

Desktop

VarioMICRO: program for EA instrument

MICROvCube_ShortManual: condensed operator manual

MICROvCube_Manual: full operator manual

Data: the folder that contains personnel folders and data

Template_CHNS_CNS: Excel template to record the weights of your samples

EA_Guide.pdf: this comprehensive guide to running the EA instrument

Methods

Principle of Method: Elemental Vario Micro Cube determination of total N and/or total C in soil, sediments, plant, or biologic material by flash combustion. Complete combustion of the sample is achieved by dropping a known mass of sample into a combustion tube containing Cr (III) Oxide and Cobaltous/ic Oxide Silver catalysts. A stream of purified oxygen is added to the quartz tube generating a flash combustion reaction which increases the temperature from 1020°C to 1800-2000°C. The flash combusted samples' carbon is converted to CO₂ and the nitrogen is converted to N₂ and NO_x. These combustion gases are carried via a stream of UHP Helium through a reduction furnace filled with reduced copper wires where NO_x is reduced to N₂. The gas stream now passes through a sorbent trap to remove water (Magnesium Chloride). The resulting N₂ and CO₂ gas is separated on a stainless steel mesh packed chromatographic column and detected quantitatively by a Thermal Conductivity Detector (TCD).

Sample Set Up

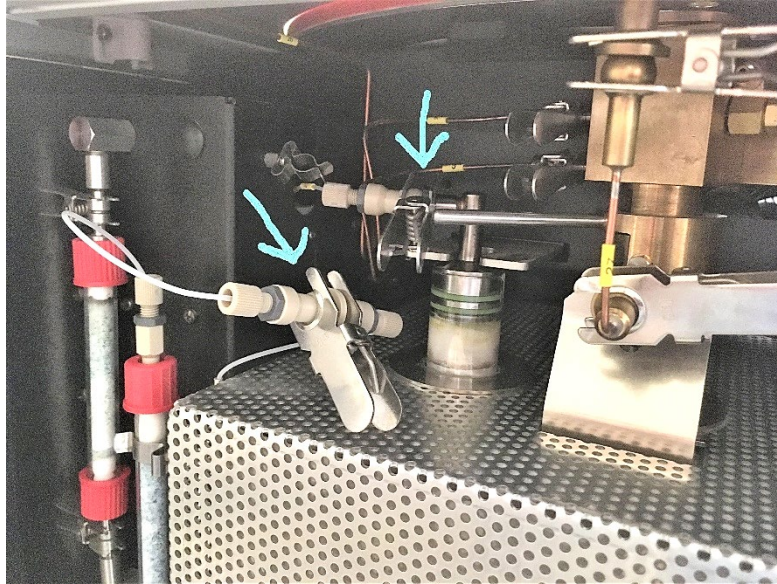
- You can run up to 60 samples per run
- Use the microbalance to weigh out your samples into tins
- Record the weights in the “Template_CHNS_CNS” Excel document
 - For surface soil, weigh out 15-20 mg
 - For deeper soil, weigh out 20-40 mg
 - For plant matter, weigh out ~9 mg
 - For biochar, weigh our 5-9 mg
- Carefully fold the material into the tins
- The tins should be in small spheres or cubes
- Ensure the encapsulated samples are not leaking
- Also weigh out 5 standards with the sulfanilamid standard
 - The standards should weigh 4.900-5.100 mg
- Store all samples in a microplate

Maintenance Check

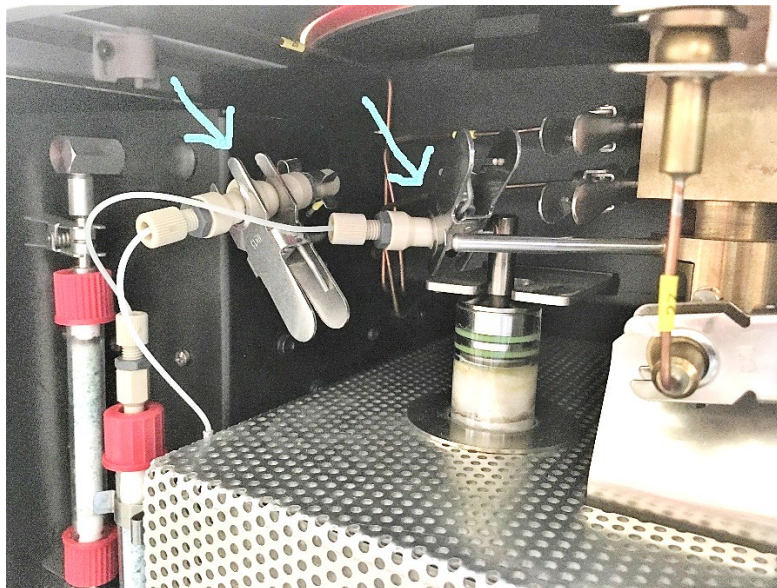
- **VarioMICRO** → click “OK” (there is no password)
- Click the rectangle at the bottom left to check for needed maintenance
- The ash finger needs to be changed the most often (every 60 samples)
- Check the # of samples run and the # at which the consumable needs replaced
 - Red: needs to be changed
 - Yellow: will need to be changed soon (potentially before running)
 - Green: does not need to be changed
- Open the EA door and check the two drying tubes
 - The drying tubes are fully used when they are completely blue
 - The large drying tube will turn blue from top to bottom
 - One run of 60 soil samples can use up to 1/4 of the large drying tube
- If any consumable needs to be replaced, see the “[Maintenance](#)” section

Changing EA Mode

- VarioMICRO → click “OK” (there is no password)
- Options → Mode → CHNS or CNS
 - CHNS → **while EA is off**, open EA door and change analysis configuration to not use the large drying tube



- CNS → **while EA is off**, open EA door and change analysis configuration to use the large drying tube



Data Analysis

Important: see the "[Maintenance Check](#)" and "[Changing EA Mode](#)" sections before turning on the EA or running it

Press the green button on the bottom right of the EA instrument to turn the instrument on
VarioMICRO → click "OK" (there is no password)

Click the moon icon "Sleep" and click "Sleep"

Leave the instrument on for ~24 hours before using

Open main valves for gas tanks (Helium and Oxygen)

Check gas pressures and flow rates!

- **Total amount of gas ≥ 300 psi** (right sensor on regulators)
- **Running the EA without proper gas pressure will destroy the detector!**
- **Flow rate: ~ 400 kPa** (left sensor on regulator)

Click the moon icon "Sleep" and click "Wakeup"

Wait for the instrument to heat up (combustion tube = 1150 °C; reduction tube = 850 °C)

Tasks:

- Options → Diagnostics → Rough leak test → click "ok" → wait for Ads. Column temperature to be cooler than 32 °C → click "Start"
- If leak test passes and no maintenance needs to be performed (see the "[Maintenance Check](#)" section), close leak test and move on to running your samples
- If the leak test fails, see the "[Fine Leak Test](#)" section

Use sign-in sheet:

- Record the date, your name, type and # of samples, amount of gas remaining (psi), etc.

Running your samples:

- File → Open → open the template named "Template_CNS" or "Template_CHNS" depending on which mode you are running (see the "[Changing EA Mode](#)" section → File → Save As → rename the file to identify your samples in the run
- Copy and paste in your sample weights and names (and moisture content if running CHNS mode) and change the method according to your sample type as follows:

- Slots 1-5 are blanks (empty, no samples)
 - Slot 1: Weight = 100; Name = Blank1; Method = Blank with O2
 - Slot 2-5: Weight = 100; Name = Blank; Method = Blank with O2
- Slots 6-10 are standards
 - Slots 6-7: Weight = 4.900-5.100; Name = RunIn; Method = 5mgChem90s
 - Slots 8-10: Weight = 4.900-5.100; Name = sulfanilamid; Method = 5mgChem90s
- Slots 11-70 are your samples
 - Method = Soil2 for soil; Biochar for biochar; Plant9mg for plant matter
- Slots 71-73 are blanks (empty, no samples)
 - Slot 71: Weight = 100; Name = Blank1; Method = Blank with O2
 - Slot 72-73: Weight = 100; Name = Blank; Method = Blank with O2
- Note: delete rows if needed, but the last 3 rows must be blanks as detailed above
- Save your file
- System → Carousel position → Reference run → OK (moves carousel so that first sample in run will start with #1 slot)
- Remove plastic lid from the carousel and place your standards and samples in the carousel slots from 6–70
- Make sure that none of the tins are leaking!
- Make sure all the tins fit inside the slots easily so they will drop freely into the combustion tube
- Replace the lid for the carousel
- Check the bottom part of the program to ensure the lights are green and that the other parameters appear to be normal
 - Combustion tube = 1150 °C
 - Reduction tube = 850 °C
 - Ads. Column = 40 °C
 - Pressure = ~1200-1300 mbar
 - TCD flow = 200 mL/min
 - He flow = 196 mL/min
 - O2 flow = 6 mL/min

- Click the green icon (“auto analysis”) to start running your samples
- To stop a run, use the red icon (“stop auto analysis”)
- To restart a run, right click the sample No. for the first sample you want to run and click “Current sample”; then click the green icon (“auto analysis”) to restart your run
- When the analysis is done, the instrument will automatically go to “sleep”
- Ensure the instrument is sleeping and then close the gas flows for the two gas tanks
- Before turning off the EA, make sure the combustion tube is at 600 °C and the reduction tube is at 400 °C
- Press the green button on the bottom right of the EA instrument to turn the instrument off
- Open the EA door to allow heat to escape until the EA has fully cooled (because turning the EA off stops the fan which cools the interior)

Exporting your data

- File → Export as csv → Export

Maintenance

The EA has many consumables that need to be replaced fairly frequently:

- Ash finger = every ~60 samples
- Reduction tube = every ~250 samples
- Large drying tube = every ~300 samples
- Combustion tube = every ~2000 samples
- Small drying tube = every ~2000 samples

It is highly recommended to read the MICROvCube_ShortManual before attempting to perform maintenance

The EA is extremely hot while it is on

It is dangerous to perform maintenance while the instrument is on

You must read the comprehensive manual instructions and warnings to perform this maintenance and wear the proper PPE

If the instrument is on, go to Options → Maintenance → Replace parts and follow the instructions (make sure to wait until the pressure is below 700 mbar before performing maintenance)

It is highly recommended to perform maintenance or change the EA mode while the instrument is off and completely cooled

To replace the consumables, see the MICROvCube_ShortManual, page 4 (Mode of operation: CNS/S)

All chemicals must be worked with under the fume hood

In addition to replacing the consumables, sometimes the ball valve will need to be cleaned; this is especially true when leaking samples have been run; to clean the ball valve, see the MICROvCube_Manual, page 228-236 (note: VarioMICRO admin: easadmin; page 97)

Important: do NOT apply grease to any of the components of the EA that lead to the lines, such as O-rings, etc.; the high-vacuum grease is applied very lightly ONLY to the top plugs for the combustion and reduction tubes where the plugs come into contact with the inside of the glass tubes

Fine Leak Test

If the rough leak test does not pass, several common issues can be checked before running the fine leak test:

- Lack of pressure build up? This usually means that either:
 - An O-ring is missing; check all the O-rings associated with any recent maintenance
 - One of the tubes has a crack; check the tubes and try replacing them
- Very minor leak? Try adjusting some of the connections associated with any recent maintenance
- Slow building leak? This is most often due to the ball valve needing cleaned; see the [“Maintenance”](#) section

If none of the above are the cause of the leak, perform a fine leak test

The fine leak test is performed while the instrument is on and fully heated

Warning: Caution during this work! You must read the comprehensive manual instructions and warnings to perform the fine leak test and wear the proper PPE

Options → Diagnostics → Fine leak test → click “ok” → wait for Ads. Column temperature to be cooler than 32 °C → click “Start”

The dialog box will lead you step by step through the fine leak test and helps you to localize the leak

The leak test components kit is required for this operation

Update Record

Prepared By: Cole Gross

Date: November 2019

Update By: Cole Gross

Date: May 2021

Changes: Updated multiple directions and added methods, maintenance, mode, and leak sections

Update By:

Date:

Changes: