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General Operating Procedure for: Flame mode of Atomic Absorption Spectroscopy (PinAAcle 900T)

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Contents

1. Purpose

The purpose of this GOP is to regulate the operation of flame mode of PinAAcle 900T atomic absorption spectroscopy.

2. Scope

This GOP applies to all personnel using and managing PinAAcle 900T atomic absorption spectroscopy.

3. Procedure

3.1 Switching on the system

3.1.1 Make sure the burner is correctly installed and the end cap is secured; press the white button to lock the latch if not (fig.1).

3.1.2 Make sure the door of the atomizer compartment is shut (fig.2).

Fig. 1

Fig. 2

3.1.3 Disconnect the waste tube with the instrument (fig.3), pour about 500ml water to the waste tube and reconnect the waste tube with instrument.

Fig. 3

3.1.4 Switch on the valve of C2H2 gas supply at cylinder (fig.4) and make sure there is enough for the running; the total pressure should be more than 4 bars during your test. Contact LFST to order if not.

3.1.5 Switch on the valve of C2H2 gas supply at wall outlet. Make sure the supply pressure is between 0.9-1 bars (fig.5).

3.1.6 Power on the air compressor and set it to AUTO. The supply pressure for air should be 0.4Mpa (fig.6).

Fig. 4

Fig. 5

Fig. 6

3.1.7 Make sure the ventilation system (fig.7) is working, connect LFST if not.

3.1.8 Make sure the lamp is installed (fig.8).

Fig. 7

Fig. 8

3.1.9 Switch on the computer and the spectrometer.

3.1.10 Wait till  appeared at the bottom right corner of desktop, which means the instrument have been connected with PC. Click on  to enter the system.

3.1.11 Self-diagnose will be conducted. All boxes will be ticked if the diagnose passed (fig.9). Contact LFST if not.

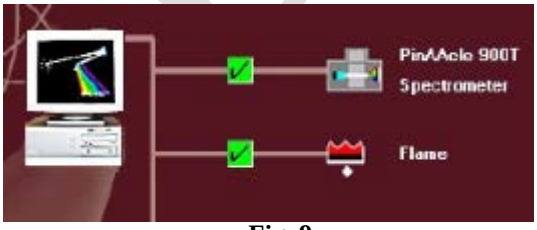

Fig. 9

3.1.12 Click on **File > change technique > flame** (fig. 10) if the current system is furnace; if not, skip this step.

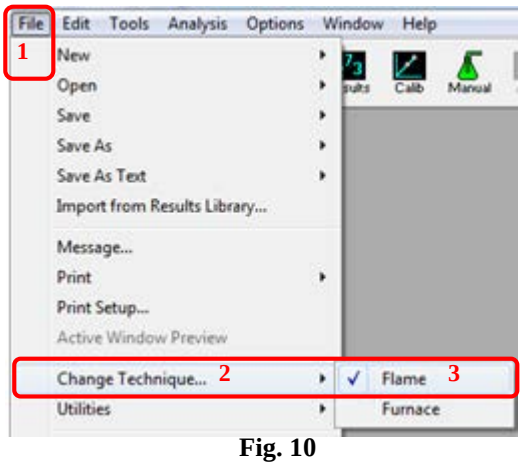

Fig. 10

3.1.13 Click on 

to open the workspace, which usually contains **manual analysis control**, **flame control**, **calibration display** and **results**. (fig.11)

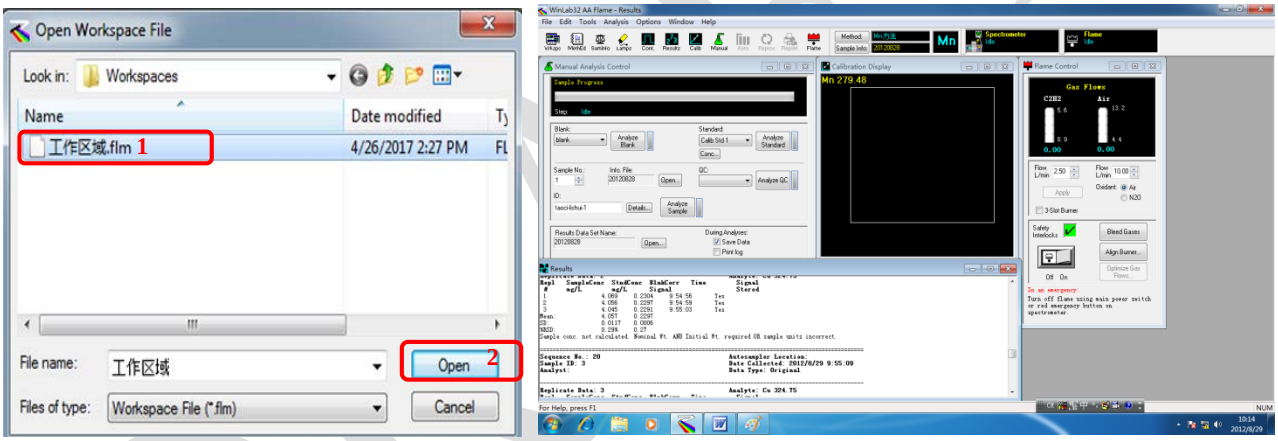

Fig. 11

3.1.14 Check the safety interlocks in the window of flame control. A green box with a check mark shows that the interlocks are satisfied (fig.12). Contact LFST if it shows a red box with an X.

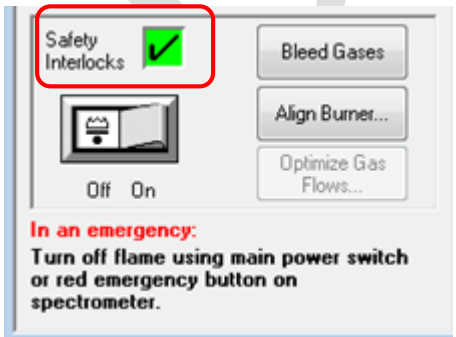

Fig. 12

3.1.15 Click on 

to open the lamp setup window. Click on  behind the target lamp to switch on the lamp (fig.13). Lamp should be warmed up for 15 min before ignite the flame. Then close lamp window.

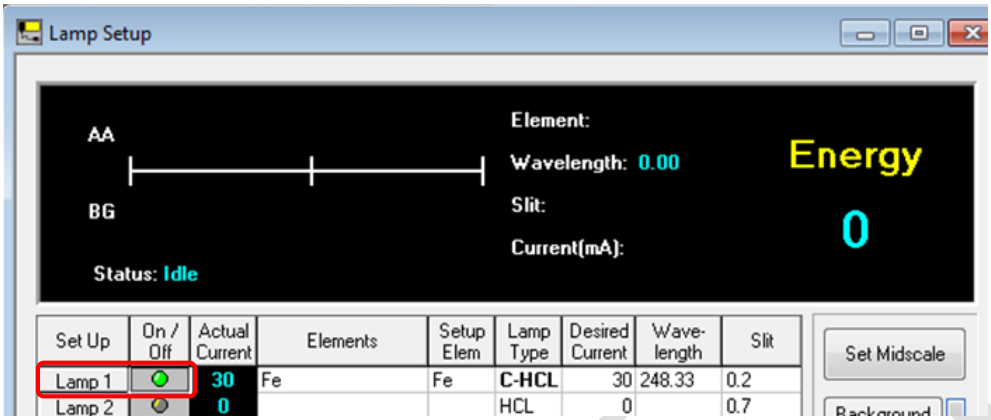

Fig. 13

3.2 Setting up a method

3.2.1 Click on **Tools > Recommended conditions** for reference, which offers you the linearity range of different element. (fig.14).

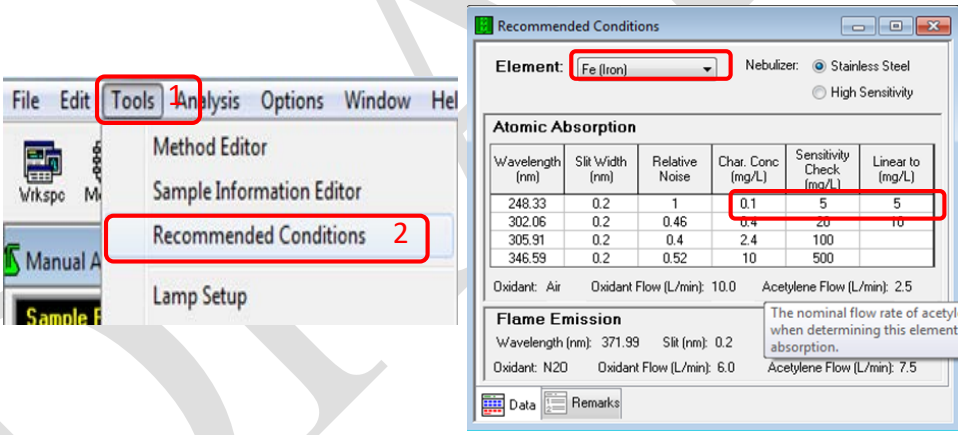

Fig. 14

3.2.2 Click on **file > new> method**, select element through the pull-down menu, and click OK (fig.15). Or **file > open> method** to open an existed method.

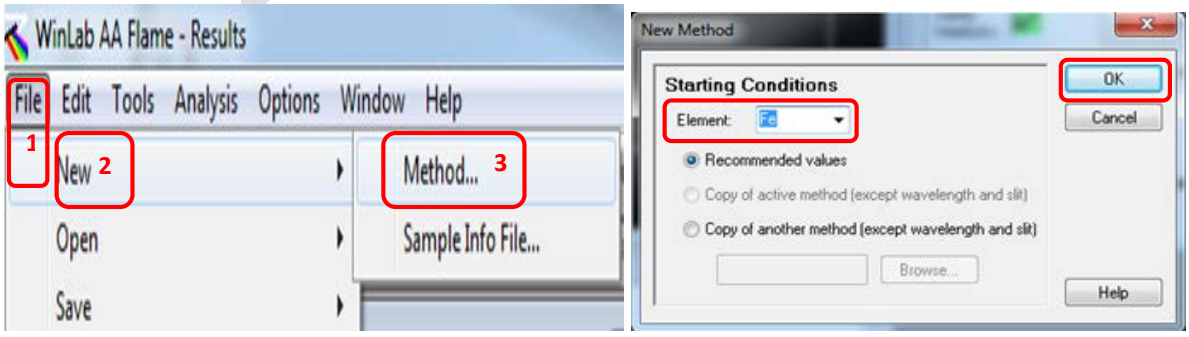

Fig. 15

3.2.3 Set parameters for **Spectrometer** (fig.16).

Define element: always use the default parameters;

Settings: set delay time to 2s and others use the default.

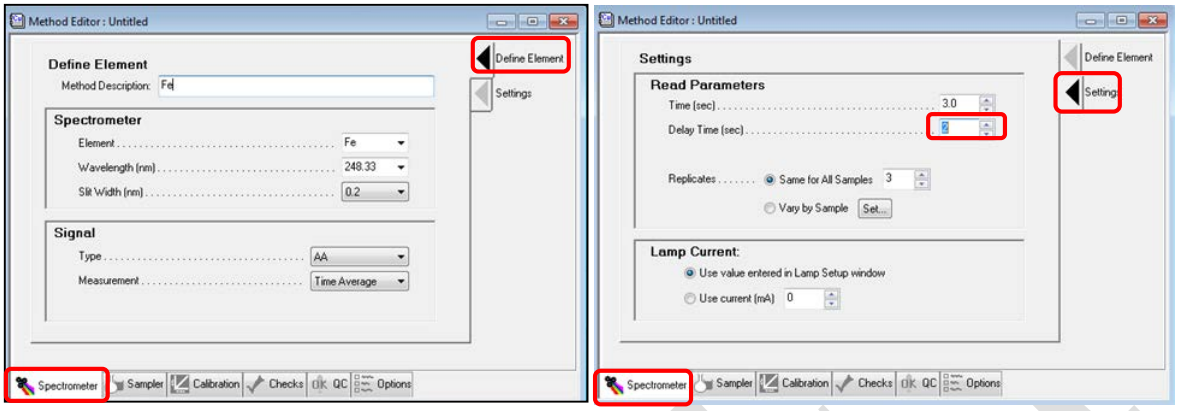

Fig. 16

3.2.4 Set parameters for **sampler**: use the default parameters (fig.17).

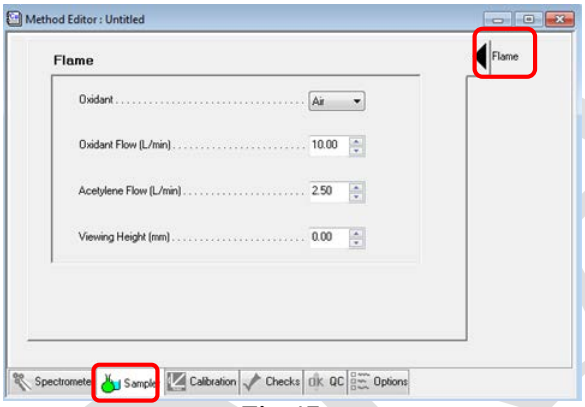

Fig. 17

3.2.5 Set parameters for **calibration**: set equation (linear, calculated intercept), units and standard concentrations (fig.18).

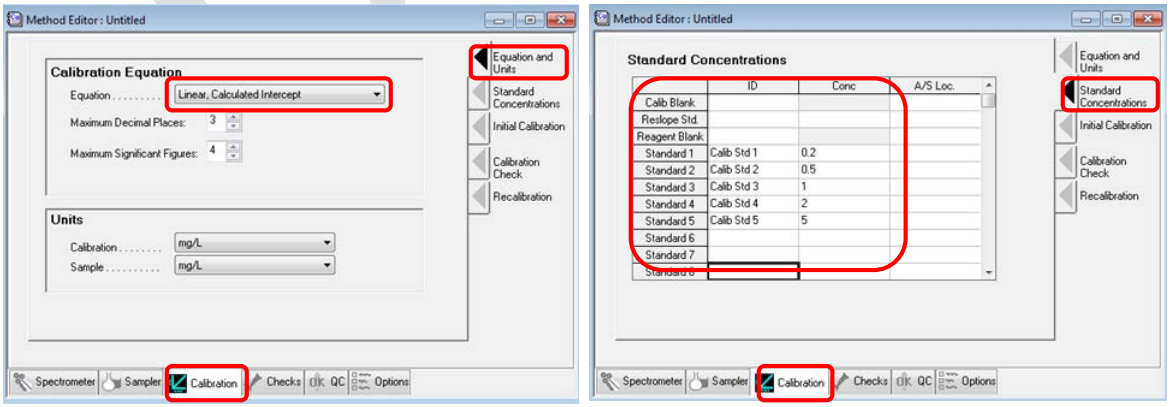

Fig. 18

3.2.6 **File > save as > method** (fig.19). Name your method and click on OK.

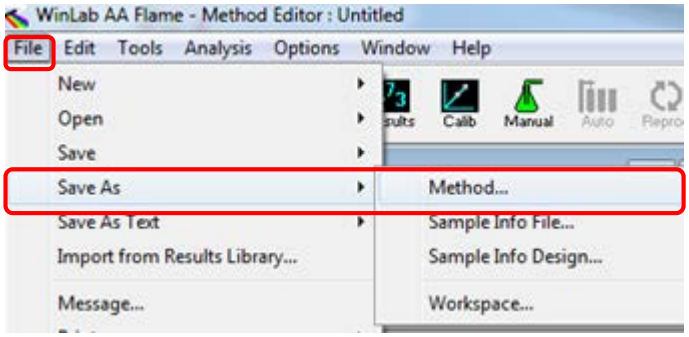

Fig. 19

3.2.7 In the window of **manual analysis control**, click on **open** behind results data set name and in the select result data set dialog appears, type a new data set name and click on ok to confirm (fig.20).

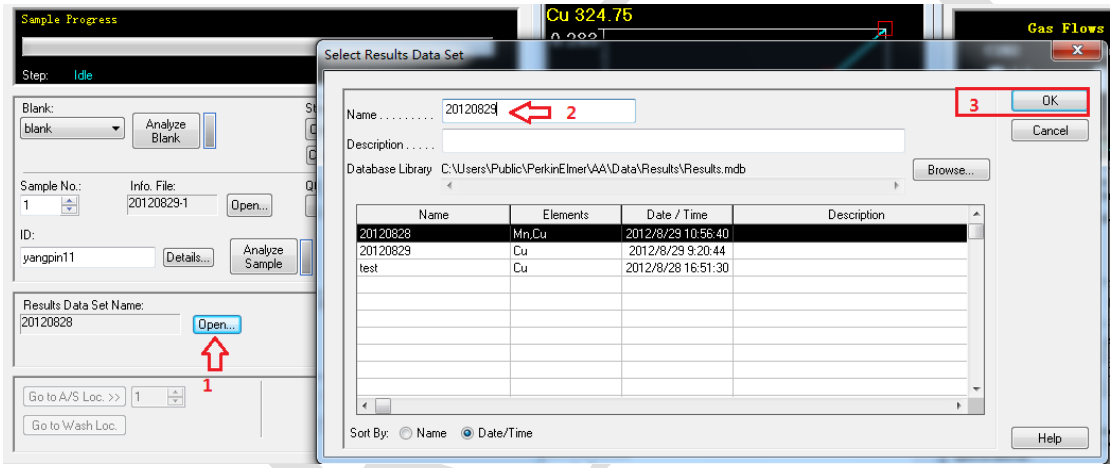

Fig. 20

3.3 Analysis

3.3.1 Create a sample information file if you intend to use one. Set sample information (fig.21) and then save (**file >save as >sample info file**).

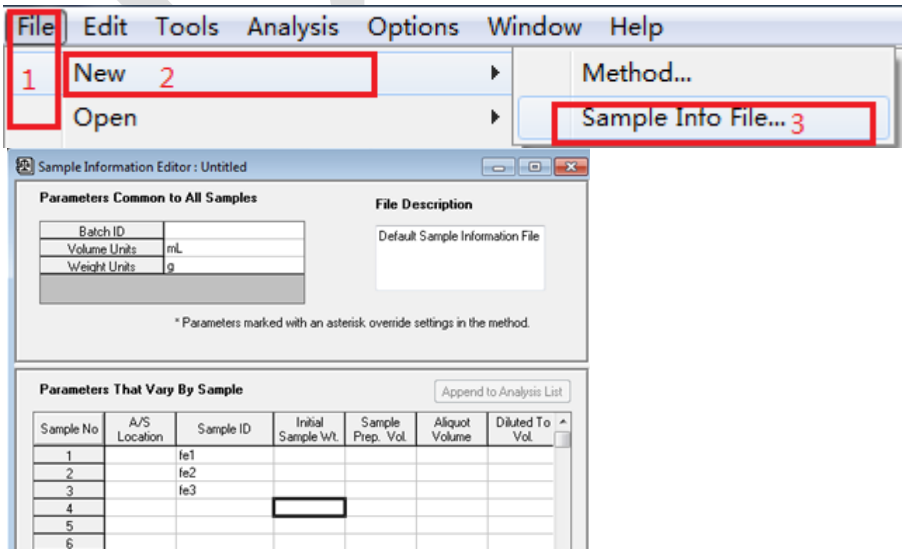

Fig. 21

This step is optional; you do not have to use a sample information file. You can enter the sample ID directly in the manual analysis window before you analyze each sample.

3.3.2 Ignite the flame (fig.22).

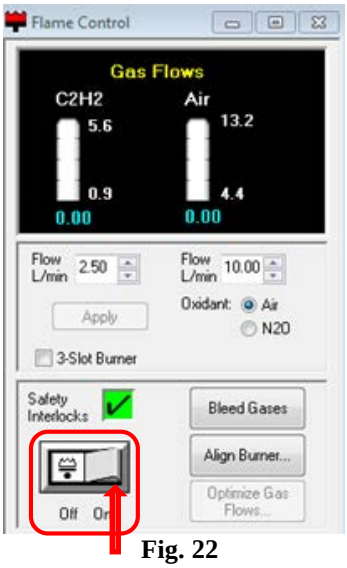

Fig. 22

3.3.3 Click on **analysis > clear results display** if you need to clear previous data.

3.3.4 Place the sample tube in calibration blank and click on 

in manual analysis control window, analyze blank for twice.

3.3.5 Analyze standards. Check the result after all standards have been tested (fig.23). R>= 0.999, contact LFST if not.

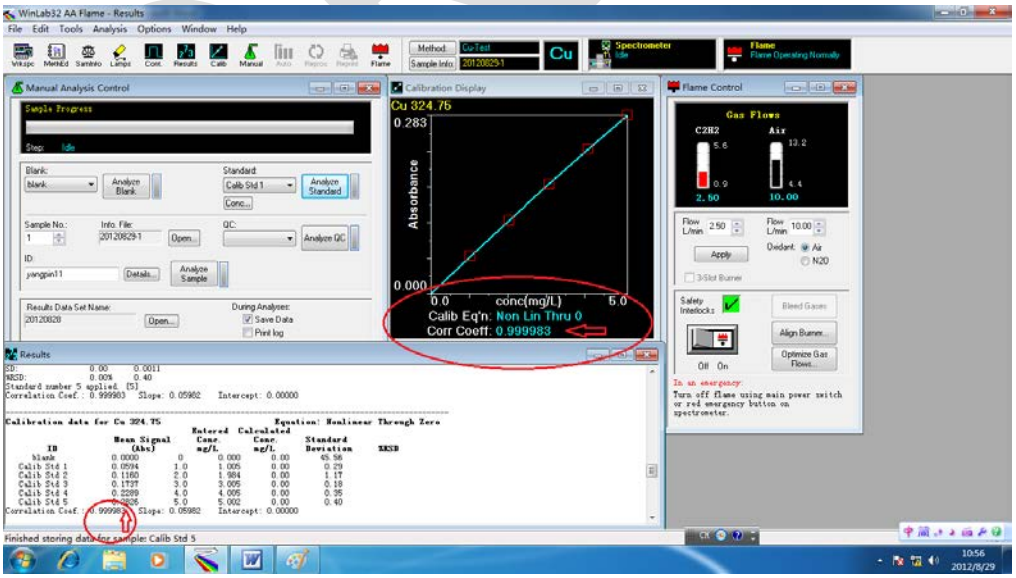

Fig. 23

3.3.6 Analyze samples. Click on **open** behind **info.file** to load sample info file if needed. Place the sample tube in corresponding sample. Select sample No and then click on Analyze sample. (fig.24)

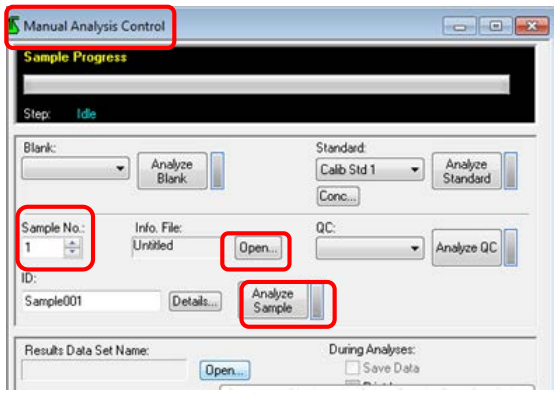

Fig. 24

3.4 Shutting down instrument

3.4.1 With the flame still burning, aspirate the correct rinsing solutions, in the sequence listed below, to rinse the nebulizer and burner.

- If you used only aqueous solutions during the analysis:

  Aspirate deionized water for five minutes.

- If you used organic solvents during the analysis:
  - Aspirate an organic solvent that is miscible with both the solvent you used during the analysis and water for five minutes.
  - Aspirate acetone for five minutes.
  - Aspirate 1% nitric acid for five minutes.
  - Aspirate deionized water for five minutes.

3.4.2 Click **off** 

to extinguish the flame.

3.4.3 Click on 

to open the lamp setup window. Click on  to switch off the lamp.

3.4.4 Shut the valves of the C2H2 gas supplies to the spectrometer at the cylinder and wall outlets.

3.4.5 Click on 

in the window of flame control to vent the burner gas lines to the atmosphere.

3.4.6 Pull the plug of air compressor and drain the waste (fig.25).

Fig. 25

3.4.7 Click on **window > close all windows** (fig.26).

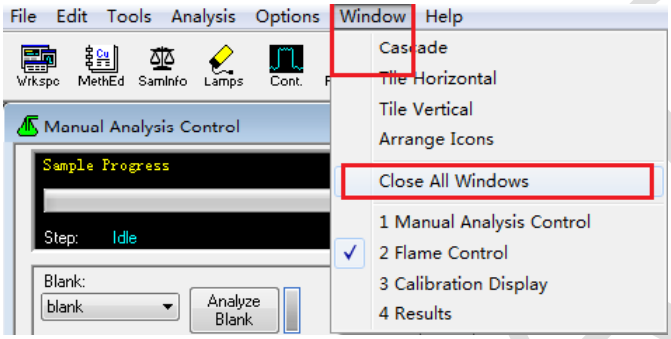

Fig. 26

3.4.8 Exit Winlab, switch off the spectrometer and computer.

Document information:

Document No:	GOP-AAS-01	Version No:	0.1 (draft)
Drafted/ modified by:	Yili Cheng	Effective date:	
Comments:			