Xi'an Jiaotong-Liverpool University
西交利物浦大学

General Operating Procedure for:

Multi N/C 3100

Department of Health and Environmental Sciences

By Yili Cheng

Contents

Liquid part

I. General notes on your analyses

1. Dilute your samples if it is strongly acidic.
2. Use clean, non-particulate glass containers to prepare and storage your samples.
3. Take the following precautions to minimize the influence of laboratory air when preparing and storing solutions in the range of < 1mg/l:
   - Keep the empty space above the liquids as small as possible;
   - Cover the sample cups on the sample tray with film (white side up).
   - Remove the source of organic vapors.
4. Prepare no less than 20ml sample.

II. Standard solution

1. TIC 1000 ppm (mg/l): 4.41625 g Na_2CO_3 and 3.5g $NaHCO_3$ dissolved in 1000ml ultra-pure water.
2. TOC 1000 ppm (mg/l): 2.1254 g potassium hydrogen phthalate ($C_8H_5KO_4$) dissolved in 1000ml ultra-pure water.
3. TN 1000 ppm (mg/l): 3.61098 g KNO3 and 2.35925g $(NH_4)_2SO_4$ dissolved in 1000ml ultra-pure water.
4. Dilute them to the concentration you need.

III. Switching on the analyzer

1. Make sure the waste hose is connected to a suitable waste container.
2. Open the O2 valve, if the total O2 pressure is less than 3MPa, contact Yili or Xiao.

3. Sufficient phosphoric acid is available in the reagent bottle (0.5ml for each TIC detection).
4. Sufficient ultra-pure water is available.
5. Make sure halogen trap is usable.(the color of copper and brass wool didn't change.)

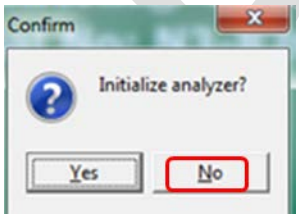

6. Make sure the analyzer is not connected with external solids module.

7. Open the valve at the pressure reducer of the gas supply) and make sure the preliminary pressure is 4 to 6 bar.

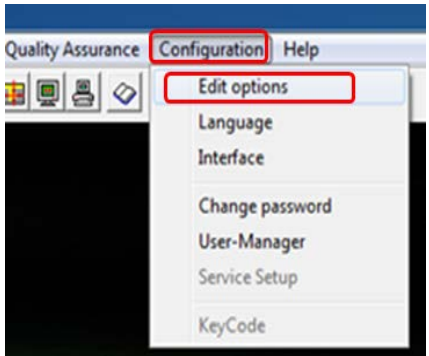
8. Switch on the analyzer and sampler.

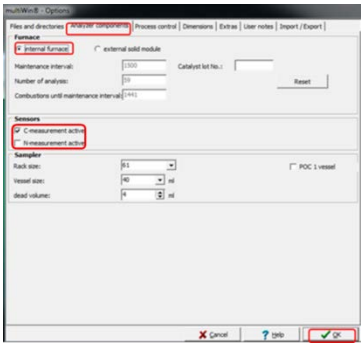
9. Switch on the PC.
10. **Wait till the LED on the analyzer illuminates green**, double click on **[mutiwin]** on the PC.
11. Log in. The password is “Admin”.

12. Don’t initialize the analyzer right now.



13. In the window **options/ analyzer components** (menu command **configuration> edit options**)





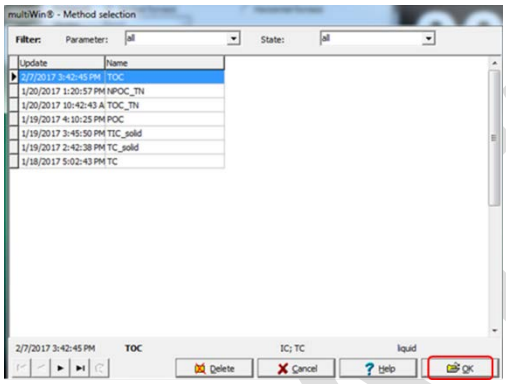
activate the internal furnace and the sensors you need.

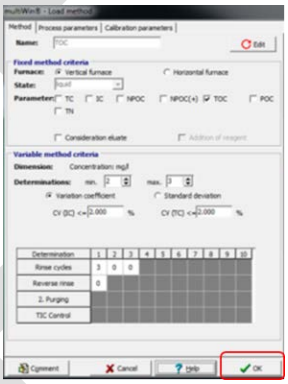
IV. Loading or creating a method.

Load an existing method

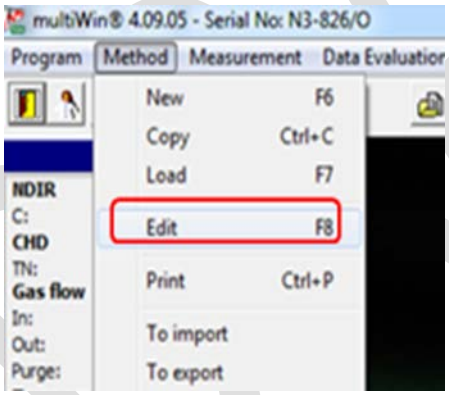
1. Click on the **[load method]** button at the bottom of the window.

2. Choose the method you need, and click on **[ok]** button.



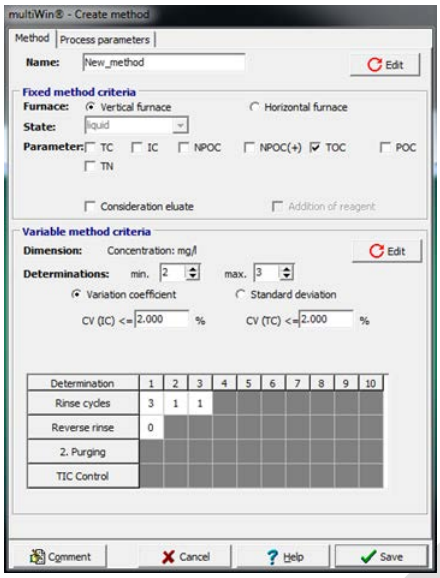


3. If you want to make some modification of the method, **Method> edit**.



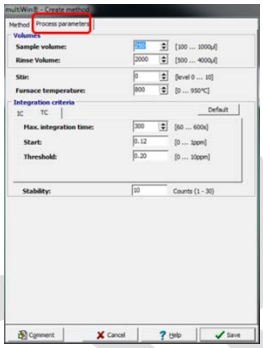
Create a new method

1. **method >new**
2. Enter the name of the new method. Furnace: vertical furnace. Choose the parameter need to be measured (TC, NPOC, TIC, TN), repeat time (2-3), CV (2%)



3. Click on **[procedure parameter]**, choose sample volume (250ul), max. integration time(e.g. 300s), set stir level if needed and furnace temperature. Then Click on **[save]**.

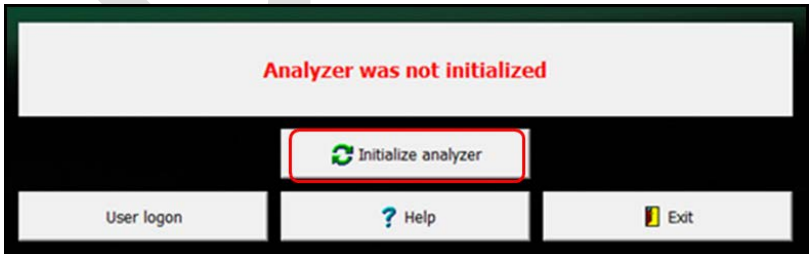
   Note: the integration criteria for different parameter (such as TC and IC) should be set separately.



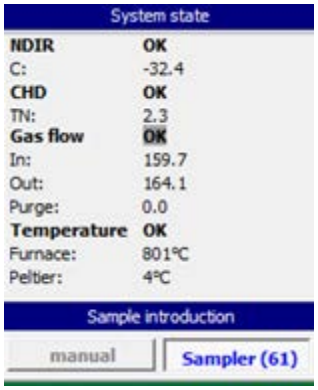
4. Click on **[yes]** to save this method, click on **[save]** again to use it as current method.

V. Initializing the analyzer

1. Click on the button **[initialize analyzer]**.



2. Wait till none components are shown in red in the window **system state**.



If the analyzer is not ready for measurement after 30 minutes (one or several components are still shown in red in the window **System state**), contact Yili or Xiao.

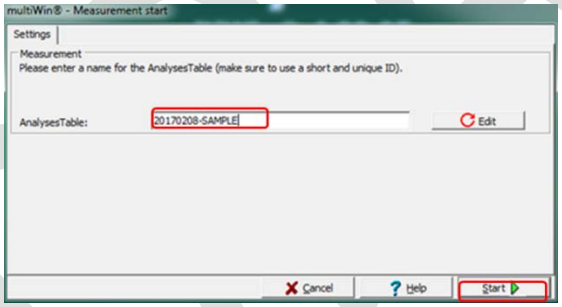
3. Click on **[sampler]** (if it is not in blue) in the window **system state.**

VI. Measuring daily factor

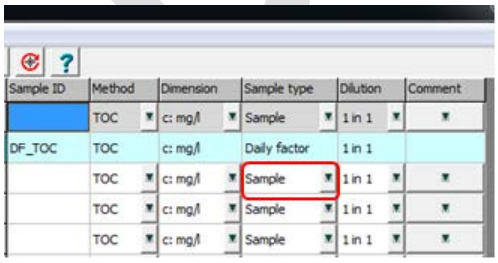
Use one standard sample to check and correct the calibration.

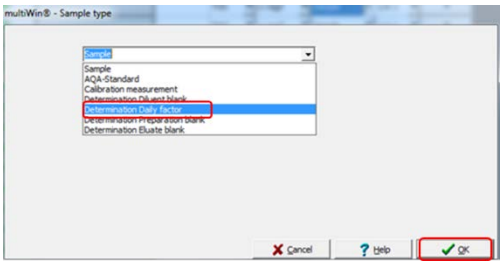
1. Click on the **[Start measurement]** button.

2. Enter the name of the **analyses table,** then click on **[start]**.

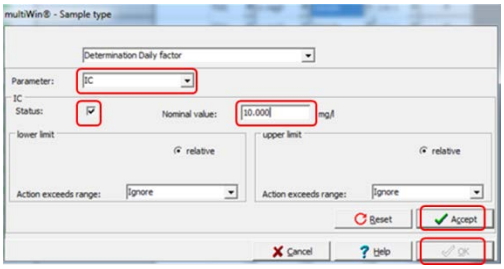


3. Click the cell under **sample type** (in the line of your sample position), Choose "**Determination Daily factor**", then click on **[OK]**.

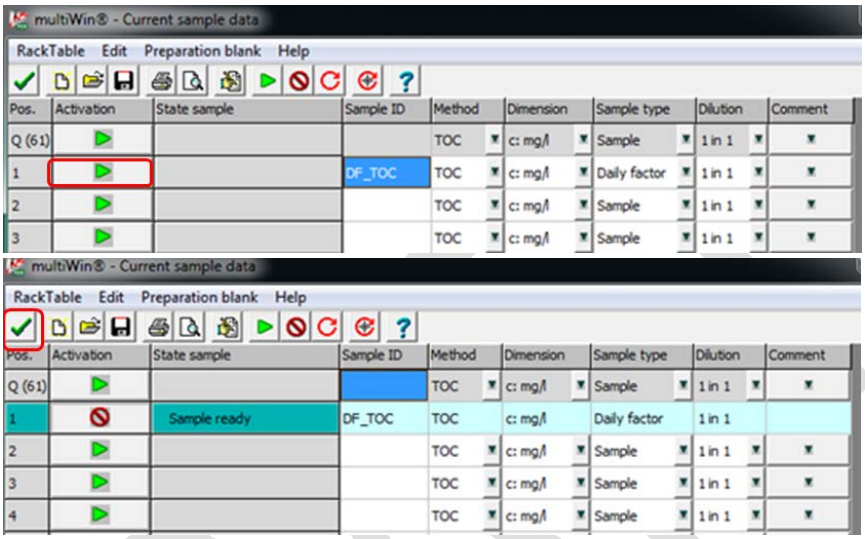




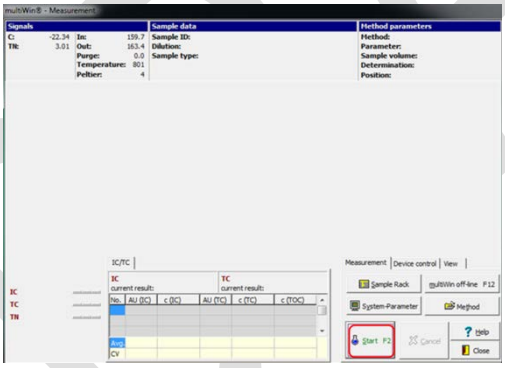
4. Complete the "sample type". Then Click on **[accept]** and **[OK]** in turn.



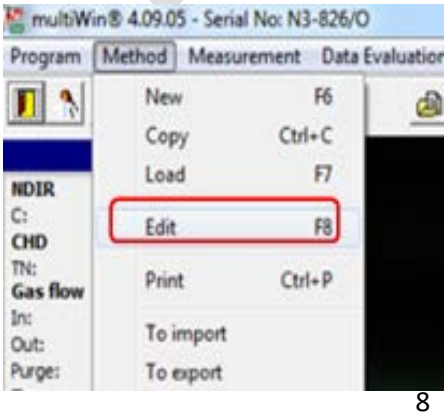
5. Click on [▶] in the line of your sample for daily factor, till it becomes [⊘] then click on [✔],

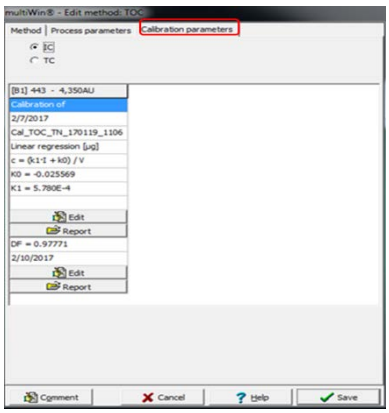


6. Click on **[START F2]** to begin the measurement.



7. if you want to see the daily factor of current method,
Method> **edit method**> **calibration parameter**.



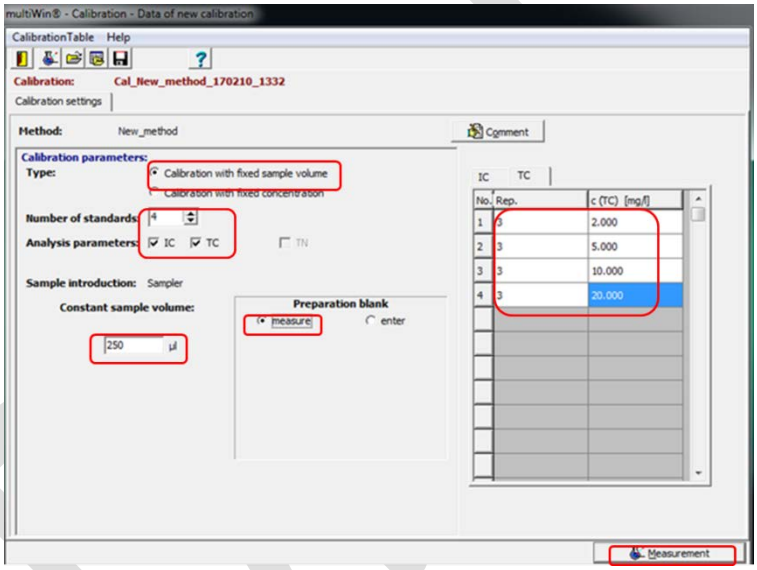


8. The daily factor can be within the range of 0.8 - 1.2, if it satisfies this request, you can measure your sample right now.
   If not a complete calibration is required.

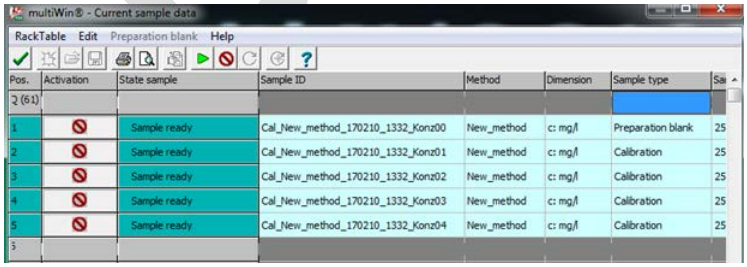
VII. Calibration

1. Click on **[calibration start]**.

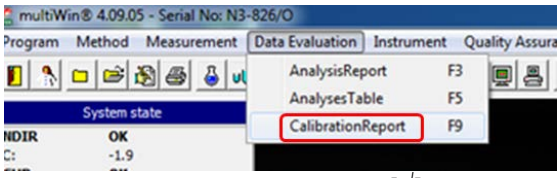
2. Choose the method or load an existed calibration table follow the instructions on the monitor screen.
3. Edit **calibration settings** then click on **[measurement]**.
   Note: the concentration should be set for each parameter separately.



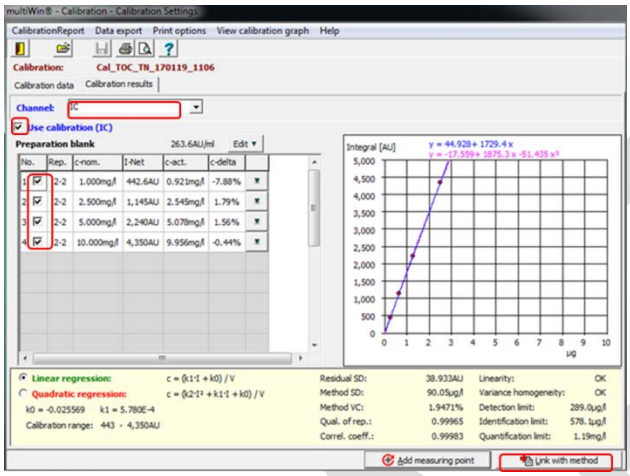
4. Click on [▶] till it becomes [⊘] then click on [✔],



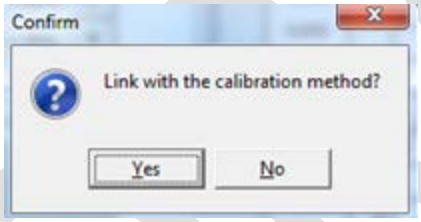
5. Click on **[START F2]** to start the measurement.
6. The calibration report will open when the calibration finished or through "**data evaluation> calibration report**"



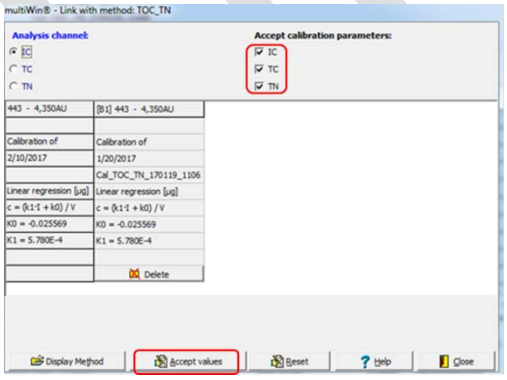
7. Choose calibration channel, use **[√]** to activate the calibration curve. Then click on the button [**link with method**].



8. "link with the calibration method?"
   [Yes] the link is made with the calibration method (default).
   [No] the calibration parameters are linked to the selected method.



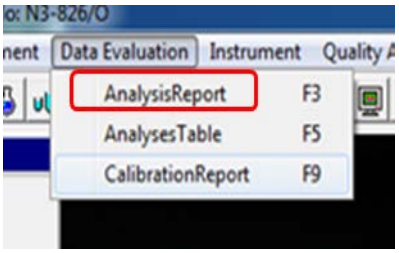
9. Choose calibration parameters, then click on the button **[accept values]** to save calibration value.

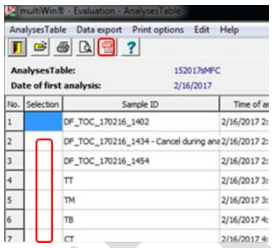


VIII. Measurement

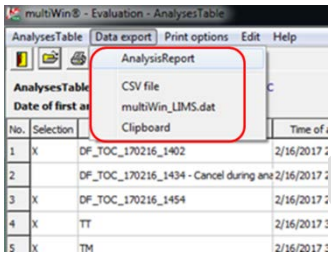
1. Click on **[start measurement]**.
2. Enter the name of analyses table, and click on **[edit].**

3. Edit the sample table (sample ID), Click on [▶] till it becomes [⊘] then click on [✔],
4. Click on **[START F2]** to begin the measurement.
5. Measure pure water as sample to wash the system until no obvious peak exits.
6. If you want to export your data, **Data evaluation> analysis table**, select your sample, click on **[pdf]** to export in PDF.



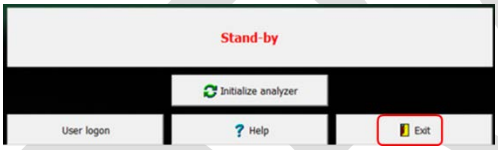


Or click on **[data export]** and choose the export type you need.

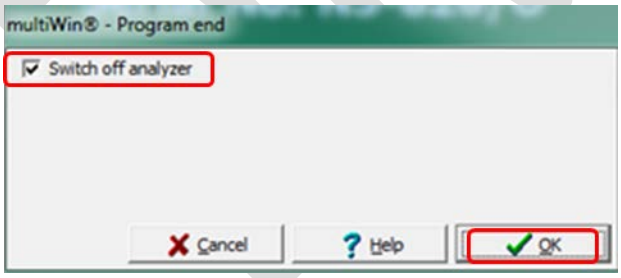


IX. Switching off the analyzer

1. Click on **[exit]** to exit the software.



2. Choose **[switch off analyzer]** and click on **[OK]**.



3. Wait for half an hour for the cooling of the machine.
4. Close the analyzer and the sampler.
5. Close the computer.
6. Close the valve on the O2 cylinder and then the valve at the pressure reducer of the gas supply.

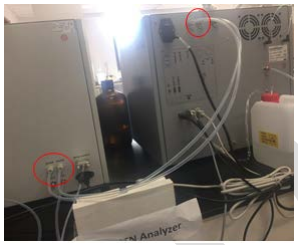
HT1300 (TC solid module)

I. General notes on your analyses

1. The sample boat should be totally dried (dry it in HT1300 for several minutes before measure your samples).
2. The sample should be sieved to power articles and totally dried.
3. A max. 3000 mg of sample may be weighed in per measurement.
4. To prevent explosive incineration cover samples with high organic carbon concentration with quartz sand.

II. Switching on the solids module

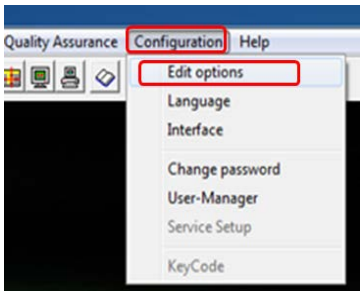
1. Open the O2 valve, if the total O2 pressure is less than 3MPa, contact Yili or Xiao.
2. Make sure halogen trap is usable. (The color of copper and brass wool didn't change.)
3. Make sure the dryer is usable. (It should be renewed when 2/3 of the dryer became baked)
4. The hoses are connected properly to the analyzer. (Analyte to analyte and pump to pump).

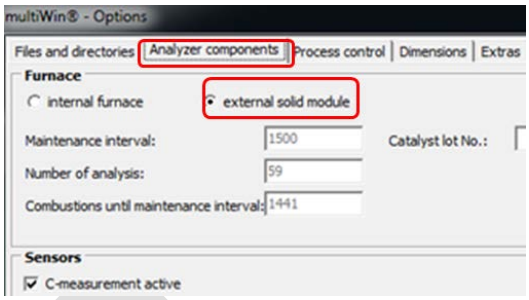


5. Open the valve at the pressure reducer of the gas supply and make sure the preliminary pressure is 4 to 6 bar.
6. Switch on the solids module from the main switch at the front.

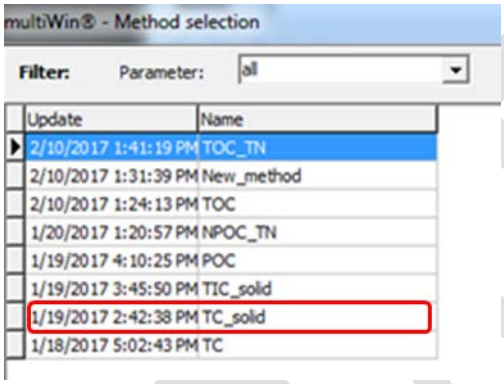
7. Switch on the analyzer.
8. Switch on the PC.
9. **Wait till the LED on the analyzer illuminates green**, double click on **[mutiwin]** on the PC.
10. Log in. The password is "Admin".
11. Don't initialize the analyzer right now.

12. Make sure the activated furnace is external solid module.
    In the window **options/ analyzer components** (menu command **configuration> edit options**) activate the external solid module and C-measurement.. Click on **[ok]**.

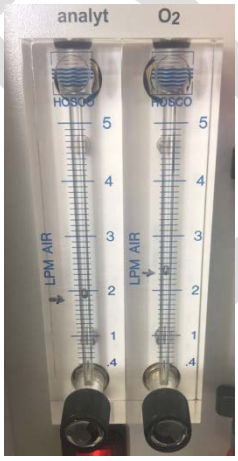




13. Click on the **[load method]** button, choose "**TC_solid**" and then click on **[ok]**.



14. Click on the button **[initialize analyzer]**.
    If the analyzer is not ready for measurement after 30 minutes (one or several components are still shown in red in the window **System state**), contact Yili or Xiao.
15. Set the oxygen flow at the rotameter "oxygen" 0.5 l/min more than "analyt". (the position marked by arrow).

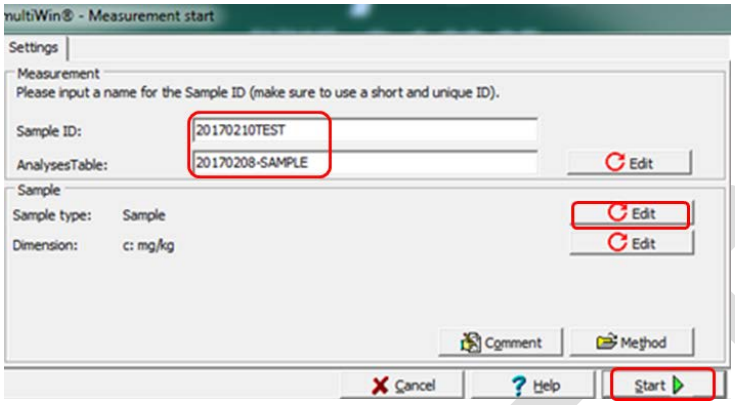


III. Measurement

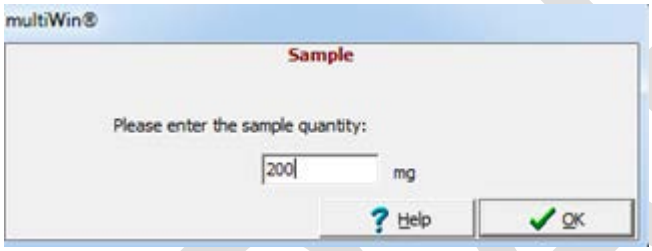
The following step is used for the measurement of both the daily factor and your samples.

1. Weigh the finely ground sample into the sample boat.

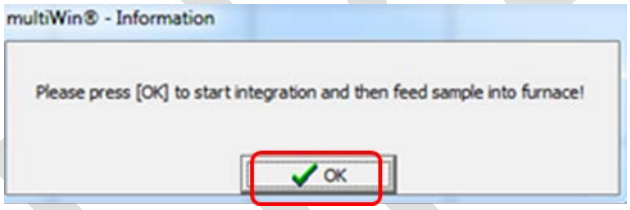
2. Measurement> start measurements.
3. Enter the sample ID and a name for the analysis table, define the sample type. With **[start]** open the window measurement. (For daily factor, click on **[edit]** behind the sample type, and choose daily factor.)



4. Click on **[start F2]** and follow the subsequent prompts of the multiWin program.
5. In the window **sample** enter the sample volume in **[mg]** and click on **[ok]**.



6. Click on **[ok]** in the window ask for feed sample.

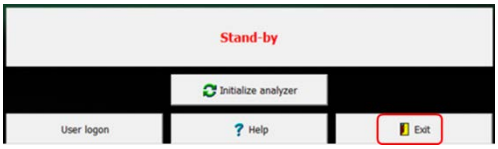


7. Place the sample boat onto the table and slide the sample boat with the loading tool into the hot zone of the combustion tube until the stop at the loading tool makes contact with the front edge of the rack.

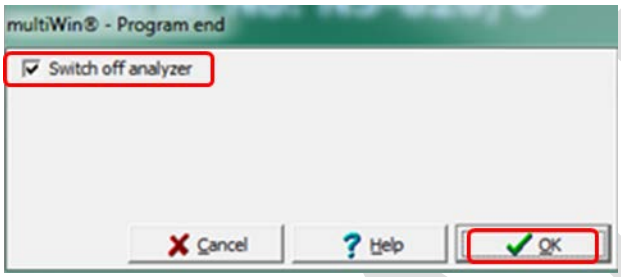
8. Once the measurement is complete, pull the sample boat back out of the combustion tube with the loading tool.

IV. Switching off the analyzer

1. Click on **[exit]** to exit the software.



2. Choose **[switch off analyzer]** and click on **[OK]**.



3. Wait for half an hour for the cooling of the machine.
4. Close the analyzer, sampler and solid module.
5. Close the computer.
6. Close the valve on the O2 cylinder and then the valve at the pressure reducer of the gas supply.

TIC solid module

I. General notes on your analyses

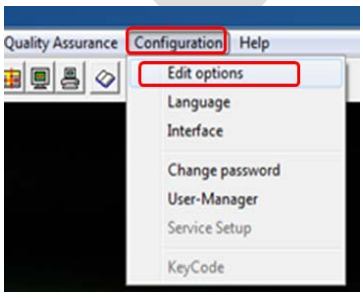
1. The sample should be sieved to power articles and totally dried.
2. A max. 3000 mg of sample may be weighed in per measurement.

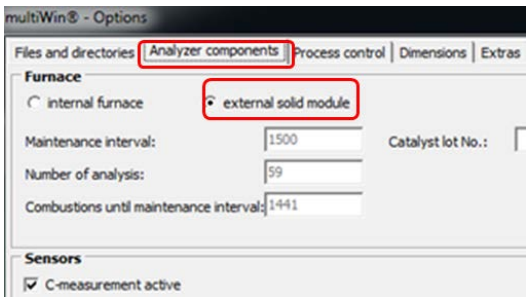
II. Switching on the solids module

1. Open the O2 valve, if the total O2 pressure is less than 3MPa, contact Yili or Xiao.
2. Make sure halogen trap is usable. (The color of copper and brass wool didn't change.)
3. Make sure the dryer is usable. (It should be renewed when 2/3 of the dryer baked)

4. The hoses are connected properly to the analyzer. (Analyte to analyte).

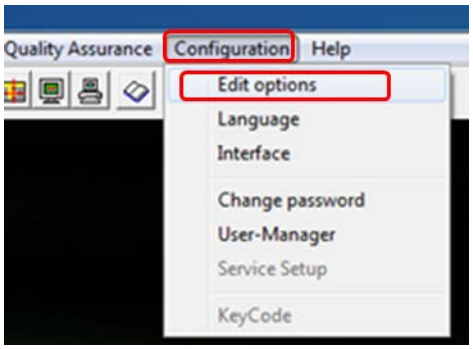
5. Open the valve at the pressure reducer of the gas supply and make sure the preliminary pressure is 4 to 6 bar.
6. Switch on the TC solid module (at the front of the module), TIC solid module (at the back) and the analyzer.
7. Switch on the PC.
8. **Wait till the LED on the analyzer illuminates green**, double click on **[mutiwin]** on the PC.
9. Log in. The password is “Admin”.
10. Don’t initialize the analyzer right now.
11. Make sure the activated furnace is external solid module.
    In the window **options/ analyzer components** (menu command **configuration> edit options**) activate the external solid module. Click on **[ok]**.

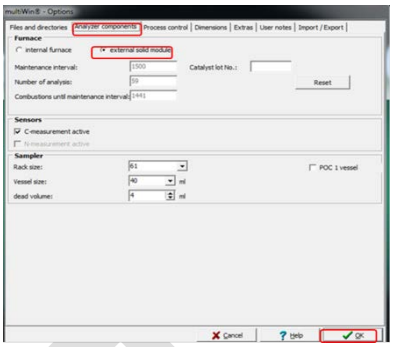




12. Click on the **[load method]** button, choose “**TIC_solid**” and then click on **[ok]**.

13. In the window **options/ analyzer components** (menu command **configuration> edit options**) activate the external solid module and C-measurement.





14. Click on the button **[initialize analyzer]**.
    If the analyzer is not ready for measurement after 30 minutes (one or several components are still shown in red in the window **System state**), contact Yili or Xiao.
15. Set the oxygen flow at the rotameter “oxygen” 12 l/min.

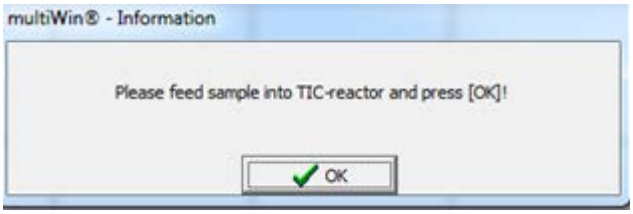
III. Measurement

The following step is used for the measurement of both the daily factor and your samples.

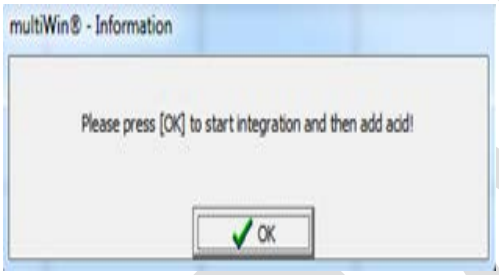
1. Click on **[start measurement]**.

2. Enter the sample ID and a name for the analysis table, define the sample type for the sample to be measured. With **[start]** open the window measurement. (For daily factor, click on [edit] behind the sample type, and choose daily factor.)

3. Click on **[start F2]** and follow the subsequent prompts of the multiWin program.
4. In the window **sample** enter the sample volume in **[mg]**.
5. Feed sample into TIC-reactor and press **[ok]**.

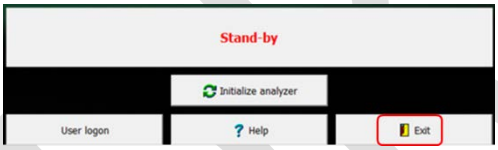


6. Press [ok] to start integration and then add acid. You can turn on the stirrer to speed up the reaction.
   Notes: always close the stopcock at the acid dispenser in periods of non-use and between the measurements.

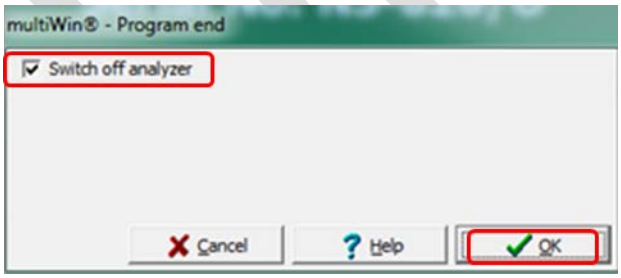


IV. Switching off the analyzer

1. Click on **[exit]** to exit the software.



2. Choose **[switch off analyzer]** and click on **[OK].**



3. Wait for half an hour for the cooling of the machine.
4. Close the analyzer, sampler and solid module.
5. Close the computer.
6. Close the valve on the O2 cylinder and then the valve at the pressure reducer of the gas supply.

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