Carbon isotope discrimination - part II - gas analysis - v.01

Stephen Driever,Steven Burgess

Abstract

Protocol for measurement of carbon isotope descrimination during photosynthesis.

See the following references for more background:
H. Griffiths (1993) Photosynthesis and Production in a Changing Environment pp 181-1927
Farquhar et al. (1989) Annu. Rev. Plant Physiol. Plant Mol. Bioi. 1989. 40:503-37

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Protocol

Start up the IRMS

Step 1.
Open inlet valve to vacuum (RL)

Step 2.
Switch between Ref and Sample (2x F1) Reference gas

Taking a reference-reference measurement

Step 3.
Purge reference lines
Open "RF"
Open "RP"

NOTES

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Software view. Diagram of valve system, arrows indicate direction of gas flow

Step 4.
Purge reference inlet
Close RF"
Open "RI"

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Software view. Diagram of valve system, arrows indicate direction of gas flow

Step 5.

Isolate the reference lines and prepare for loading reference gas

Close 'RP'

Open 'RF'

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Software view. Diagram of valve system, arrows indicate direction of gas flow

Step 6.

Manually open valve and fill the reference loop with gas

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Make sure the second valve (top of image) is closed so gas only fills the reference loop. Open the first valve (depicted) for ~5s to fill collection loop.

Step 7.

Close the valve to the reference gas

Step 8.

Open the second valve, to let the reference gas from the connector piece into the inlet

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Make sure the first valve is closed, then open up the second valve (depicted) to let reference gas out of collection loop into the mass spec. You should see a change in the beam intensity as gas is detected. Wait until this stabilizes so all the gas has left the loop.

Step 9.

Close the second valve

Step 10.

Repeat steps 6-9 two times

Step 11.

Flood both sides of the device with reference gas

Close "LV" vacuum line

Open "RP"

Open "SP"

Open "SF"

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Software view. Diagram of valve system, arrows indicate direction of gas flow

Step 12.

Isolate valves

Close "RF"

Close "RI"

Close "SF"

Close "SI"

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Software view. Diagram of valve system, arrows indicate direction of gas flow

Step 13.

Check Beam 1

Look at the beam intensity, it should be =< 1e-12. You should expect a value of around 5e-9

Set beam intensity

Step 14.

V, then B : set target to 6e-9 (Enter)

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This is to minimize the possibility of adding technical artifacts to measurements by always analysing gas at ~ the same concentration

Step 15.

Do this for both Sample and Ref side

Step 16.

Manually adjust variable volume if the target beam intensity is below or above 6e-9

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Take small steps! adjusting the volume +/-5 is sufficient in most cases. The intensity will drfit down over time as gas is consumed by the detector so always good to check both sample and reference immediately before measuring

Null reference as first run

Step 17.

D (Data processing) + sample name: “Irefref”

Step 18.
Cold finger: N

Step 19.
Run (make sure to feed new paper to the printer!)

Step 20.
Write values down in lab book

Step 21.
Open LVRun actual samples:

Load sample gas

Step 22.
Go to Sample side (use F1 F1 to switch from Ref to Sample)

Step 23.
Vacuum Sample side
Open "SP"
Open "SF"

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Software view. Diagram of valve system, arrows indicate direction of gas flow

Step 24.
Check Bean 1 intensity => 1e-12

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This is to check that the lines are clean, 1e-12 is the value you get when there is little gas left in the system

Step 25.
Return Variable Volume Sample to 0;
V A (zero) 0

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This is done to reset the bellows for injection of the next sample.

Step 26.
Connect closed sample tube (SI closed)

Step 27.
Vacuum sample inlet
Close "SF"
Open "SI"
(SP already open)

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Software view. Diagram of valve system, arrows indicate direction of gas flow

Step 28.
Pirani gauge will go to 1e-3, wait 10 more seconds

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The Pirani gauge gives an indication of the gas pressure in the inlet loop. A value of 1e-3 means the loop is clean of gas so you are ready to inject.

Step 29.
Isolate from pump
Close "SP"
Close "SI"
Open "SF"

Step 30.
Carefully open collection tube (transducer value will rise)

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Software view. Diagram of valve system, arrow indicates an increase in signal from the Pirani gauge as gas build up behind valve SI

Step 31.

Load sample gas into system

Open 'SI'

Wait for beam 1 intensity to stabilize.

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Software view. Diagram of valve system, arrow indicates direction of gas flow. Beam 1 value will rise (if not, then only water in sample; close sample immediately and vacuum!)

Step 32.

Close off inlet

Close "SI'

Close "SF"

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Software view. Diagram of valve system, arrows indicate direction of gas flow

Step 33.

Adjust Beam intensity: V B 6e-9

Step 34.

Adjust reference beam intensity

Switch to Ref side (F1 F1)

V B 6e-9

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This is done due to drift in reference beam intensity as gas is used

Measure gas

Step 35.

Esc

menu D

sample ID

Cold finger: N

Make sure there is paper in the printer!

Step 36.

Note down values in lab book

Post-measurement

Step 37.

Vacuum Sample side

Open "SF"

Open "SP

Return variable volume Sample to 0; V, A, 0

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Software view. Diagram of valve system, arrows indicate direction of gas flow

End of day

Step 38.

Open all valves on both sides except SI and RI

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This is done to clean out the system ready for the next use. Do not turn off the vacuum pump, it should run continuosly to avoid wearing it out.

Access the latest run data

Step 39.
Esc (exit manual control menu)

Step 40.
F (Filer)

Step 41.
D (Data logger)

Step 42.
Select CO2: E (Edit current template)

Step 43.
Use cursor to scroll through to bottom of page for results (don’t change anything!)

Step 44.
Esc to exit