SOP: Elemental Analysis of Soil Carbon and Nitrogen

Overview:

This standard operating procedure (SOP) describes a protocol for high-temperature (900-1200 °C) combustion of soils to determine total carbon (C) or nitrogen (N) content (%) via the Elementar Vario MAX cube ® (Langenselbold, Germany). Key consumables are aspartic acid, crucibles, quartz wool, zinc, copper, copper oxide, tungsten, corundum balls, cotton, Sicapent ®, wire mesh rondes, spacers, spare tubes, oxygen gas, and helium; and key safety considerations are the use of heat protective gloves (capable of holding \geq 950 °C) when doing maintenance work and manipulating reduction/ combustion/ post-combustion/ drying tubes. Soils that are ground to pass a <2 mm sieve are typically used.



Safety:

All standard safety protocols and online safety training via UIUC <u>Division of Research</u> <u>Safety (DRS)</u> are required.

Personal protection (PPE) for this procedure include:

Body Protection: Laboratory coat

<u>Hand Protection</u>: Nitrile gloves when manipulating samples/ aspartic acid, heat protective gloves (capable of holding \geq 950 °C) when doing maintenance work and manipulating reduction/ combustion/ post-combustion/ drying tubes

Instrumentation & Consumables:

Sample preparation (blanks, run-ins, aspartic acid and soils)

- Crucibles (steel)
- Aspartic acid (C₄H₇NO₄, MW: 133.11)

- Analytical balance (three decimal places sensitivity)
- Spatula

Maintenance work (replacing parts)

I. Reduction tube

- Corundum balls
- Wire mesh ronde
- Wire mesh separators (10)
- Tungsten (MW: 183.84)
- Copper oxide (CuO, MW: 79.54)
- Copper (MW: 63.55)
- Quartz wool
- Zinc (MW: 65.38)

II. Combustion tube

- Spacer
- Wire mesh ronde
- Corundum balls
- Copper oxide (CuO, MW: 79.54)

III. Post-combustion tube

- Spacer
- Wire mesh ronde
- Copper oxide (CuO, MW: 79.54)
- Platinum catalyst
- Quartz wool

IV. Drying tubes

- Cotton
- Sicapent ®

Detailed Procedure:

I. C/N determination

1. Sample preparation

*Note that liquid samples could be run, but this protocol focuses on C and N measurement in (solid) soil samples

- 1.1. Place three empty crucibles on the position of 1-3 of the sample plate. They will be used as **blank** samples.
- 1.2. Measure ~ 50 mg of aspartic acid into three crucible and place them in sequence after the blanks. They will be used as **run-in** samples. Record the soil mass (mg) of each sample as it will need to be entered into the sample sheet.
- 1.3. Measure ~ 50 mg of aspartic acid into three crucibles and place them in sequence after the run-ins. They will be used as **aspartic acid** samples. Record the soil mass (mg) of each sample as it will need to be entered into the sample sheet.
- 1.4. Measure ~ 250-500 mg of dried soils into each crucible and place them in sequence after the aspartic acid samples (higher end ~350-500 mg recommended for samples low on N). Record the soil mass (mg) of each sample as it will need to be entered into the sample sheet.

Note: The photo below shows the blank, run-in, and aspartic acid samples on the sample plate. At the beginning of the experiment, and every 24 hours, one must run 3 blanks, 3 run-in's, and 3 aspartic acid's, before running soil samples.



2. Software settings

- 2.1. Open the EAS Vario Max Cube program in the desktop.
- 2.2. File > New (a new empty sample sheet* will appear) to create a new project:



*Where Hole Pos. corresponds to the position in the sample plate (marked by numbers, can be modified manually), Weight is the mass in mg of the corresponding sample, Name is the arbitrary label of the corresponding sample, and Method is the corresponding measuring technique for each kind of sample (e.g., **blank [O2]** for blanks, **aspartic acid 1** for run-in's and aspartic acid's, and **soil** for soil samples).

- 2.3. Set up the first 3 rows to run blanks.
 - Under Weight [mg], manually type 1.00 (this is an arbitrary value just for the software to accept the sample, the crucible remains empty)

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Γ	No.	Hole Pos.	Weight [mg]	Name	Method
į	💵 1	1	1,00	Blnk	blank [O2]
	2	2	0.00		
1	2				

• Under Name, select "Blnk"

No.	Hole Pos.	Weight [mg]	Name		Me
1	1	0.00	Blnk		
2			Windsor	~	
3			G3-7		
4			G3-6		
5			G3-5		
			G3-3		<u> </u>
			G3-2		<u> </u>
/			aspartic acid		
8			Runin		
9			Bink		
10			lest alutamic acid		
11			giutamic acid	~	<u> </u>
			func acia	•	

• Under Method, select "blank [O2]"

1 🞽	🗟 🖨 🚨	50 2 10	r 🖪 🖻 🔍	C 💮 🖻 ≫ 4 → 4
No.	Hole Pos.	Weight [mg]	Name	Method
🐠 1	1	0.00	Blnk	blank [O2] 🔹
2	2	0.00		blank [O2] 🔥
3				butter
4				cereals
5				cneese
6				fishmeal
7				glutamic acid
8				milk
9				milk powder
10				plant
11				sausage 🗸 🗸
12				

2.4. Set up the next 3 rows for RunIn

- Type the weight in mg of each corresponding run-in sample (~50 mg)
- Under Name, select "RunIn"
- Under Method, select "aspartic acid 1" *Note that there are three different aspartic acid methods (aspartic acid 1, aspartic acid 2, and aspartic acid 3). The differentiation between these is based on the weight [mg] of aspartic acid to use (aspartic acid 1 for 0-120 mg, aspartic acid 2 for 120-600 mg, and aspartic acid 3 for 600-1100 mg). In our case, as we are using ~50 mg of aspartic acid, the corresponding method is **aspartic acid 1**.
- 2.5. Set up the next 3 rows for aspartic acid
 - Type the weight in mg of aspartic acid (~50 mg) in each corresponding sample
 - Under Name, select "aspartic acid"
 - Under Method, select "aspartic acid 1"

- 2.6. Set up the following rows for soils
 - Type the weight in mg of soils in each corresponding sample (~250-500 mg, recommended)
 - Under Name, type the name ID of each soil sample
 - Under Method, select "soil"

At the end of these steps, the overall data sheet must look like this:

10000	C)		5 PA		4 (G (B ×	4 4 4	1 im (0										
N	lo.	Hole Pos.	Weight [mg]	Name	Method	N Area	C Area	N [%]	C [%]	N Factor	C Factor	N Blank	C Blank	C/N ratio	Protein [%]	Protein Factor	Moisture [%]	Memo
•	1	1	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
٠	2	2	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	(C
•	3	3	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
•	4	4	50.00	Runin	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
0	5	5	50.00	Runin	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	(
•	6	б	50.00	Runin	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
•	7	7	50.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
•	8	8	50.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
٠	9	9	50.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
•	10	10	250.00	S1	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
9	11	11	250.00	S2	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	ł,
•	12	12	250.00	\$3	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
٠	13	13	250.00	S4	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
•	14	14	250.00	\$5	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	ł
•	15	15	250.00	S6	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
•	16	16	250.00	S7	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
	17	17	250.00	S8	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
•	18	18	250.00	S9	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1
•	19	19	250.00	S10	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	1

*Note that every **20-25** samples, must run one or two blanks (empty) and an aspartic acid sample:

No.								a second second	No. AND						10		_
	Hole Pos.	Weight [mg]	Name	Method	N Area	C Area	N [%]	C [%]	N Factor	C Factor	N Blank	C Blank	C/N ratio	Protein [%]	Protein Factor	Moisture [%]	Memo
1	1	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
2	2	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
3	3	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
4	4	50.00	Runin	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
5	5	50.00	Runin	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
6	б	50.00	Runin	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
7	7	50.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
8	8	50.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
9	9	50.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
10	10	250.00	S1	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
11	11	250.00	52	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
12	12	250.00	\$3	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
13	13	250.00	S4	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
14	14	250.00	S5	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
15	15	250.00	S6	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
16	16	250.00	S7	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
17	17	250.00	S8	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
18	18	250.00	S9	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
19	19	250.00	S10	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
20	20	250.00	\$11	soil	0	0	0.000	0.000	1 0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
21	21	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
22	22	1.00	Bink	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
23	23	50.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
24	24	250.00	\$12	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
25	25	250.00	\$13	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	
26	26	250.00	S14	soil	0	0	0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00	

3. Sample running

*Note that when not in operation, the machine stays in a sleeping mode:

37								
Process:		Detector:				Temper	atures [ºC]:	
Sleeping	TC detect.	1487			Combust.		899111111	
	TCD temp.	59.7 °C			Post comb.		898111111	
					Reduct.		8241111	
Maintenance					Column 1	11	31	
reduction tube					Column 2	11	31	

To run samples:

3.1. Click on the moon or the clock symbol to wake up the machine:

F	ile E	dit	View	System	Math.	Statistics	Calibratior	Options	Win	dows H	Help			
	1 🖆	ž 🔒	۵ 🖨	19 (2)	¥ 🗈	r 19	C 💮	B % 4		$\Leftrightarrow \Rightarrow$	0		0	
	No.	Ho	le Pos.	Weigh	it [mg]	Name	м	ethod		N Area	C Area	N [%]	C [%]	N Fa
	1		1		1.00	Plak	bl-	ank [O2]		(0	0.000	0.000	1 (

3.2. A new window will appear. Click on the "Wake up now" button.

00	v	v	0.0000	0.000	0.2000	0.0	v
00	0	0	0.0000	0.000	6.2500	0.0	0
00	Slee	n / Wake u	n ontions			0.0	0
00	Jice	p / mane a	populous			0.0	0
00	5	Bleeping				0.0	0
00			deactivated			0.0	0
00						eep 0.0	0
00		Sleeping	at end of sa	mpies		0.0	0
00	(Sleeping	at sample No	p.:	0	0.0	0
00	6	Cut off c	arrier gas			0.0	0
00	1	Reduce o	wen 1 temn	• • • • • • • • • • • • • • • • • • • •		0.0	0
00			wen i temp.			0.0	0
00	l	Reduce of	oven 2 temp.	: <u>0</u> ℃		0.0	0
00	[Reduce o	oven 3 temp.	: 0 °C		0.0	0
00						0.0	0
00	1	Vake up				0.0	0
00		Time: 12:0	AM 🖨			0.0	0
00					Wał now	(e up 0.0	0
00	[Date: 1/1/2	2000 🜩			0.0	0
00	(One-time	e wake up at	date/time ment	ioned above	0.0	0
00	(🔿 Daily wal	ke up at time	mentioned abo	ve except on:	0.0	0
00	1		Tu Dw	е Пть Г	∣Fr □Sa □	l su 0.0	0
00						0.0	0
00	[Continuo	us run after	wake up if samp	oles are available	0.0	0
00						0.0	0
00					OK Car	ncel 0.0	0
00	L		0.0000	0.000	0.2500	0.0	0
							_

*Note: before clicking the Wake up now button, make sure that the <u>Sleeping at the end of</u> <u>samples, Cut off carrier gas, and One-time wake up at [...]</u> settings are selected, as displayed above. This way the machine will automatically go to seep when it finishes running the last samples. This is convenient especially when samples are left running overnight.

3.3. Wait for the process to be on Standby, and the temperature, flow, and pressure bars to be green colored (before running samples).

 37												×	_
Process: Standby	TC detect. TCD temp.	Detector: 0 59.8 °C			Combust. Post comb Reduct.	Temperature 	:s [°C]: 		MFC He O2/He He	Flow [ml/min]: 		Press [mbar]: Input	Delta [mbar] +92
Maintenance					Column 1 Column 2	30 30							

3.4. To start – automatically – running samples (auto-analysis, e.g., setting the machine to run from the first to the last sample), click on the long green button in the toolbar.

Fi	ile E	dit View	System Math.	Statistics Calibrat	tion Options Win	dows H	elp						
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Γ	No.	Hole Pos.	Weight [mg]	Name	Method	N Area	C Area	N [%]	C [%]	N Factor	C Factor	N Blank	С
•) 1	1	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	
4	2	2	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	
4	3	3	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	
4	4	4	50.00	Runin	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	
4	5	5	50.00	Runin	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	

OR, if you want to run a single sample, click on the short green button (this will only run the current sample).

File Edit View System Math. Statistics Calibration Options Windows Help

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Γ	No.	Hole Pos.	Weight [mg]	Name	Method	N Area	C Area	N [%]	C [%]	N Factor	C Factor	N Blank C
	1	1	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0
•	2	2	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0
4	3	3	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0
4	4	4	50.00	RunIn	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0
4	5	5	50.00	RunIn	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0

*Note: the short red button can be used to stop the auto-analysis, as well as the singleanalysis.

I	File	Edit	t View	System Math.	Statistics Calibra	tion Options Win	dows H	elp		_	_			
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Γ	N	lo.	Hole Pos.	Weight [mg]	Name	Method	N Area	C Area	N [%]	C [%]	N Factor	C Factor	N Blank	CE
	•	1	1	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	
	•	2	2	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	
	•	3	3	1.00	Blnk	blank [O2]	0	0	0.000	0.000	1.0000	1.0000	0	
	•	4	4	50.00	Runin	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	
	•	5	5	50.00	RunIn	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	

Once the analysis is finished, the machine will go back to sleeping mode if the <u>Sleeping</u> <u>at the end of samples</u> option was selected. If not, one can manually turn on the sleeping mode by clicking one more time in the moon symbol at the toolbar, and then clicking the "Sleep now" button in the appearing window:

	~	~	0.0000				
000	0	0	0.0000	0.000	6.2500		0.00
000	0	0	0.0000	0.000	6.2500		0.00
000	Slee	n / Wake i	in ontions				0.00
000	Sicc	p/ Wake a	ip options				0.00
000	S	leeping					0.00
000	(deactivated				0.00
000						eep ow	0.00
000	,	Sleeping	at end of sa	mpies			0.00
000	(Sleeping	at sample N	D.:	0		0.00
000	6	Cut off o	arrier gas				0.00
000	ſ	Reduce	oven 1 temp	• 0 ec			0.00
000			oven i tempi				0.00
000	L	Reduce	oven 2 temp.	: 0 °C			0.00
000	[Reduce	oven 3 temp.	: 0 °C			0.00
000							0.00
000	V	Vake up —					0.00
000	1	ime: 12:0	0 AM 🗘				0.00
000						ke up /	0.00
000	L	Date: 1/1/	2000 🖵				0.00
000	(One-time	e wake up at	date/time men	tioned above		0.00
000	(🔵 Daily wa	ke up at time	mentioned abo	ove except on:		0.00
000	ſ	Mo.]⊤u, ⊡w	e. 🗌 Th. 🗆	Fr. Sa.	Su.	0.00
000	-		,				0.00
000	[Continue	ous run after	wake up if sam	ples are available		0.00
000				_			0.00
000					OK Car	ncel	0.00
000			0.0000	0,000	0.2500		0.00

*Note: to save the project and retrieve it later for further work directly in the Vario Max Cube ® software: File > Save As > type a name. Then to open it: File > Open.

4. Exporting data

- 4.1. Export/Import > Export as CVS (for Excel)
- 4.2. A new window will appear. Click on OK.



A	utoSave 💿		୨• ୯ [,]		Rosa_M	P_3NASAhal	fSD_4NA • Si	aved +			<u>ب</u>	Search (Alt+Q)							·
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1	NO.	HOIE POS. W	veignt (r Name	Method	N Area	C Area	N [%]	C [%]	N Factor	C Factor	N Blank	C Blank	C/N ratio	Protein [%]	Protein Factor	Moisture	Memo	Into	Date Time	
2	1	1	1 BINK	blank [O2]	453	208	0	0	1	1	45:	208	0	0	6.25	0			16.09.2021 1	/:32:16
3	2	2	1 BINK	blank [O2]	105	1/16	0	0	1	1	10:	1/16	0	0	6.25	0			16.09.2021 1	/:39:54
4	3	3	1 BINK	Diarik [U2]	100	138	10 553	25.000	1	1	100	138	2 4000	0	0.25	0			16.09.2021 1	1:47:32
0	4	4	61 Runin	aspartic acid 1	13474	50974	10.552	35.880	1	1			3.4009	05.948	0.25	0			16.09.2021 1	1:58:24
0	5	5	55 Runin	aspartic acid 1	13803	51208	10.475	35./1/	1	1			3.4098	05.408	6.25	0			16.09.2021 1	5:09:20
-	0	0	50 Rumm	aspartic acid 1	12830	4/042	10.679	30.430	0.0703	0.074			3.412	00.743	6.25	0			16.09.2021 1	5:20:10
0	/	/	04 aspartic acid	aspartic acid 1	100/1	60460	10.52	30.00	1.0106	1.0000			3.4278	65.75	6.25	0			16.09.2021 1	0:51:04
9	0	0	75 aspartic acid	aspartic acid 1	10/30	47701	10.52	30.00	1.0100	1.0069			3.4278	65.75	6.25	0			16.09.2021 1	0:41:39
10	3	10	31 aspartic aciu	aspartic acid 1	12023	4//91	10.52	1 467	1.0075	1.0002			3.4270	05.75	6.25	0			16.09.2021 1	0.01.00
12	10	10	244 SNA-1	soil	755	9597	0.116	1.407	0.9901	0.9904			12.4506	0.750	6.25	0			16.09.2021 1	01:55
12	11	11	249 3NA-2	soll	733	9030	0.115	1.475	0.9961	0.9964		0	12.7777	0.721	6.25	0			16.09.2021 1	9:10:26
10	12	12	202 SINA-5	soil	701	9633	0.114	1,454	0.9901	0.9904			12.3765	0.715	6.25	0			16.09.2021 1	2.13.15
14	15	15	233 SINA-4	son	/14	5517	0.11	1.422	0.9901	0.9904			12.5615	0.085	0.23	0			10.09.2021 1	.27.35
15	14	14	201 3NA-5	soli	800	10511	0.122	1.538	0.9961	0.9964		0	12.0457	0.76	6.25	0			16.09.2021 1	9:30:47
10	15	15	249 3NA-0	soli	645	8/22	0.101	1.332	0.9961	0.9964		0	13.2259	0.629	0.25	0			16.09.2021 1	9:45:37
17	10	10	205 3NA-7	soli	033	8/18	0.093	1.251	0.9961	0.9964			13.4730	0.58	0.25	0			16.09.2021 1	9:54:20
18	1/	1/	259 3NA-8	soli	000	8490	0.099	1.240	0.9961	0.9964			12.5375	0.021	0.25	0			16.09.2021 2	1:03:18
19	18	18	252 3NA-9	son	013	81/5	0.094	1.231	0.9961	0.9964			13.0440	0.59	0.25	0			16.09.2021 2):12:00
20	19	19	275 3NA-10	son	082	8030	0.097	1.194	0.9961	0.9964			12.3407	0.005	0.25	0			16.09.2021 2	0:20:55
21	20	20	259 3NA-11	son	/5/	10297	0.115	1.51/	0.9961	0.9964			13.21/2	0.718	0.25	0			16.09.2021 2):29:40
22	21	21	200 SINA-12	son	004	9490	0.102	1.419	0.9901	0.9964			13.9727	0.035	0.25	0			16.09.2021 20	1:38:30
23	22	22	275 3NA-13	son	009	9102	0.095	1.208	0.9901	0.9964			13.300	0.593	0.25	0			16.09.2021 20):47:28
24	23	23	209 3NA-14	son	700	0074	0.097	1.25	0.9901	0.9904			12.9327	0.004	0.25	0			16.09.2021 2	050:22
25	24	24	265 3NA-15	son	723	9374	0.099	1.200	0.9901	0.9904			12.01/1	0.021	6.25	0			16.09.2021 2	1:05:15
20	25	25	209 SNA-10	son	700	100/1	0.115	1.010	0.9901	0.9904			13.1859	0.718	6.25	0			16.09.2021 2	1:14:09
27	20	20	257 SNA-17	soli	653	9038	0.101	1.341	0.9901	0.9904			13.3144	0.03	6.25	0			16.09.2021 2	1.23:03
20	27	27	203 SNA-16	soil	603	0003	0.097	1.250	0.9901	0.9904			12.9001	0.004	6.25	0			16.09.2021 2	1.51.56
29	20	20	274 SNA-19	soil	740	9069	0.097	1.205	0.9901	0.9904			12.9735	0.008	6.25	0			16.09.2021 2	1.40:52
30	29	29	201 3NA-20	SOII	12214	9471	10.52	1.382	1.0157	0.9964			12.4315	0.695	6.25	0			16.09.2021 2	1:49:43
31	30	30	53 aspartic acid	aspartic acid 1	13214	49409	10.52	30.00	1.0157	1.0105		0	3.4278	65.75	6.25	0			16.09.2021 2.	2:00:43
32	31	31	275 SINA-21	soil	792	11087	0.110	1.503	1.0157	1.0105			13.5327	0.722	6.25	0			16.09.2021 2	
33	32	32	204 SINA-22	soil	642	9210	0.104	1.4	1.0157	1.0105		, 0	13.4/4/	0.649	6.25	0			16.09.2021 2	
34	33	33	205 3NA-25	soil	643	9108	0.101	1.39	1.0157	1.0105		, 0	13.7928	0.63	6.25	0			16.09.2021 2	
30	34	34	203 3NA-24	soil	053	8009	0.099	1.262	1.0157	1.0105		, 0	12.8055	0.010	6.25	0			16.09.2021 2	
30	35	30	204 3INA-20	soil	/11	92/4	0.107	1.357	1.0157	1.0105		, 0	12.0324	0.0/1	6.25	0			16.09.2021 2	
37	30	30	280 3INA-20	soil	888	12406	0.125	1.085	1.0157	1.0105		, 0	13.4519	0.783	6.25	0			16.09.2021 2	
38	37	3/	203 3INA-27	son	/34	10151	0.116	1.553	1.0157	1.0105		0	13.3872	0.725	6.25	0			10.09.2021 2	5:03:17
		Rosa_MP	_3NASAhalfSD_4NA	mastersheet	+											1				

4.3. Once saved, the Excel sheet must look like this:

II. Maintenance work

1. Checking/Replacing the reduction tube

The reduction tube must be replaced once all layers of tungsten are expanded and burned (e.g., change of color to black with yellow spotting). For soil analysis, this is thought to happen each ~100-120 samples. Periodic checking of the status of tungsten in the reduction tube, in between a determinate number of samples (~30-50 samples), is recommended.

- 1.1. Options > Maintenance > Replace parts
- 1.2. A new window will appear. Read the caution notice and click on Next.

CAUTION!!! Hot Parts!			00	
2			DO	Γ
			DO	Γ
		CAUTION!!!	00	Γ
	Risk of burr	ices! 00	Γ	
	Use	protective gear!	00	Γ
			00	Γ
		Next	00	Γ
		0.2500		Γ

1.3. A new window will appear. Remove the sample cover and click on OK.



1.4. A new window will appear. Please wait for a moment (1) until the next window (2) shows up. **DO NOT** click on finished **until** you have placed the reduction tube back, re-assembled the core and placed back the sample cover (see details for this below).

Replace par	l) 10000 rt	6.2500	(2) 0 0000 0.0000 0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.0000	DI DI
Ĵ	Please wait for a moment		Part can now be replaced. To continue after the replacement click to 'Finished'.	
	Finished	6.2500	Finished 0.0000 0.000 6.2500 0.0	

- 1.5. Once the "Part can now be replaced [...]" window is up, you may open the front door of the hardware and start disassembling the core.
- 1.6. Remove all clamps and metal connections from the core (see picture below, yellow marked = remove).



<u>TOP</u>

Remove all clamps and metal connections.

BOTTOM

Remove this clamp **only**.

*Note: a total of 8 clamps (7 regular, 1 small) and 3 metal connections must be removed (see picture on the right).



1.7. Careful not to drop anything into the combustion tube, remove the screws at the sample plate.



1.8. Carefully pull the core towards the front by grabbing it from the bottom part.



1.9. Carefully take out the reduction tube (third from left to right) and place it in a steel rack (use of heat protective gloves is mandatory, the tube is at > 900°C).



1.10. Insert back the new (if replacing) or current (if just checking) reduction tube, and re-assemble all parts (clamps, metal connections and screws). Make sure to check that the O-rings of every connection are still in place.

*Note: the metal connections are **color-coded** corresponding to their position (red, black, white).



After re-assembling, the core must look like this:



1.11. Once finished checking/replacing the reduction tube and reassembled all parts, put back the sample cover (the light should be on once the cover is back to place) and click on finished.



- 1.12. After replacing (e.g. inserting a **new** reduction tube)
 - Options > Maintenance > Intervals
 - A new window will appear. Manually type 0 (zero) in the "Standing" text box. This will reset the count of the samples analyzed by the current (in-use) reduction tube.

0.000	0.000	1 0000	1 0000	0	0	0.0000	0.000	6 2500	0.00			
0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6 2500	0.00			
0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00			
0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00			
0.000	0.000	Mainte	nance inter	vals						-	_	
0.000	0.000	_				_					_	
0.000	0.000	reduc	tion tube			Events			Testernuel	Internal Observing		
0.000	0.000	combu	ustion tube			Event:	7		Interval	standing		
0.000	0.000	post o	combustion t	ube		reducti	on tube		75	5 119		
0.000	0.000	drying) tube "N" (lo	ing)		Mode as	signments					
0.000	0.000	drying	g tube "N" (si	hort)			-					
0.000	0.000	crucib	le container									
0.000	0.000						IS					
0.000	0.000						iss					
0.000	0.000						I/CNS		If becoming	due:		
0.000	0.000						C/Solid		Indication	on only		
0.000	0.000					🗹 ТО	С			on and abort		
0.000	0.000					🗌 ТО	C/CNS		the auto	run		
0.000	0.000											
0.000	0.000											
0.000	0.000											
0.000	0.000					Only	with IR					
0.000	0.000											
0.000	0.000	Total		197		Ne	ew D	elete Sa	ive	Close		
0.000	0.000	Total:		172								
0.000	0.000	1,0000	1.0000	0	0	0.0000	0.000	6 2500	0.00			
0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	0.2000	0.00			
0.000	0.000	1.0000	1.0000	0	0	0.0000	0.000	6.2500	0.00			

*Note that this window also serves to check how many samples have been run with the current reduction tube. This serves as a notice to be aware if - and when checking/replacing may be needed.

2. Packing a new reduction tube

To prepare a new reduction tube, a series of compounds must be added into the corresponding tube, using a funnel and a metered ruler (follow the instructions depicted below, these may also be found in the short manual and in a separate sheet near the machine).



*Note that the combustion, post-combustion, and drying tubes may also need replacing. However, their lifetime is considerably greater than the one of the reduction tube. Therefore, their replacement will not happen very often. If needed, please see the instructions depicted below (these may also be found in the short manual and in a separate sheet near the machine).





3. Leak check

- 3.1. When should a leak check be performed:
 - Significant increase/decrease of the daily factor (the value of daily factor should be around 1 ±0.01)
 - Blanks with high nitrogen values
 - Decrease of the pressure values "Inlet" and "Outlet".
- SOP: C/N determination by high-temperature combustion | UIUC Soils Lab | Last revised 21 July 2021

- Too high helium flow at the "Inlet".
- Too low helium flow at the "Outlet".
- 3.2. Performing a leak test to find the leak
 - Click Options > Diagnostics > Leak test.
 - If the leak test did not pass, the dialog leads you step by step through the leak test and helps you to localize the leak.
 - The leak test components kits (supplied with the analyzer) is required for this option.

References:

Short manual of vario Max cube. Elementar.

Suggested reading:

Tabatabai, M. A. and Bremner, J. M. 1990. "Automated instruments for determination of total carbon, total nitrogen, and total sulfur in soils by combustion techniques". In Soil Analysis: Modern Instrumental Techniques, , 2nd Edn., Edited by: Smith, K. A. 174–203. New York, NY: Marcel Dekker. [Google Scholar]

Yeomans, J. C., and J. M. Bremner. "Carbon and nitrogen analysis of soils by automated combustion techniques." *Communications in Soil Science and Plant Analysis* 22.9-10 (1991): 843-850. <u>https://doi.org/10.1080/00103629109368458</u>

Citation:

SOP: Elementar C/N Analysis. 2021. Soils Lab, University of Illinois Urbana-Champaign. Urbana, IL. Accessed at: <u>https://margenot.cropsciences.illinois.edu/methods-sops/</u>

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