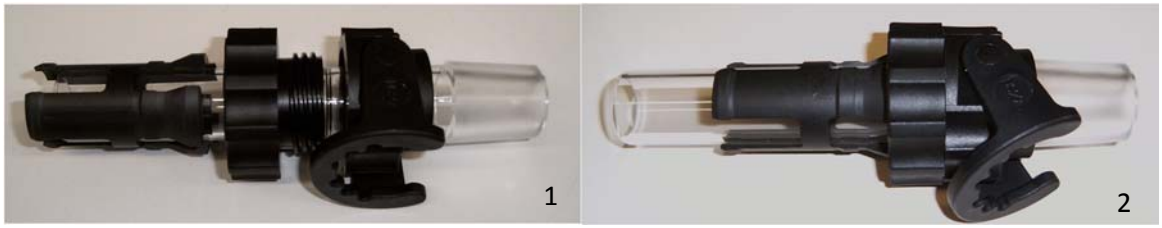


Figure 6: Hollow glass shaft with attachments

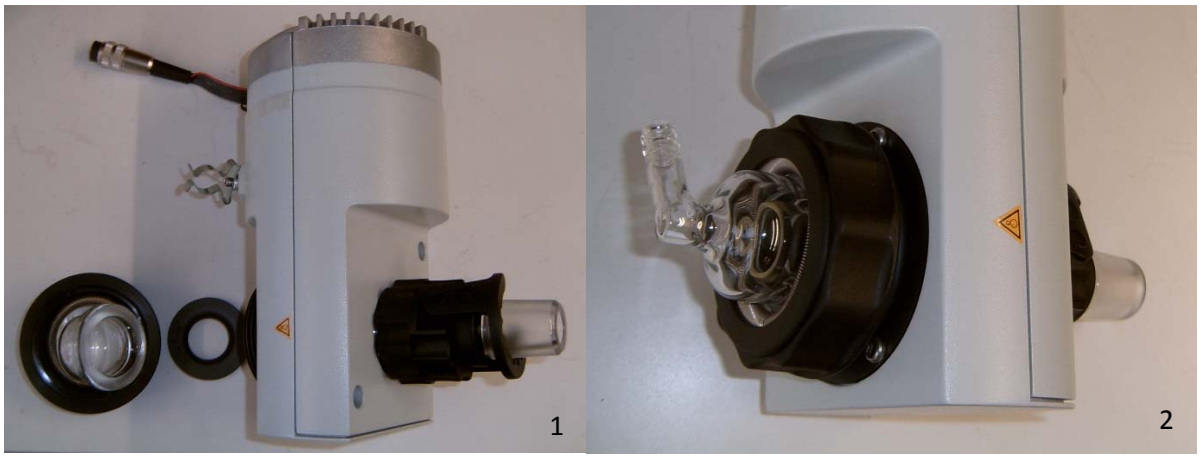


Figure 7: Adjustment of the hollow glass shaft at the motor

Put the metal rack for the motor on the plate as shown in figure 8. The motor is orientated over the pedestal to stand safely. Now put a Raschig tube on the glass hollow shaft to adjust the position of the motor. When the tube lies horizontally on all four rolls one can fix the clip at the backside of the motor with the four screws.

Figure 8: Mounting of the motor

Now fix the power supply and control box at the top of the metal rack with the screws (figure 9), and slide the power supply unit into the provided attachment. Then plug in the wire of the power supply and put the wires from the motor and the control box together.

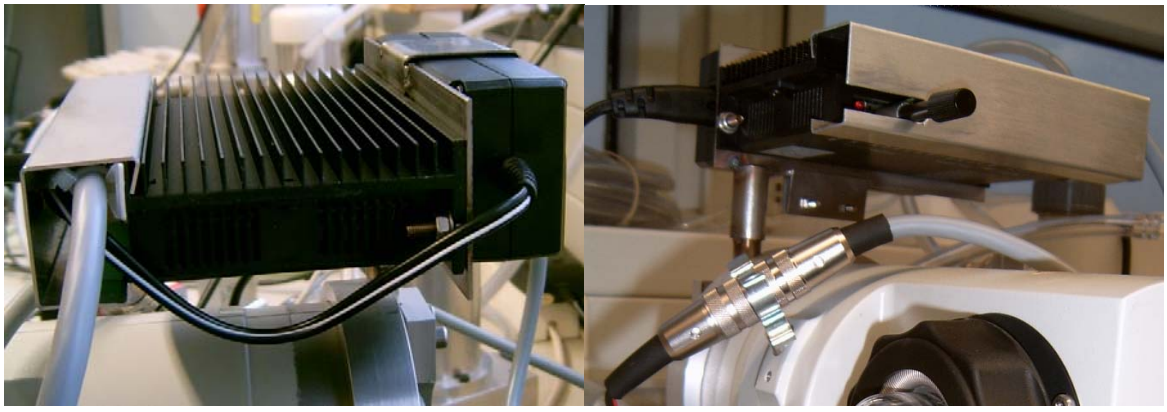


Figure 9: Control box with its wires

From pump via flow meter to the gas meter the thinner tubing with 6 mm inner diameter is used. From the gas meter via wash bottle to the motor one has to use the thicker tubing with inner diameter of 8 mm. Use the hose clip to seal the transition from the pump to the hose (figure 2).

For the first filling of the Raschig tube with NaOH solution and for taking first samples see below (Section 3B).

3. Sample Change

Note: For the chemical absorption of atmospheric CO_2 we use highly concentrated sodium hydroxide solution. This strong alkaline solution destroys clothes and human skin. Please use gloves and safety glasses and protect your clothes. Be very careful in all handling procedures when changing the samples! If, by accident, it happens that some solution gets on the skin, first use a dry tissue to remove the droplet and then wash carefully with tap water.

After two weeks of absorbing the CO_2 from about 25 m^3 air the sodium hydroxide has to be changed. First, the NaOH solution is being filled backed into the glass bottle and the same Raschig tube will be filled with new sodium hydroxide solution:

A. Emptying the Raschig tube

1.) Switch off pump and motor at the multiple socket outlet.
2.) Fix the empty sample bottle with help of the white-blue plastic fitting and the metal clip at the end of the Raschig tube
3.) Take off the Raschig tube from the hollow motor glass shaft.

Attention: The tube can get off suddenly. Be careful not to lose NaOH solution from the tube.

The Raschig tube with the fixed empty bottle now has to be put vertically on the plastic pedestal. Now the sodium-hydroxide-(carbonate-)solution is running back into the glass bottle. Keep the tube for at least 5 minutes in this vertical position so that all the solution drains back into the bottle.

Figure 10: Plastic stand with Raschig tube and fixed sample bottle

4.) Usually there are about 50 ml of solution remaining in the tube. Because of this fill about 50 ml distilled water from the top into the Raschig tube to dilute the remaining solution and some crystals. This solution drains into the bottle additionally. Be careful not to add too much water.

5.) Now remove the bottle, close it and label it with the following data:

- name of the station & number of the sample
- sample time start and end
- gas meter start and end

6.) Remove the plastic fitting from the Raschig tube and clean it with water or diluted acid.

B. Filling the Raschig tube with new NaOH

1. Carefully clean the hollow glass shaft sticking in the motor and the glass shafts of the Raschig tube with a dry tissue. Then grease the ground-in stopper of the hollow glass shaft with the dosing syringe (figure 11). **Good greasing is important to be able to remove the Raschig tube after two weeks of sampling.**
   Then put the Raschig tube horizontally on the shaft and turn it slightly. Fix it with the black attachment (figure 9).

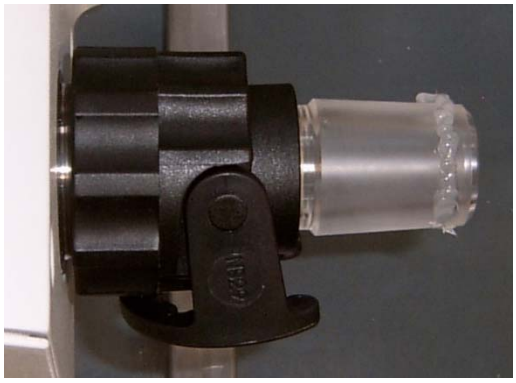


Figure 11: Greasing of the hollow glass shaft

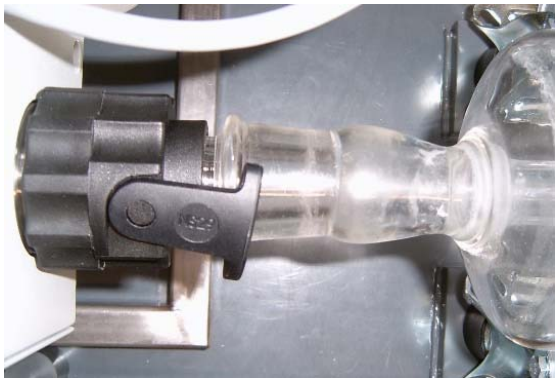


Figure 12: Fixation of the Raschig tube

2. In this position use the funnel with plastic tubing at the outlet of the tube to refill it with the content of one new glass bottle of sodium hydroxide solution (200 ml). Immediately close the bottle.
3. Restart the motor and make sure that the Raschig tube is rotating on all four rolls exactly horizontally and slowly (about 40 revolutions per minute).
   Attention: Turn control knob just a little to the right!
4. Refill the wash-bottle with distilled water (if distilled water is not available you can also use clean rain water or demineralized water; but it should be carbonate free). Grease the glass stopper and fix it with a metal clip.
5. Switch on pump and motor and check the flow rate at the flow meter. Flow rate can be regulated at the needle valve and should be ca. 75 l/h for a two-week integration period.

6. Note the sample change in the log sheet with:
   - sample No.
   - start date & time
   - gas meter start value
   - flow rate

   Please also note special changes.
7. If possible please check the actual flow rate and the gas meter value and note it together with date and time regularly. If necessary, adjust the flow rate. If the air is very dry, the wash bottle should be refilled during one sampling interval so that the sodium hydroxide solution does not become too concentrated or clumped together.

Please send the samples about every 2-3 months to the following address:

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