

FLASK SAMPLE COLLECTION USING THE AUTOMATED PORTABLE
SAMPLING UNIT (PSU)

Revision 1.0 (MS Word)

(applies to HPD Sherpa 70 with optical valve sensors and new
program)

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INTRODUCTION

The Automated Portable Sampling Unit (PSU) sampler replaces the MAKS sampler used since 1990 to collect air samples throughout the NOAA/CMDL air sampling network. The automated PSU is similar to the MAKS but it incorporates two major improvements. First, a condenser has been added to remove a significant amount of water vapor from the sample by cooling the air stream. This change will improve the CO₂ measurements and the ¹⁸O/¹⁶O precision from samples collected at humid tropical locations. Second, the sample collection procedure has been automated by adding a Micro Controller Unit, electronic valves, and a pressure switch. To collect a sample, the sample collector simply turns on the power and presses the sample mode button. The PSU will cool and flush the condenser, flush the flasks with dry air, and then pressurize the samples. A set of light emitting diodes (LED's) indicates the status of the PSU during and after sample collection. The increased automation will make sample collection easier and more reliable.

GENERAL GUIDELINES

The objective of our project is to determine the concentrations of carbon dioxide (CO₂), methane (CH₄), carbon monoxide (CO), and the isotopic ratios (¹³C/¹²C and ¹⁸O/¹⁶O) of CO₂ in “background” air. By “background” we mean air that is representative of large space scales (on the order of 1000 km²) and that has not been influenced locally. Since many human activities will generate the species we are trying to measure, it takes some thought and care to avoid contaminating the sample. It only takes a small amount of locally polluted air to completely invalidate a sample.

The detailed procedures for collecting air samples with the PSU are given in the following sections. Several general guidelines for collecting samples are given here.

1. Always carry the PSU outside to collect the sample. Never collect a sample by putting the intake out a window, door, ceiling hatch, or by connecting it to another air intake system.
2. Walk at least 75 m into the wind (upwind) from all buildings, vehicles, machinery, animals, etc.
3. Avoid structures or terrain features in the immediate vicinity or upwind of the sampling site that could affect the wind speed or direction.
4. It is best to sample when the wind speeds are greater than 2 m s⁻¹. If low wind speeds are persistent, collect a sample anyway and make a note on the sample sheet that it is a “last resort” sample.

5. Collect samples at regular time intervals. This is usually once or twice per week, depending on the site.
6. Avoid exposing the flasks to light, especially sunlight. Keep the PSU closed as much as possible during sampling. After collecting the sample, leave the flasks in the PSU until you are indoors again. Then remove the flasks from the PSU and place them in their shipping box. Store the boxes in a relatively cool area.
7. Safety glasses have been provided with the PSU. We strongly advise you to wear these or some other eye protection when handling the flasks and collecting samples. This will help prevent eye injuries if a flask breaks and all the glass is not contained by the tape.

DETAILED SAMPLING PROCEDURES

I. Install the flasks in the PSU

NOTE: This step should be performed indoors, if possible, to avoid exposing the flasks to direct sunlight. If it must be done outside, try to avoid direct exposure by using whatever shade is available.

1. Take two flasks from the shipping box and record the flask numbers and the date on the sample sheet.
2. Open the PSU, fold up the mast (but do not extend it yet), and remove the flask holding plate from the threaded rod.
3. Remove the red plastic covers from the male connectors on the flasks and wipe the connectors with tissues to remove any old grease or dirt. Also ensure that the female Teflon connectors in the PSU are free of dirt.
4. Place both flasks in the plastic holder with the higher numbered flask in the front (nearest you) and all the stopcocks on the right hand side. Position the stopcock handles up so they can be easily turned.
5. 2.5-L flasks have both connectors on one end. The one in the center is attached to a “dip tube” that extends to the bottom of the flask. This connector is always used as the inlet to the flask. The other connector is offset to one side; this connector is always used as the outlet from the flask.
6. Connect the inlet (center) connector of the flask farthest from you to the female connector labeled “pump.” Rotate the Teflon connector clockwise while pushing toward the flask. Connect the outlet (side) connector of the flask nearest you to

the female connector labeled “return.” Connect the outlet (side) of the farther flask to the inlet (center) of the nearer flask using the piece of tubing with two female connectors attached to it. Make sure all the connections feel tight.

NOTE: LEAK-TIGHT SEALS AT THESE CONNECTIONS ARE ESSENTIAL FOR COLLECTING GOOD SAMPLES

7. Replace the flask holding plate; gently tighten the wing nut; fold down the mast; close and latch the sampling case. You are now ready to bring the PSU and the sample sheet to the sampling site.

II. Setting up at the sampling site

1. Walk at least 75 m into the direction the wind is coming from (upwind) away from all vehicles, buildings, machinery, and animals.
2. Choose a sampling site free of upwind structures or terrain features that would interfere with the free flow of air to the PSU intake.
3. Choose the sampling site so that you can walk at least 10 m downwind during sampling.
4. Find a fairly level spot and set the PSU down on the bottom of the case. Open the case and lift the mast into the upright position. Make sure the mast hinge is latched in position.
5. Pull the rubber plug and the top (innermost) section of the mast out from the mast base and then extend the mast, one section at a time, to its full height (~5 m). In high winds it is not necessary to extend the mast to its full height. Extend the mast at least 2 m and note this variation on the sample sheet. Be careful not to kink the intake line while raising the mast. Try to keep the intake line wound around the mast as you extend it.

III. Collecting samples

Sample collection with the PSU is highly automated. The LED display will indicate whether or not sampling was completed successfully. In this section only the steps necessary to collect a sample are given. A detailed description of the sequence of events that occurs during sampling is given in Section VII.

1. Open all of the stopcocks. The order in which you open them is not important, but they must be completely open. To open the Teflon O-ring stopcocks, turn the handle counter clockwise until the O-ring is visibly clear of its seat.

2. Turn on the power. The LEDs and the valves will go through a test sequence. Press the “Sample Mode” button to start sample collection. Shut the PSU and walk at least 10 m down wind from the sampler.
3. The automated sampling procedure takes ~10 minutes. When sampling is finished, the pump will automatically shut off. Record this time (sample ending time) on the sample sheet.
4. After 10 minutes, return to the sampler, open the case, and note which LED’s are lit. If the “Good” LED (green) is lit, sampling was completed successfully. If this is the case, continue with the next step. If the “Good” LED is not lit, refer to Section VII for additional explanations and instructions. Indicate on the sample sheet which LED’s are lit.
5. Close the stopcocks in the following order:
 - 1) Stopcock connected to “pump” connector
 - 2) Stopcock connected to “return” connector
 - 3) The remaining two stopcocks in any order

Closing these stopcocks requires a careful touch: overtightening will break the flasks. To close the stopcock, turn the handle clockwise until the O-ring visibly seals against its seat, then gently tighten the stopcock while watching the sealing surface. The sealing surface should be ~1 mm thick (slightly less than the thickness of a dime). Closing the stopcocks is a critical step: insufficient closure will ruin the sample; overtightening will break the flask. With practice and care you will acquire a “feel” for this. You should always visually check the stopcock for complete closure.

6. Turn off the PSU power. Pack the mast down into itself, one section at a time. Be careful not to get dirt in the intake line if it touches the ground. Fold the mast in on top of the tubing. Latch the case shut before moving the AIRKIT.

IV. Unloading the samples

1. Fill in the sample sheet with available meteorological information. Use the “Notes/Comments” section to note any observations concerning sampling conditions, procedures, need for supplies, etc.
2. Unload the flasks indoors and try to avoid exposure to light.
3. Wipe the flask connectors with tissues and replace the red plastic covers on the ends of the connectors.

4. Please check to ensure that the flask numbers, date, and time are recorded correctly on the sample sheet. Pack the flasks and sample sheet back in the shipping box.

V. Dry Mode

After collecting a sample, it is necessary to run the PSU through the Dry Mode to purge any water collected in the condenser. It is very important to do this after every sample collection.

1. Plug the power supply into the PSU. Turn the PSU power switch on. All the LED's will light briefly. Press the "Dry Mode" button. The "Dry Mode" LED (yellow) will light and the pump will turn on. The pump will run for 30 minutes to purge the condenser. When the Dry Mode is finished, the pump will turn off, and the "Dry Mode" LED will turn off.
2. When the Dry Mode is finished, turn off the power switch and leave the power supply plugged in to charge the battery.

VI. Battery Charge Mode

1. Plug in the power supply. The "Charge" LED (yellow) will light. The "Charge" LED goes out when the battery is charged.

The battery and charger have been chosen so that the battery can be left on the charger between sample collections without being damaged. This ensures a full charge for every sample collection. If it is not practical to leave the unit on the charger, then charge the battery for at least 24 hours before each sample collection.

VII. Automated PSU Functions and LED Message System

The purpose of this section is to describe in detail the events occurring during automated sample collection and the significance of the LED messages. Table 1 summarizes the LED messages and gives instructions to follow when the LED's indicate that the sample was not collected successfully.

When the PSU power is turned on valves 1 and 2 move to the bypass position, the pump turns on, and the condenser begins to cool. The "High Temp" LED (yellow) is on, indicating that the condenser temperature is greater than 5° C. The air bypasses the flasks during this step to avoid flushing the flasks with moist air. This is the start of the PSU Automated Sampling Mode.

The bypass mode continues for 2 minutes. When the condenser temperature drops below 5 C, the "High Temp" LED turns off. If the condenser temperature is greater

than 5° C, automated sampling continues but the “High Temp” LED (yellow) remains lit. When the bypass mode is finished, valves 1 and 2 switch to the flush position, allowing dried air to flush through the flasks. The flasks are flushed for 8 minutes. If the voltage falls below 9 v, the pump and the condenser are turned off and the low “Batt” LED (red) is lit.

After flushing the flasks for 8 minutes, valve 2 is switched to the bypass position. This closes the exit path and causes pressure to build up in the flasks. The pressure is monitored by a pressure switch set to trip at 5 psig. If the pressure fails to reach 5 psig within one minute, the pump and condenser are turned off and the “Leak” LED (red) turns on to indicate unsuccessful sampling, most likely due to a leak. If the pressure switch is tripped within the one minute period, valve 1 is switched to the bypass position. The sample is now trapped in the flasks at 5 psig, and the “Good” LED (green) is lit. The pump and condenser are turned off.

Because the flask stopcocks are still open, the pressure switch is checked every 0.5 s. If the pressure falls below 5 psig, the “Leak” LED (red) will light to indicate the presence of a leak and the “Good” LED will switch off. As long as the pressure is held, the “Good” LED will light to indicate a successful sample.

When the 10 minute automated sampling cycle is finished (pump off), close the flask stopcocks and note the LED display. If the “Good” LED is on, sampling has been completed successfully. If the LED display indicates anything other than “Good”, refer to Table 1 for an explanation and suggested solutions to the problem.

After each sample collection it is necessary to remove any liquid water collected by the condenser. The PSU has a Dry Mode to accomplish this. To initiate the Dry Mode, plug the power supply into the main power, and then into the AIRKIT. With the PSU plugged in, turn on the PSU power switch. Each LED will light briefly, and the valves will go through a test sequence. Then press the “Dry Mode” button. The “Dry Mode” LED will light to indicate that the Dry Mode is in progress. The pump turns on and flushes the condenser for 30 minutes. When the Dry Mode sequence is finished, the pump and condenser are turned off and the “Dry Mode” LED turns off. Turn off the PSU power switch, but leave the power supply plugged in to charge the battery.

TABLE 1. LED Messages and Instructions

<u>Label</u>	<u>Color</u>	<u>Explanation/Instructions</u>
Sample Mode	Yellow	Automated Sample Mode is in progress
Dry Mode	Yellow	Dry Mode is in progress Dry Mode takes ~30 minutes.
Low Batt	Red	Battery voltage fell below 9 volts. Return to the lab, run the PSU through the Dry Mode, and charge the battery for at least 24 hours. If the voltage is still low on the next sampling try, contact NOAA/GMD.
High Temp	Yellow	Condenser body temperature is $> 5^{\circ}$ C. Send the collected samples to NOAA/GMD and contact NOAA/GMD.
Leak	Red	Flasks failed to reach pressure during pressurization, or fell below pressure after Sampling Mode stopped. Check the Teflon connectors for leaks and then repeat the Automated Sampling cycle. If there is still a leak, contact NOAA/GMD.
Good	Green	The Automated Sampling cycle was completed successfully. Close all 4 stopcocks and pack up the PSU.
Error	Red	Failure of valve one and/or two. The PSU will shut down when this condition occurs. Contact NOAA/GMD.
Charge	Yellow	Battery is charging. LED will go out when battery is fully charged.

CONCLUDING REMARKS

Our experience shows that while collecting good flask samples is relatively straightforward, it is not necessarily easy. Adherence to sampling procedures and attention to detail are essential to obtaining good samples. Departures from these procedures almost always result in contaminated samples. A moment's carelessness during sampling often results in a bad sample. Remember that it takes the same amount of time to collect a good sample as a bad one. It is only after you collect it and we analyze it that we know which it is. Reading and following these instructions will ensure that the maximum amount of information is obtained from the time you spend collecting the samples.

Thank you for your help in our studies of greenhouse gases. Without your conscientious cooperation this program could not function. If you have questions concerning the sampling procedures, the PSU, or if you need parts or supplies, make a note on the sample sheet or contact us directly.

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