VarioMax Cube CN SOP

USDA[1]
[1]USDA

Apr 05, 2021

1 Works for me This protocol is published without a DOI.

PDI Test

Josh Birlingmair
USDA-ARS

ABSTRACT

VarioMax Cube CN SOP.

PROTOCOL CITATION

USDA 2021. VarioMax Cube CN SOP. **protocols.io**
https://protocols.io/view/variomax-cube-cn-sop-btx4npqw

KEYWORDS

VarioMax, Cube, CN

CREATED

Apr 05, 2021

LAST MODIFIED

Apr 05, 2021

PROTOCOL INTEGER ID

48860

System Startup

1 If the VarioMax is sleeping, the temperature and pressure status view should be as shown below:

Zero input and output pressure, and zero He and O2 flow. Column temperatures should be:

- Combustion: 900C
- Post-combustion: 900C
- Reduction: 830C

2 **Set gas pressures:**

2.1 Verify He cylinder pressure > 700psi, output pressure 380kPa (3.8bar)

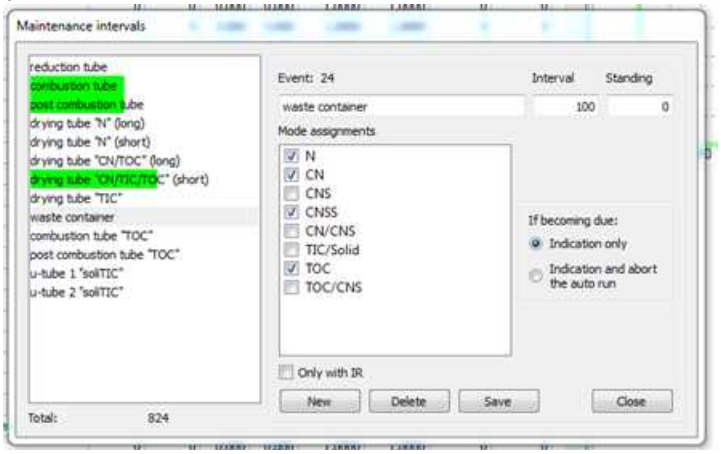
2.2 Verify O2 cylinder pressure > 100psi, output pressure 250kPa (2.5bar)

Empty crucible container:

3

3.1 Reset crucible maintenance interval under the menu command:

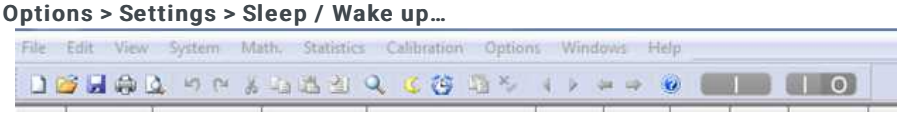
Options > Maintenance > Intervals



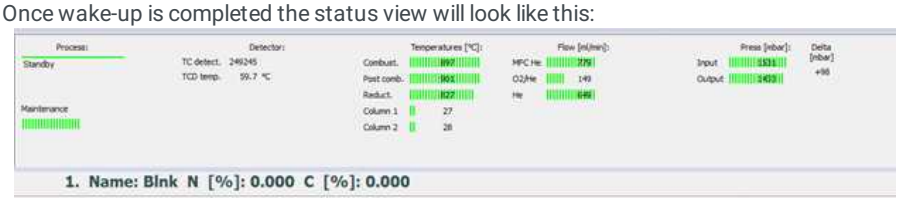
4 **Wake up instrument:**

4.1 Click on the wake-up button (clock) on the main toolbar or under the menu command:

Options > Settings > Sleep / Wake up…



4.2 Once wake-up is completed the status view will look like this:



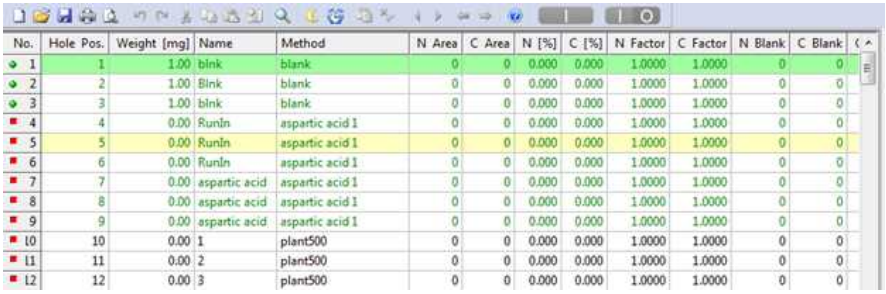
Sample Prep and Sample Run

5 **Enter sample names and weights:**

5.1 Select the appropriate template for the type of sample to be analyzed: soil or plant

- Plant template MD
- Soil template MD

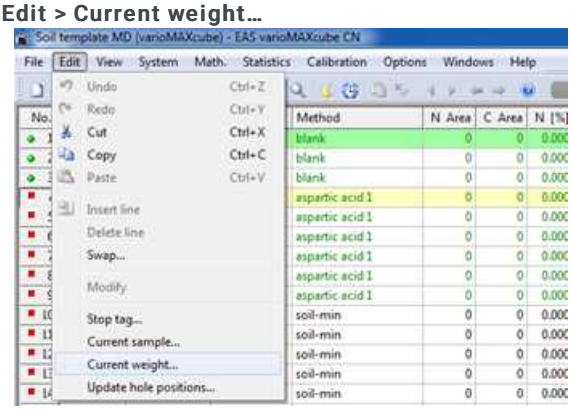
5.2 A typical sample run will contain 3 blnk, 3 RunIn, and 3 standards (aspartic acid) at the beginning of the run and a check standard every 20 samples. A reduction column can accommodate a run of approximately 120 total samples, including standards etc.



No.	Hole Pos.	Weight [mg]	Name	Method	N Area	C Area	N [%]	C [%]	N Factor	C Factor	N Blank	C Blank
1	1	1.00	blnk	blank	0	0	0.000	0.000	1.0000	1.0000	0	0
2	2	1.00	Blnk	blank	0	0	0.000	0.000	1.0000	1.0000	0	0
3	3	1.00	blnk	blank	0	0	0.000	0.000	1.0000	1.0000	0	0
4	4	0.00	RunIn	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0
5	5	0.00	RunIn	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0
6	6	0.00	RunIn	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0
7	7	0.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0
8	8	0.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0
9	9	0.00	aspartic acid	aspartic acid 1	0	0	0.000	0.000	1.0000	1.0000	0	0
10	10	0.00	1	plant500	0	0	0.000	0.000	1.0000	1.0000	0	0
11	11	0.00	2	plant500	0	0	0.000	0.000	1.0000	1.0000	0	0
12	12	0.00	3	plant500	0	0	0.000	0.000	1.0000	1.0000	0	0

5.3 Click on the corresponding Name cell to input the sample name. Assign Blanks a mass of 1. Every other sample and standard must have a non-zero mass or the sample run will stop when the current sample (green highlighted line) reaches a row with a red box next to the row number. Sample weights will be transferred directly from the balance to the yellow highlighted line in the sample spreadsheet when the “print” button on the balance is pressed. To select the row for weight transfer, use the menu command:

5.4 **Edit > Current weight…**



6 Save sample log:

6.1 Save sample spreadsheet with identifying name. For example:
Plant template MMDDYY Name Surname

6.2 **Do not change the location where this file is saved.** A copy of the file can be exported to another directory as described the Data Export section.

7 Load crucibles in sample carousel:

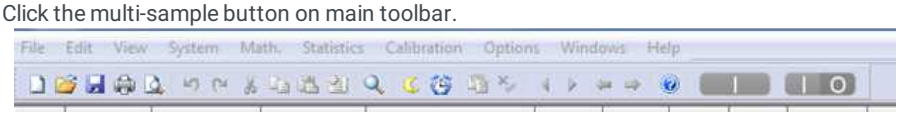
7.1 Carefully lift plastic carousel cover a few inches and slide off carousel. Note that removal or misalignment of the cover triggers a software warning.

7.2 Place crucibles in corresponding carousel position, according to sample log Hole Pos.

7.3 Replace carousel cover.

8 **Start sample run:**

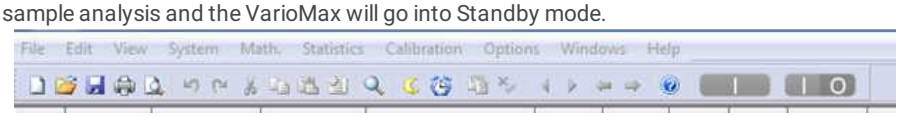
8.1 Click the multi-sample button on main toolbar.



Mid Run Maintenance

9 **Recovering crucibles:**

9.1 Click the single-sample button on the main toolbar. The sample run will pause at the end of the current sample analysis and the VarioMax will go into Standby mode.



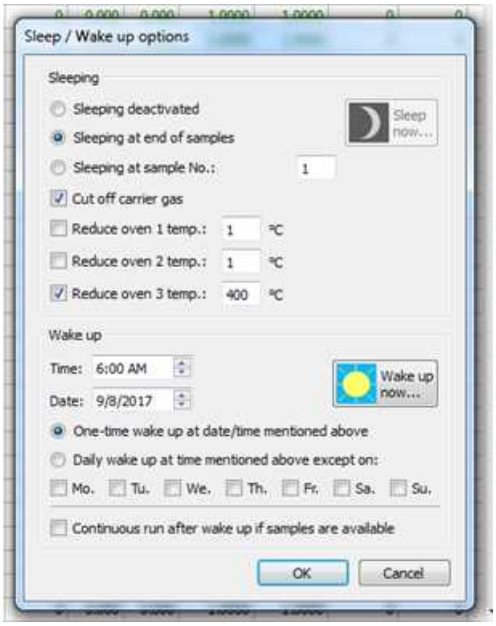
9.2 Recover empty crucibles as described in the System Startup section.

9.3 Restart the sample run by clicking the multi-sample button on the main toolbar.

10 Changing helium cylinder

10.1 Click the single-sample button on the main toolbar. When the sample completes, put the VarioMax to sleep by clicking the sleep button (moon) on the main toolbar. The Sleep /Wake up options dialog will open:

1. **Un-check all 3 checkboxes** to Reduce oven temp
2. Click Sleep now… button



10.2 Once the pressures and flows have dropped to zero you can change the He cylinder.

10.3 Now Wake-up the VarioMax using the menu command

Options > Settings > Sleep /Wake up…

1. **Re-check** the checkboxes to Reduce oven temp so that the corresponding column temperatures will be reduced at the end of the sample run.
2. Open the gas cylinders and set pressures to the following values [He: 3.8bar, O2: 2.5bar]
3. Click Wake up now… button

10.4 When pressures and flows have reached the appropriate levels, restart the sample run by clicking the multi-sample button on the main toolbar.

Data Export

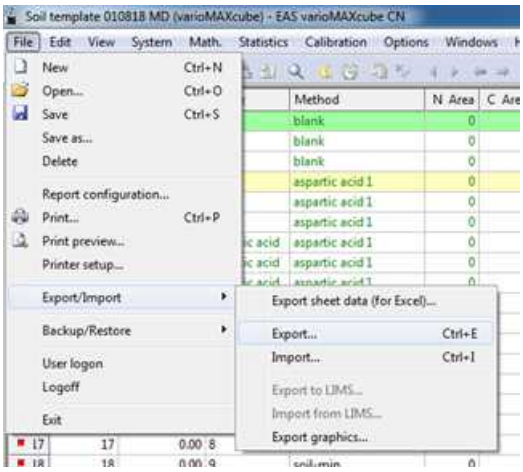
11 **Export analysis results:**

There are 2 options for data export: sqlite database file and csv

11.1 To export as sqlite database file: **File > Export/Import > Export…**

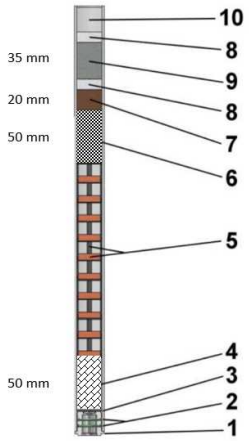
11.2 To export as csv: **File > Export/Import > Export sheet data (for Excel)…**

11.3 Export the results to the folder corresponding to supporting researcher.
…\Documents\Vmax\data\ [Baker, Feyereisen, Rice, Spokas, Venterea]



Filling the Reduction Tube

12 The following picture shows the fillings and filling heights of the reduction tube:



Legend:

1 Plug, bottom
2 O-ring (20 x 2)
3 Wire mesh ronde (Ø 22 mm)
4 Hollow corundum balls (50 mm, 18 g) [#S50 008 467]
5 Spacer (11 x), tungsten granulate (10 x 18 g) [#S12.01-0045]
6 Copper oxide (fine, 0.5 mm) (50 mm, ca. 60 g) [#S05 001 039]
7 Copper (wire shaped) (20 mm, ca. 25 g) [#S05 000 699]
8 Quartz wool (10 mm)
9 Zinc (35 mm ca. 55 g) [#S29.00-0092]
10 Plug with o-Ring (20 x 2) and quad ring

Working temperature 830 °C

Fill the reduction column by the end of the day so that it is ready to be swapped the following morning. For more details on reduction column filling see pages 265-267 of the VarioMax manual.