



High Power XRD (HPXRD)

CONTACT

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Information



HPXRD is high power X-ray Diffraction machine.

After turning on X-ray in low power,

It measures sample by raising machine to a stable high power.

You can check equipment condition on Control Board which located at bottom of HPXRD.

1) Vacuum: always 'ON'

2) Power: POWER ON → X-ray 'ON'

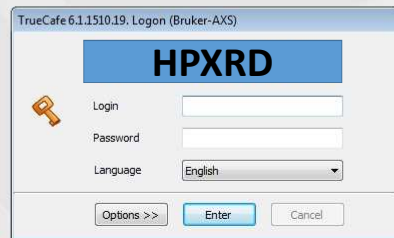
3) X-ray: located next to X-ray button,

- Voltage= 0, Current= 0 , X-ray Off
- Voltage = 20, Current=10 : X-ray On
- Voltage = 40, Current=200 : High power X-ray On



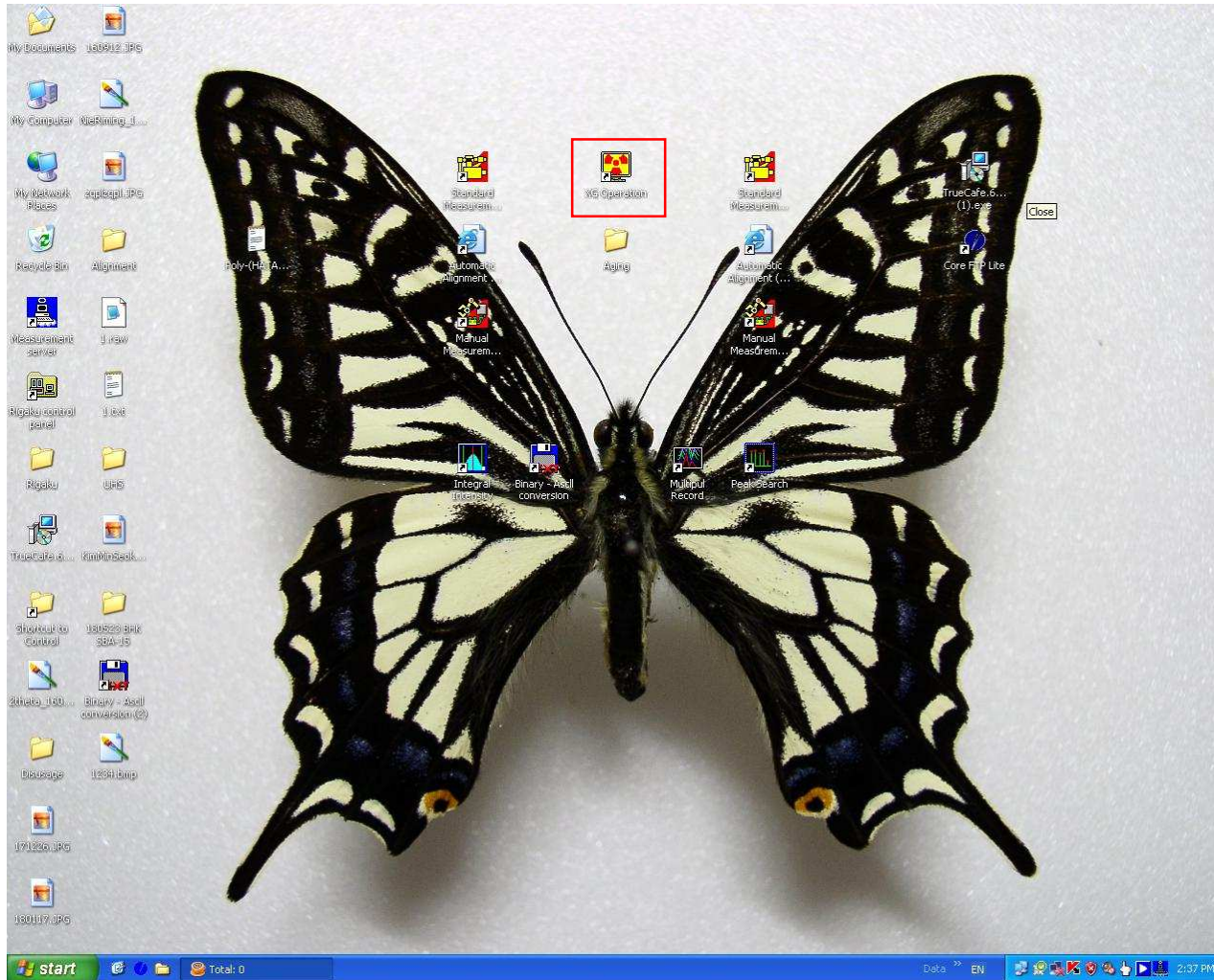


HPXRD PC Access: True Cafe



1. Enter individual ID & Password in True Café.
(One ID per person !)
2. Do not share individual ID & Password.
- Sharing has penalty or stop use for 90 days!
3. In case of machine manager,
 - request writing ID produce manual.
 - request writing corresponding manual when having problem.

X-ray Operation



XG Operation is,
X-ray Automatic On program.

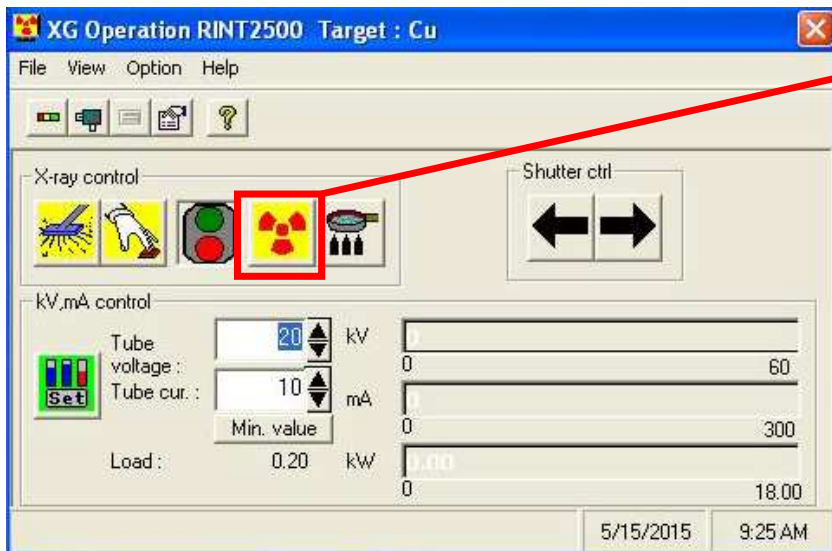
Once you do X-ray on HPXRD from 9:00~18:00,
It can be used continuously.

However,

**In case of [NIGHT use after 18:00] or [HOLIDAY use],
You should do 'Aging Process'.**

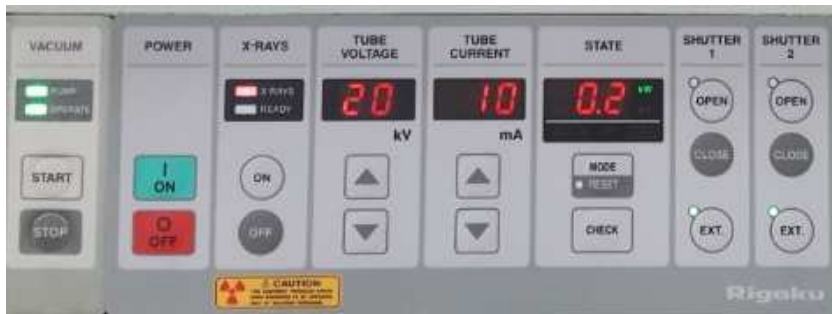
> Double click XG Operation.

X-ray Operation



1) push the 'X-ray' button.

3) All users must not enter any other number to KV, mA Control because it has fixed value **'20 KV, 10mA'**.



Sampling (while the instrument is turned on)



<Thin film Sample>

1) Cut rubber clay with scissors as size of sample.

- When rubber clay is smaller than sample, thin film can be leaned.

2) Do not mass rubber clay, and stacks in the middle of the holder.

- When you transform rubber clay, thin film can be leaned because of each different density and thickness of clay.

3) Put a thin film on rubber clay, and then press down with slide glass slowly.

※ Before that, wash the slide glass clearly.

4) Do not reuse rubber clay because it has getting lower stickiness as you use.



Sampling



<Powder Sample>

- 1) Place the heritage on the contamination pad and select the appropriate Sample Holder for the sample volume.
(Holder diameter 18mm or 5mm)
- 2) Clean the selected Sample Holder, Spatula, Slide Glass with Kimtech soaked in ethanol.
- 3) If powder is not uniform, grind with mortar.
- 4) Using Spatula, take proper amount of powder to Sample Holder Cavity and planarize it with Slide Glass.
- 5) Repeat above process until Cavity is full flat.



Door open & sample loading



1) To open the door, push the 'Door open' button.

(※Opening XRD door without pushing 'open' button, XRD Tube would be damaged with force quit.)

2) When you push the 'Door Open' button, The alarm blinks and makes a sound.

3) Grab the door handle and open the door smoothly.

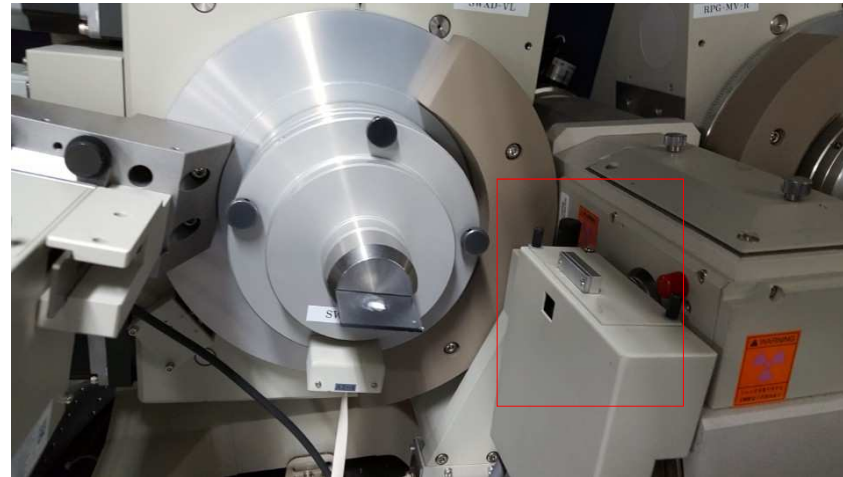
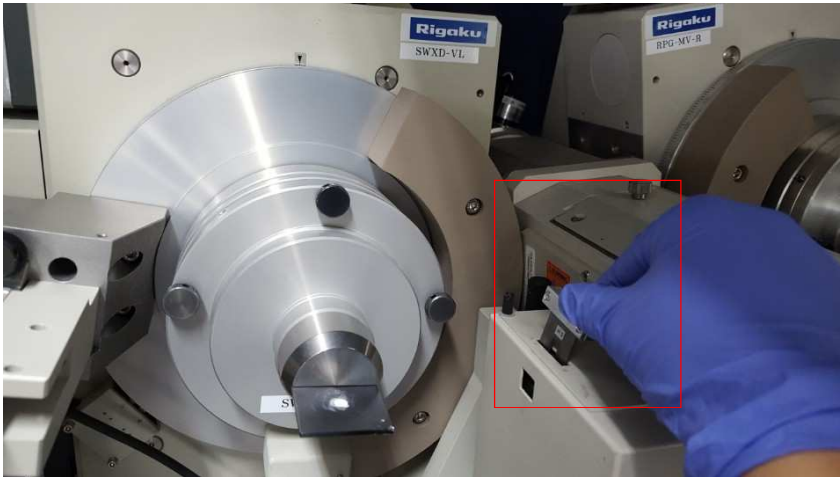
4) When you close the door, grab the both handles and close smoothly.



Door open & sample loading



- 1) In case of Auto Sampler, straight attach the magnetic sample holder and check.
 - 2) Insert holder straight in Manual Attachment and check.
 - 3) After sticking holder to Manual Attachment, match the direction of insertion and center of sample.
 - 4) According to cavity diameter of Sample Holder, select proper DHS(Divergence Height Slit) and insert direction of letters to direction of the sample.
 - 5) If DHS doesn't insert straight, do not force it and repeat until it is inserted smoothly.
- ※ Be careful that internal PCB board could be broken by force insertion.
- If you can not find any way, call the manager and ask for help.**

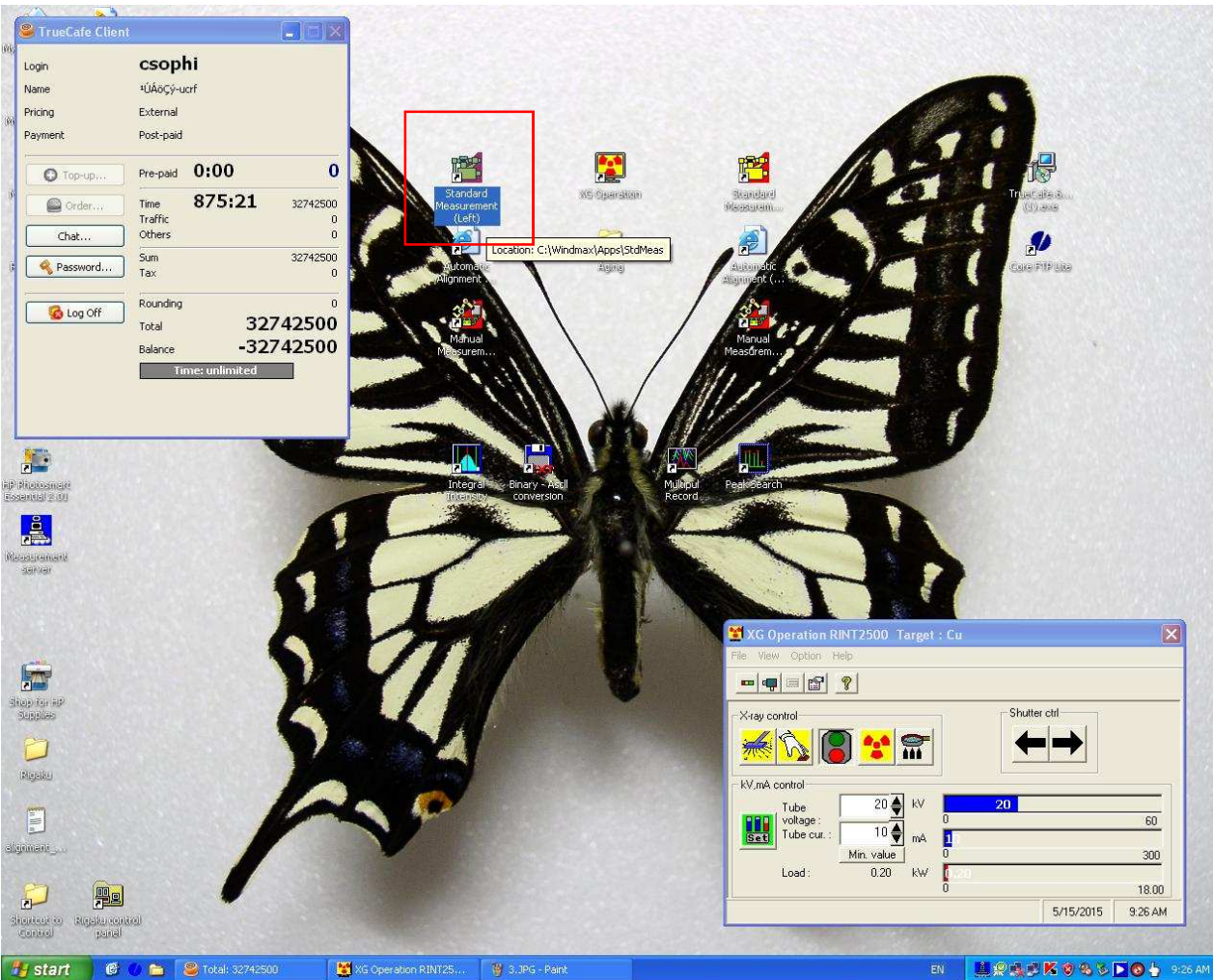


Standard Measurement (Left)

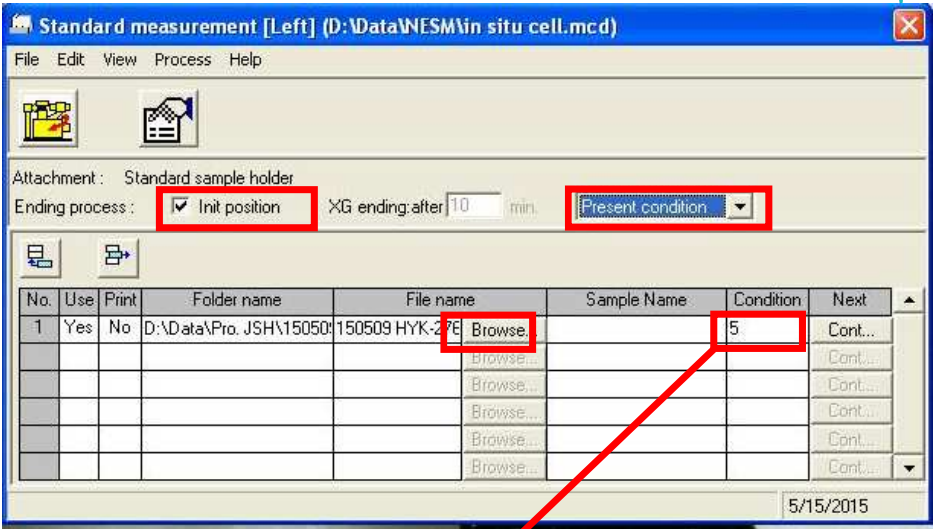
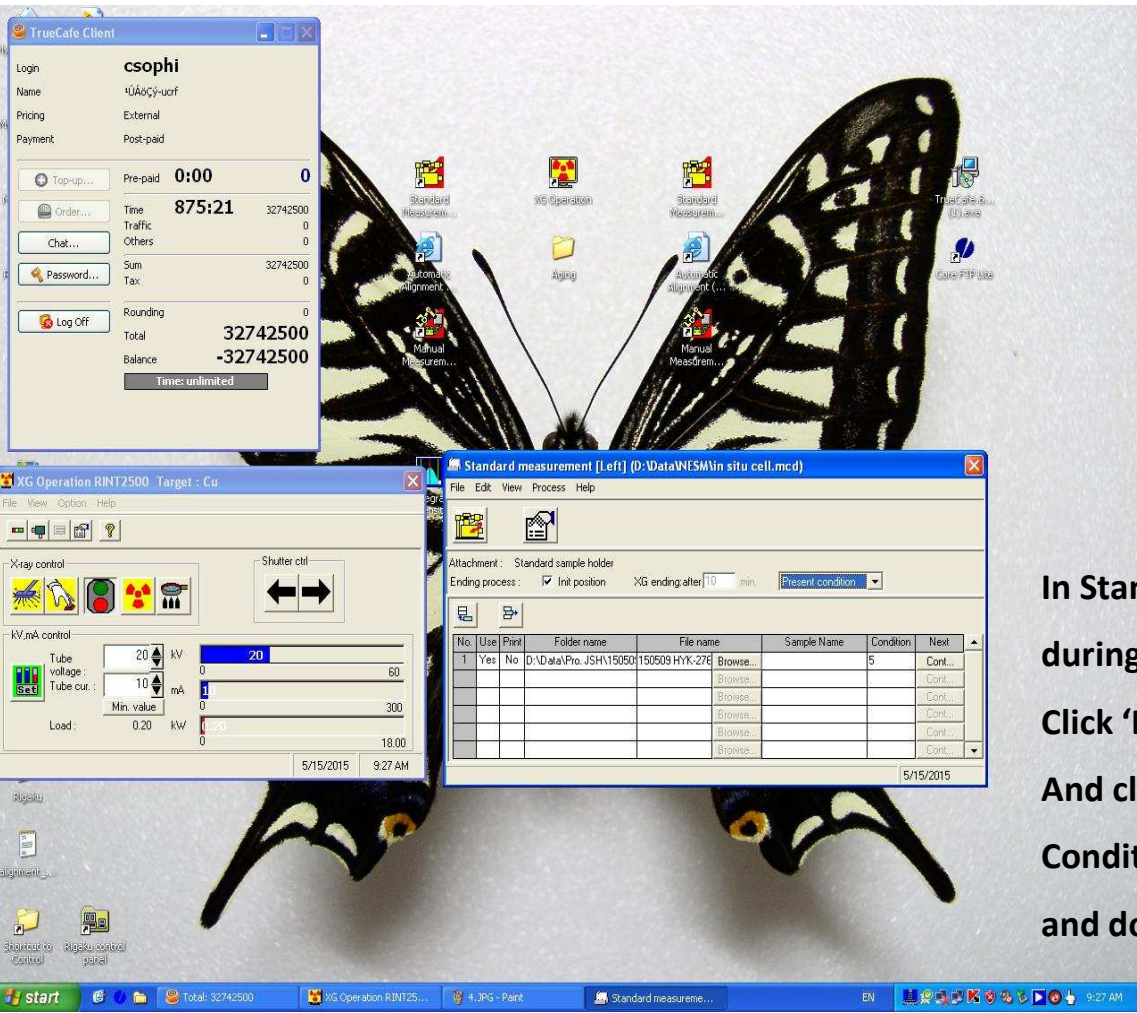


Click Standard Measurement(Left) icon
which is located to left side of XG Operation.

Standard Measurement (Left) measures One by one
using the same sample Holder.

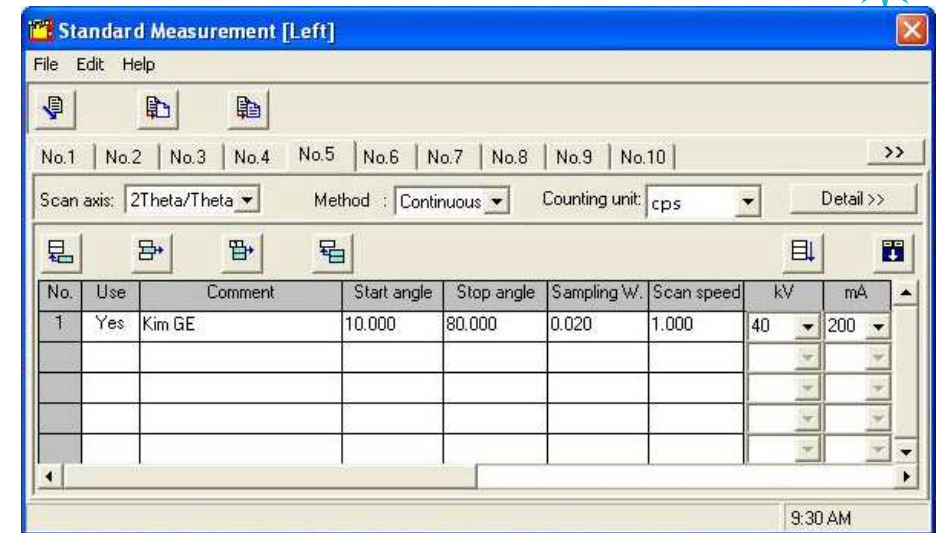
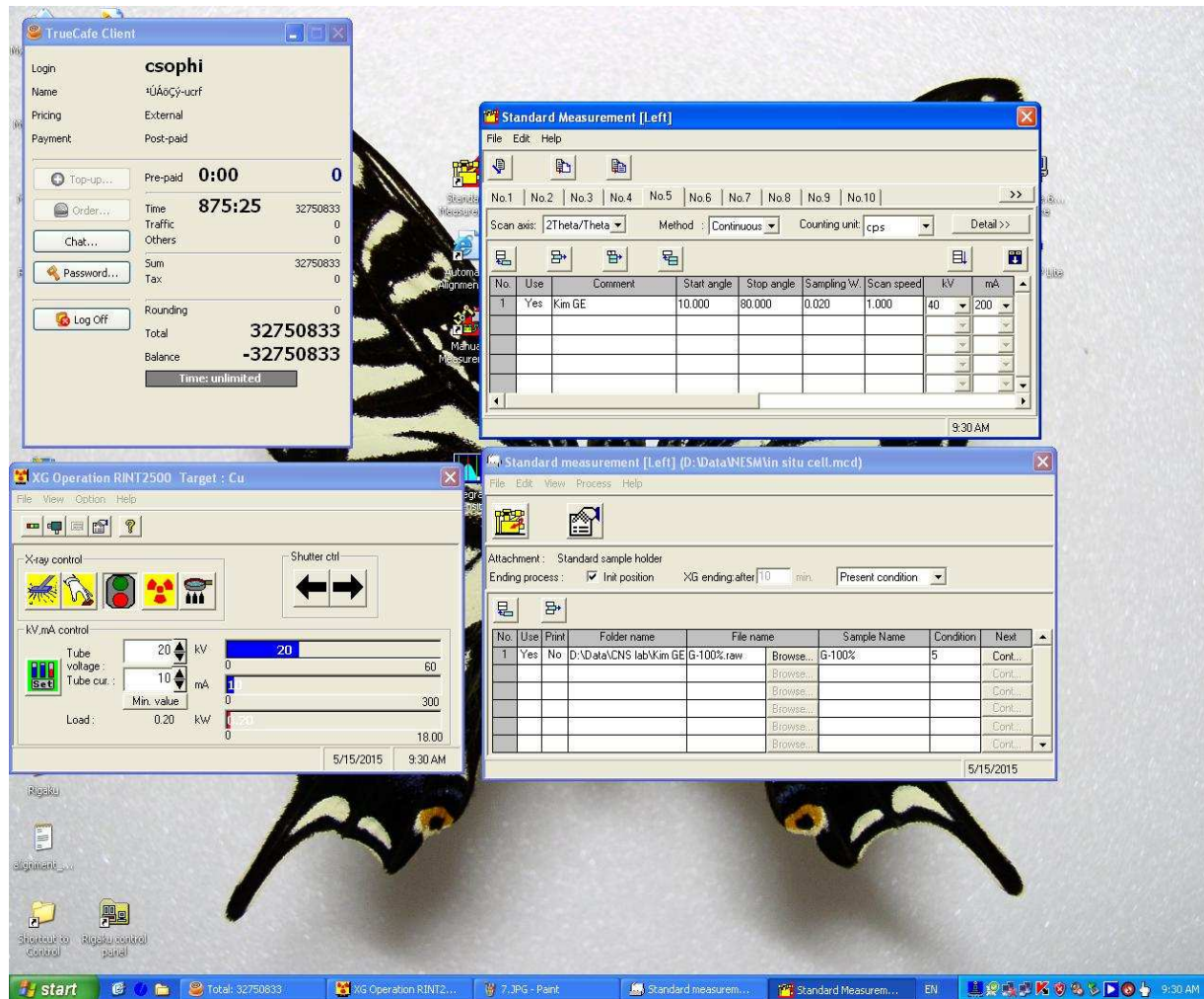


Standard Measurement (Left)



In Standard Measurement(Left),
during [weekday 9:00~18:00], check to **Int. position** and **Present Condition**.
Click 'Browse' button and fill in save location and save name
And click 'Open' button.
Condition is random of **number 2~100** except 1,
and double click number field.

Standard Measurement (Left)



In condition pop-up,

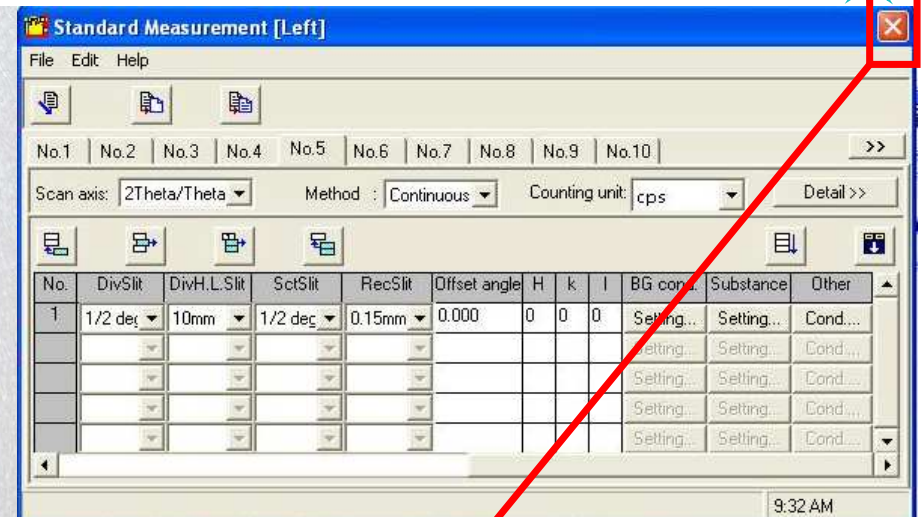
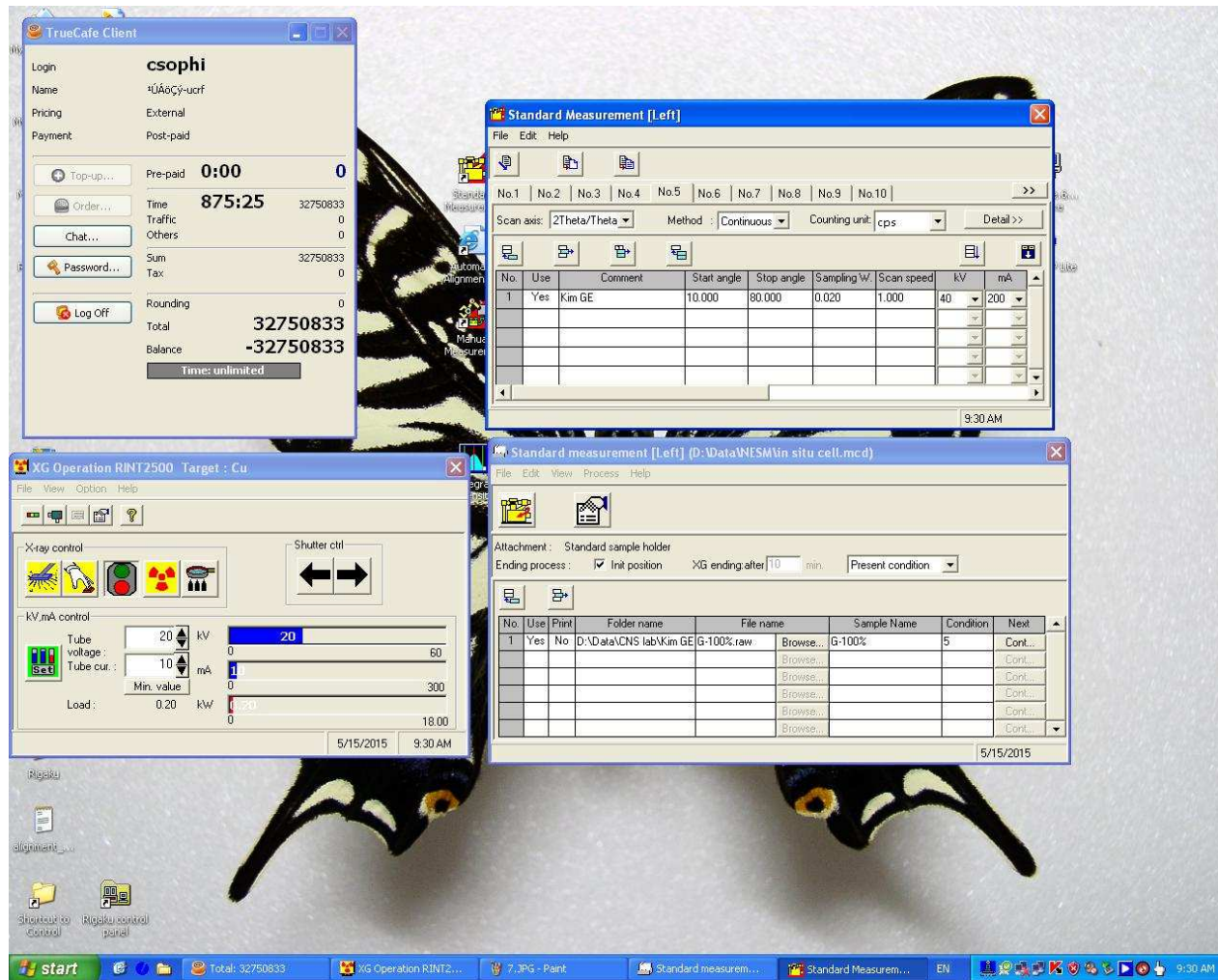
Use→**Yes**, Comment→**your choice (ex> user name..)**,

Fill in **Start angle**, **Stop angle**, **Sampling W(=0.02)**, **Scan Speed** of measurement condition.

Power→maximum **40KV, 200mA**, possible to be lower value.

And then, drag scroll bar from left to right side.

Standard Measurement (Left)



Select the Slit value of Sample holder cavity according to radius and depth value.

(Slit condition value is shown as later slide of PPT)

Click the quit button and that means Autosave.

Standard Measurement (Left)



TrueCafe Client

Login: csophi
 Name: 4040C5-ucrf
 Pricing: External
 Payment: Post-paid

Top-up... Pre-paid 0:00 0
 Order... Time 875:23 32750833
 Chat... Traffic 0
 Password... Others 0

Sum 32750833
 Tax 0
 Rounding 0
 Total 32750833
 Balance -32750833
 Time: unlimited

XG Operation RINT2500 Target : Cu

X-ray control
 Shutter ctrl
 Tube voltage: 20 kV
 Tube cur.: 10 mA
 Load: 0.20 kW

Standard Measurement [Left]

File Edit Help
 Open data file...
 Open normalizing data file...
 Close

Scan axis: 2Theta/Theta Method: Continuous Counting unit: cps

No.	Use	Comment	Start angle	Stop angle	Sampling W.	Scan speed	KV	mA
1	Yes	J00 Group	1.500	6.000	0.002	0.500	40	200

Standard measurement [Left] (D:\Data\WFSM\in situ cell.mcd)

Attachment: Standard sample holder
 Ending process: ☒ Init position XG ending after 10 min Present condition

No.	Use	Print	Folder name	File name	Sample Name	Condition	Next
1	Yes	No	D:\Data\WFSM\lab\Kim GE	G-100% raw	Browse... G-100%	5	Cont...

Standard Measurement [Left]

File Edit Help
 Open data file...
 Open normalizing data file...
 Close

Scan axis: 2Theta/Theta Method: Continuous Counting unit: cps

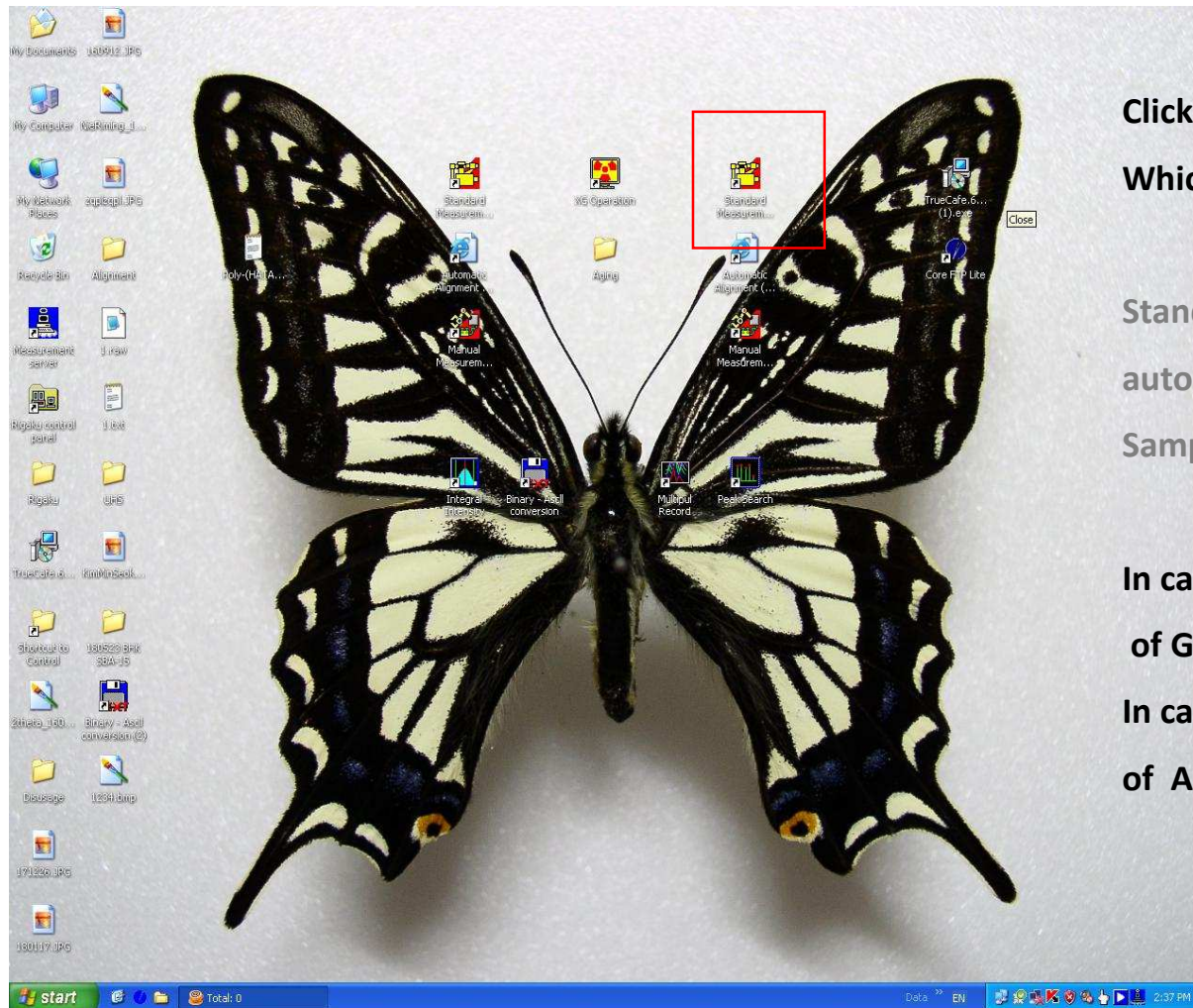
No.	Use	Comment	Start angle	Stop angle	Sampling W.	Scan speed	KV	mA
1	Yes	J00 Group	1.500	6.000	0.002	0.500	40	200

<Tip! For using the same measurement condition>

After clicking the number of 'Condition', it shows new window of Standard Measurement and then, Click 'Open Data File' in 'File'.

In new window, click the most recent measurement file.

Standard Measurement (Right)



Click Standard Measurement(Right) icon

Which is located to right side of 'XG Operation'.

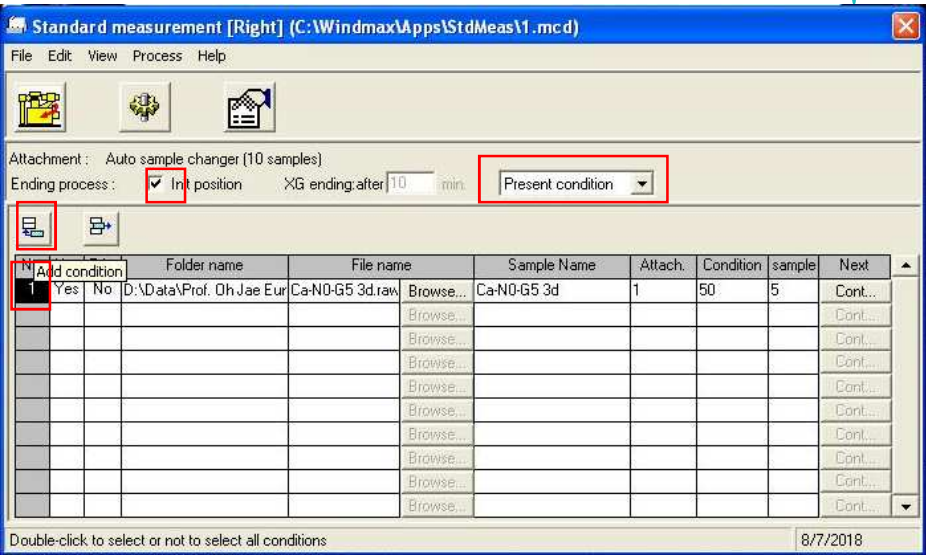
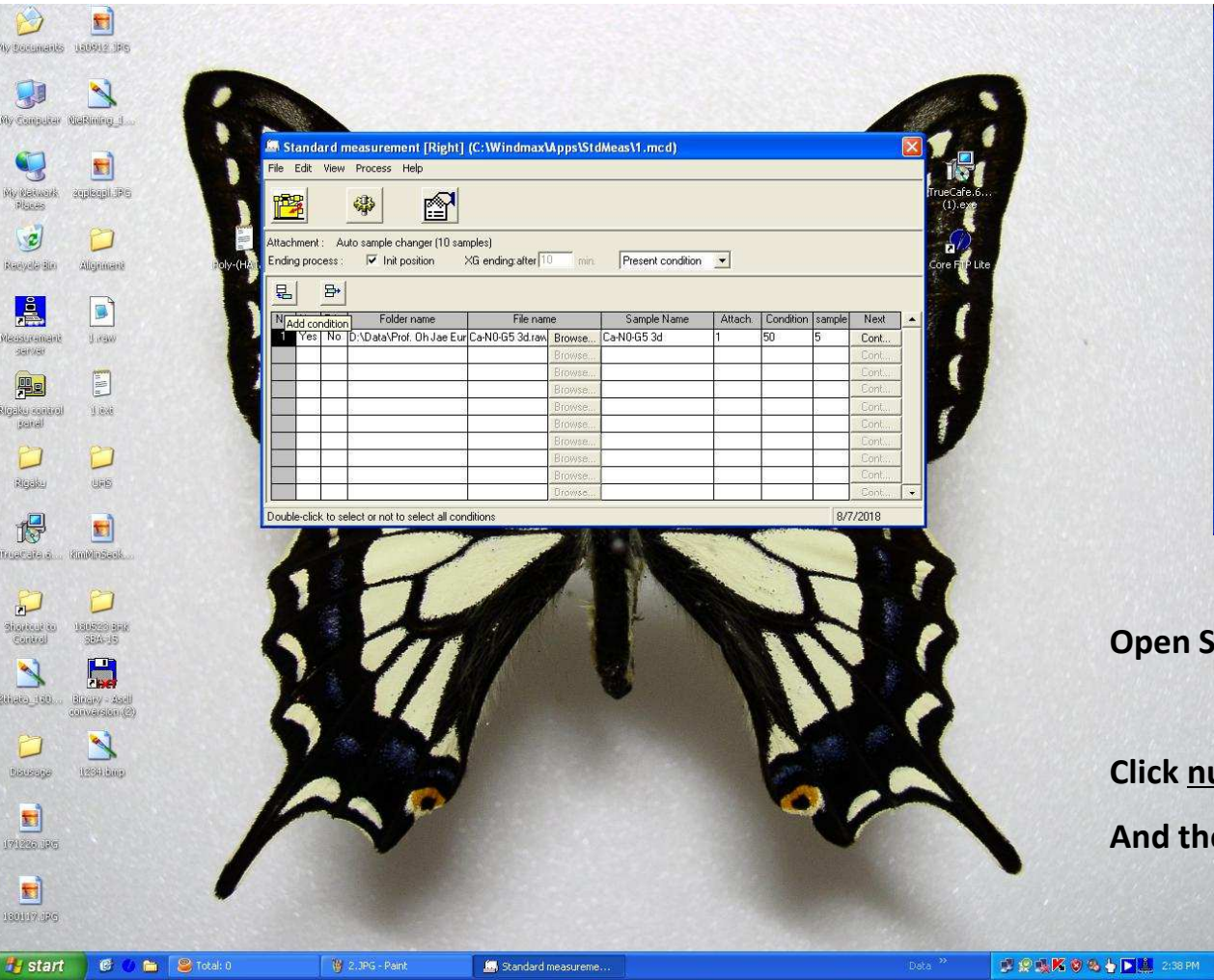
Standard Measurement (Right) measures 10 samples automatically using 'Auto Sample Changer'.

Sample Holder is circle and has 2 small magnetics on the edge.

In case of powder sample, it has 2 types of Radius 18mm, 5mm of Glass Cavity.

In case of thin film or bulk sample, it has 2 types of bottom presence of Al Holder.

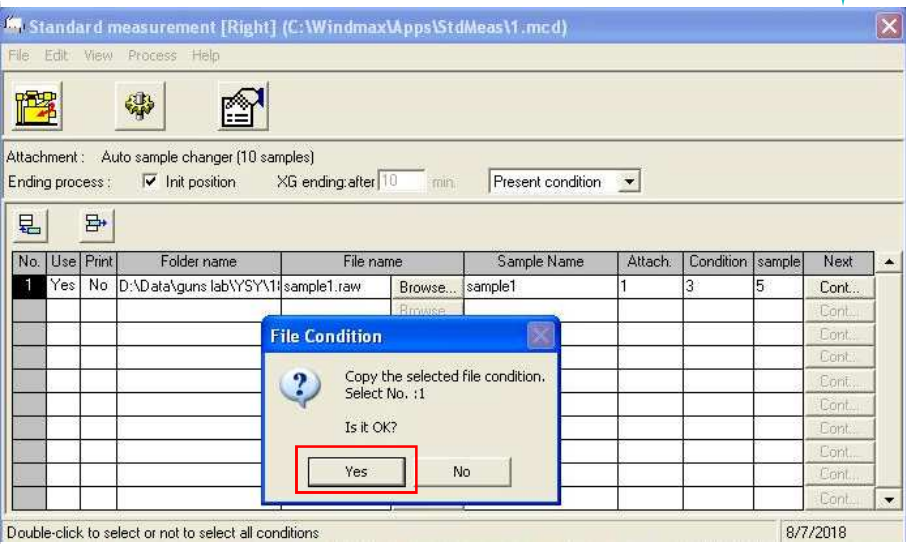
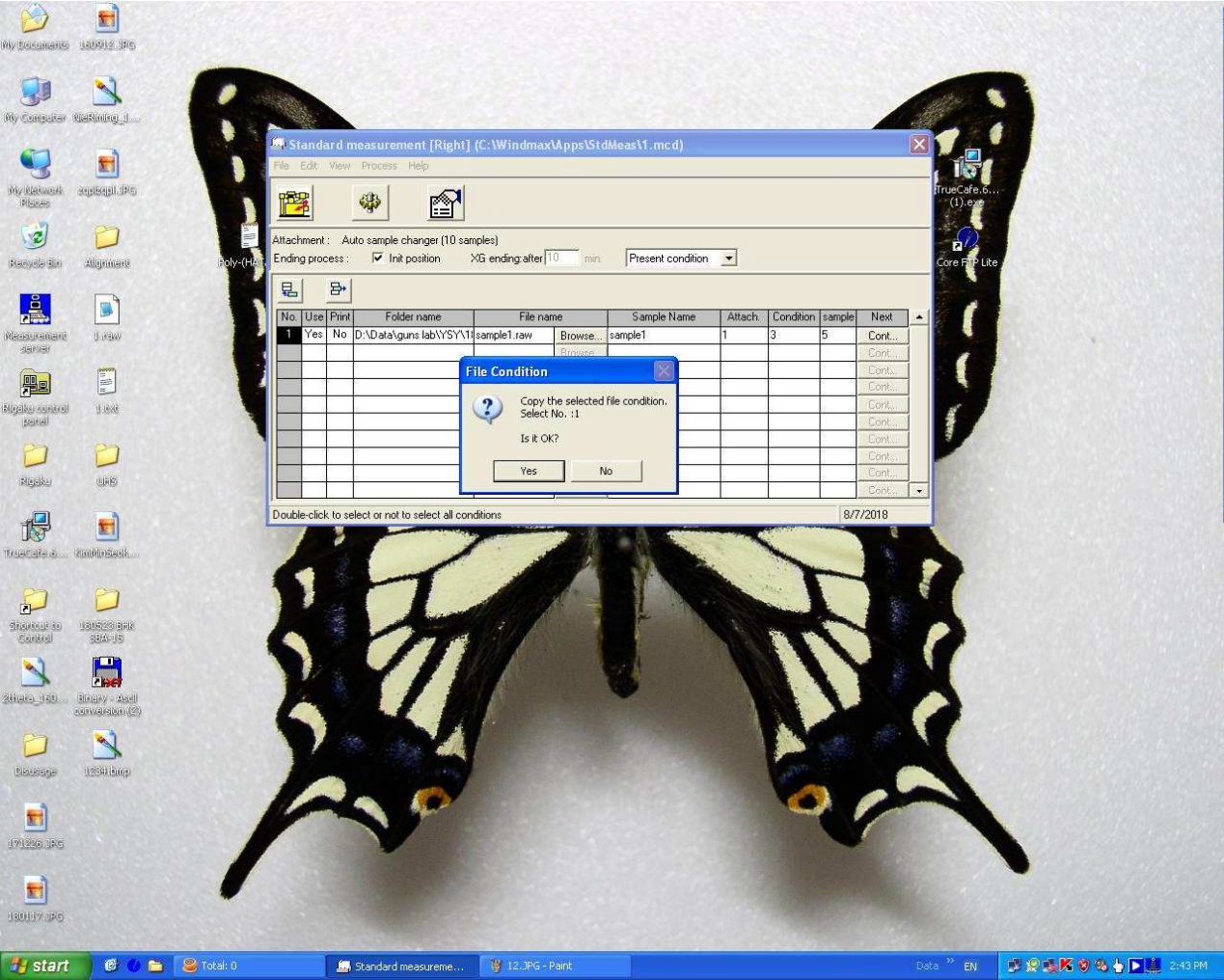
Standard Measurement (Right)



Open Standard Measurement(Right) Window.

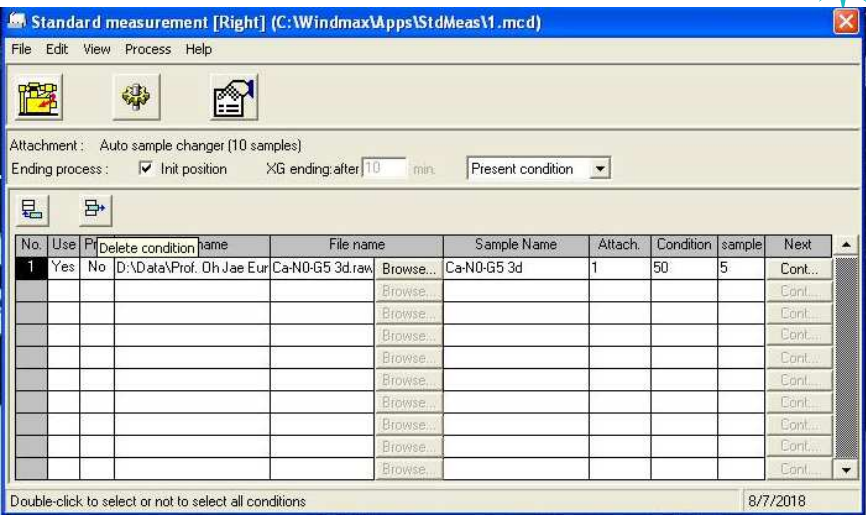
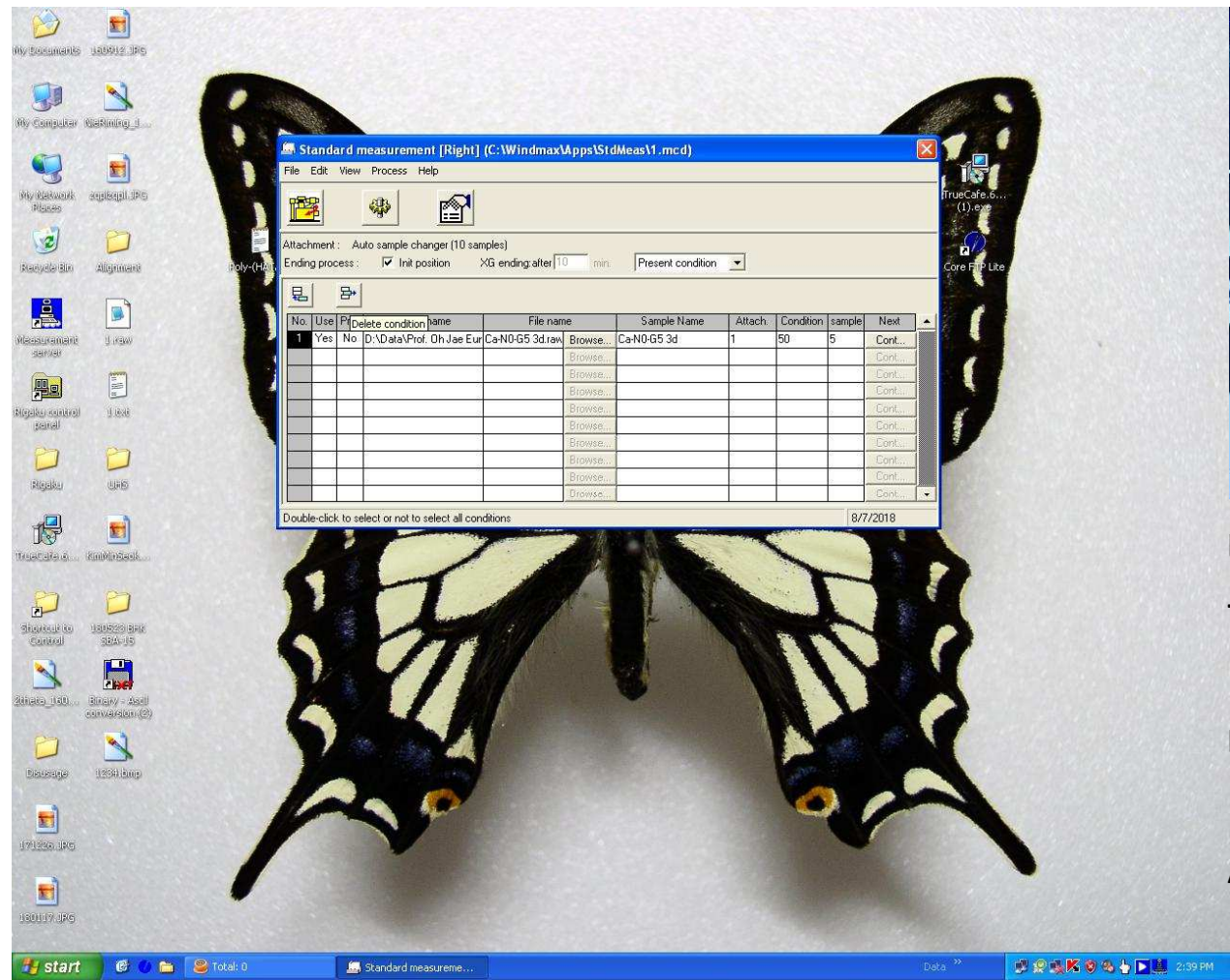
Click number 1 when you measure several samples automatically,
And then click 'Add condition' button.

Standard Measurement (Right)



After opening 'File Condition Window', click 'yes'.

Standard Measurement (Right)

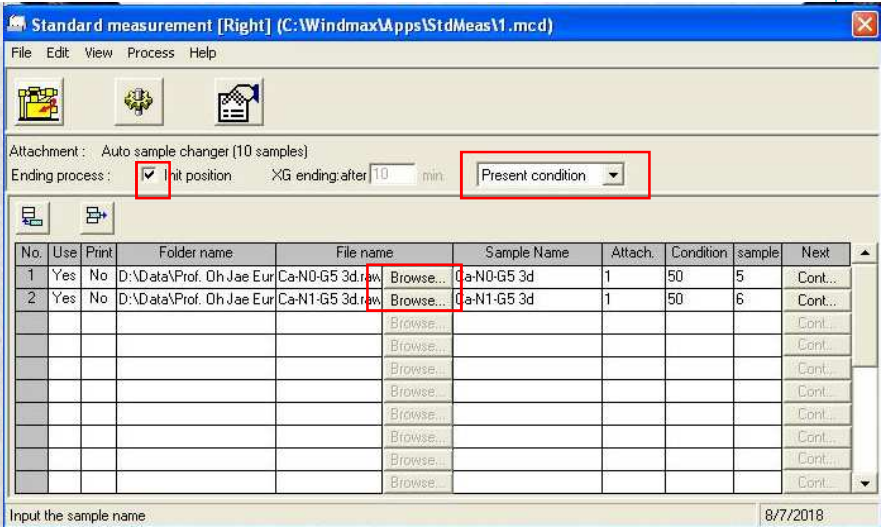
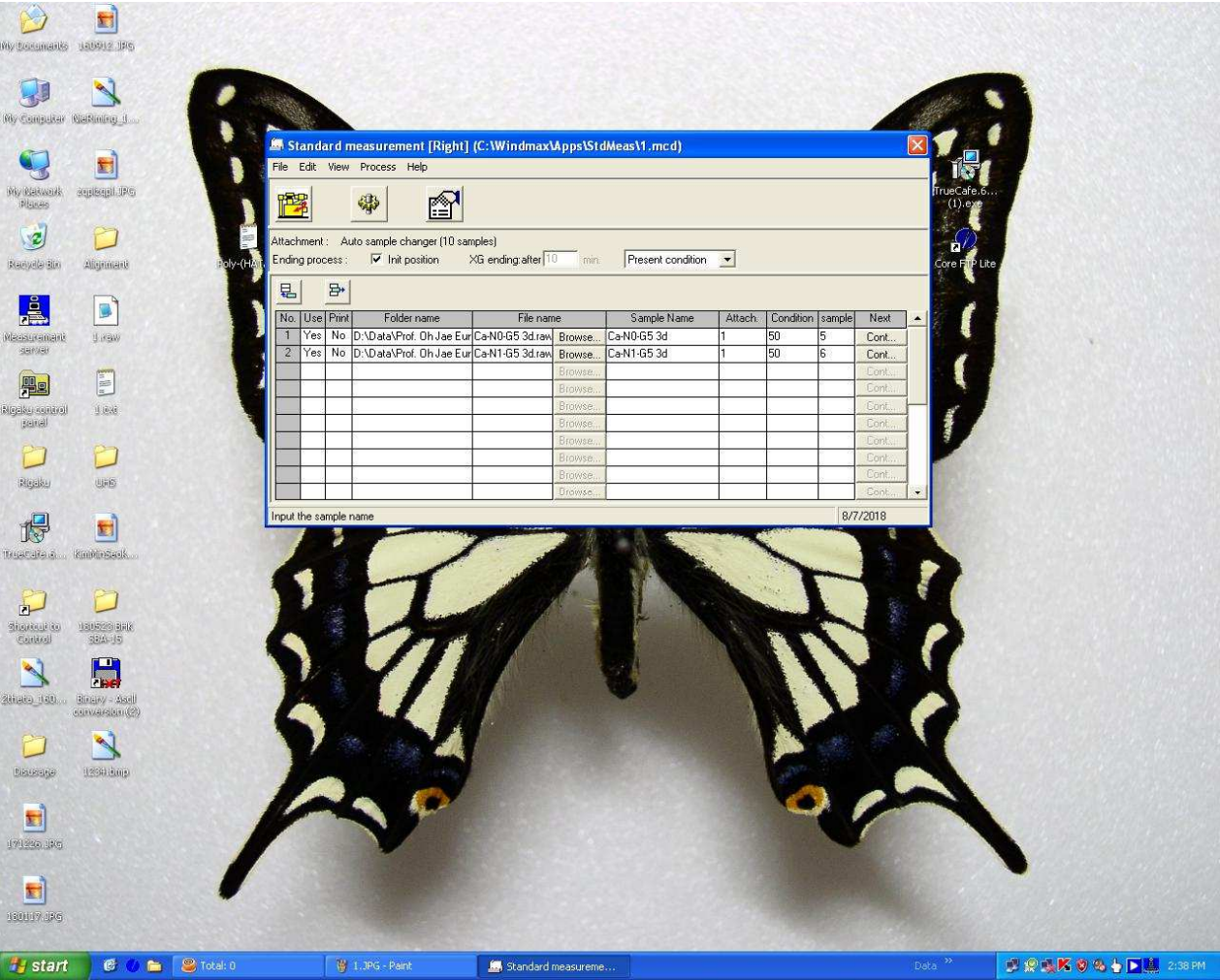


In case of deleting 'Condition', click condition number that you want to delete.

In black status of number field, click 'Delete Condition' button

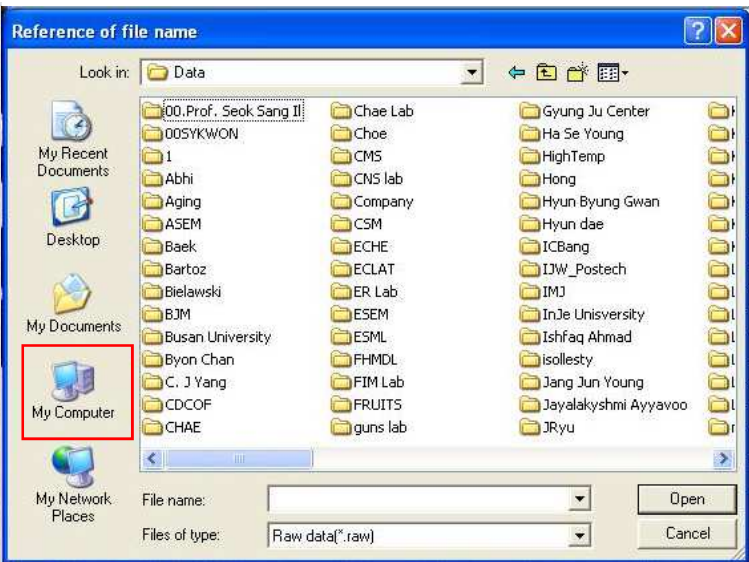
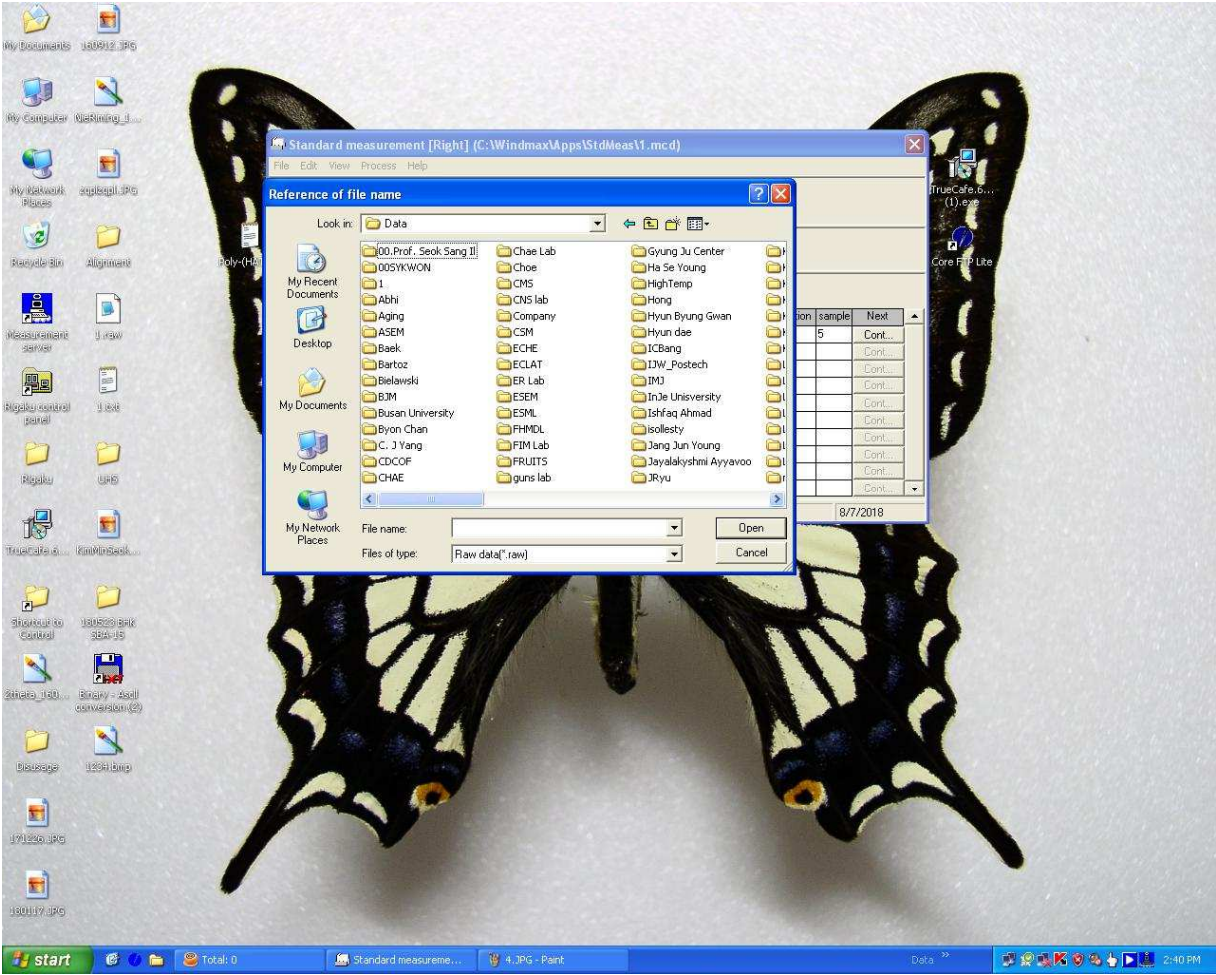
After opening new window, click 'yes'.

Standard Measurement (Right)



While weekday 9:00~18:00,
Check 'Int. position' and 'Present Condition'.
Click 'Browse' button.

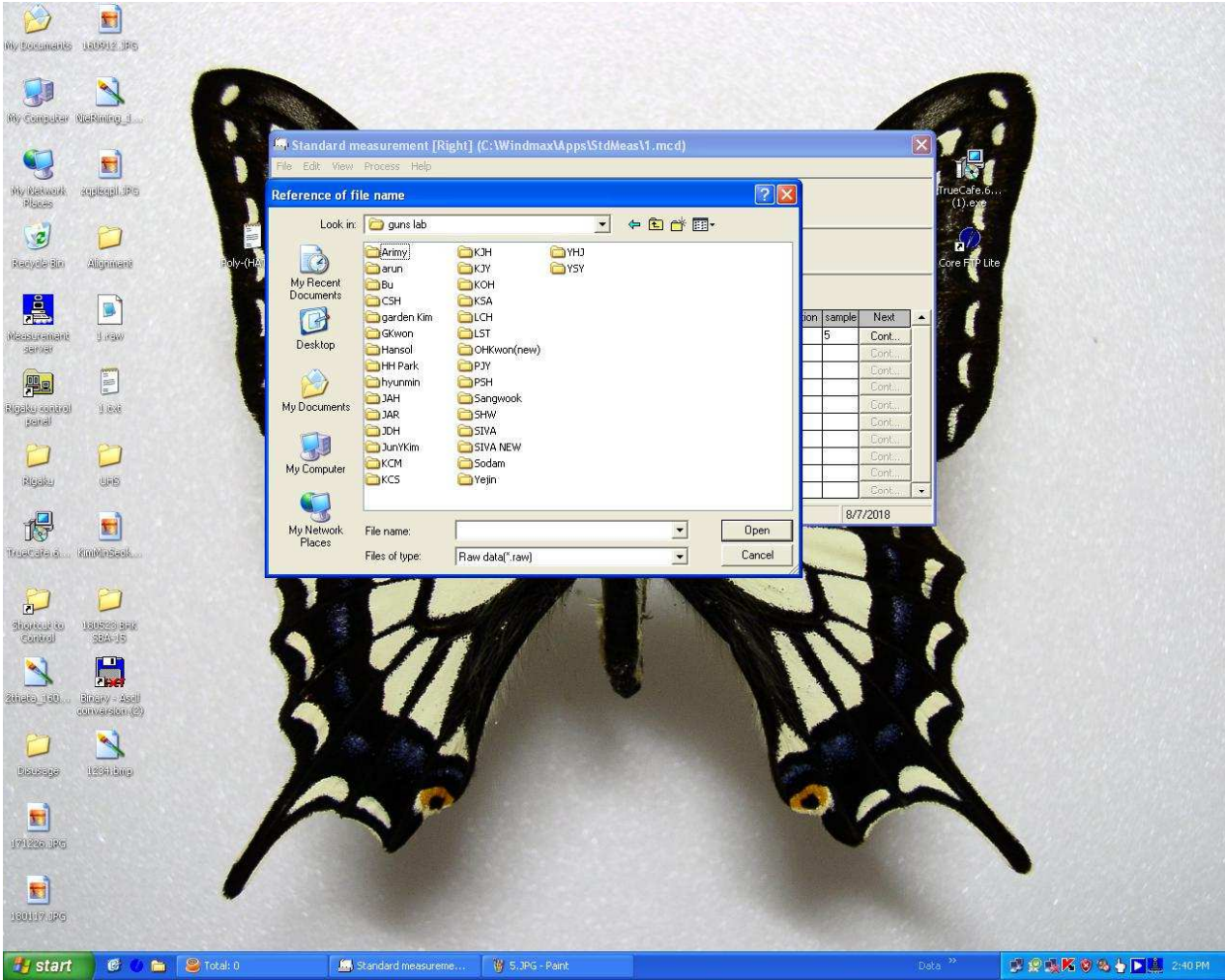
Standard Measurement (Right)



New window opens.

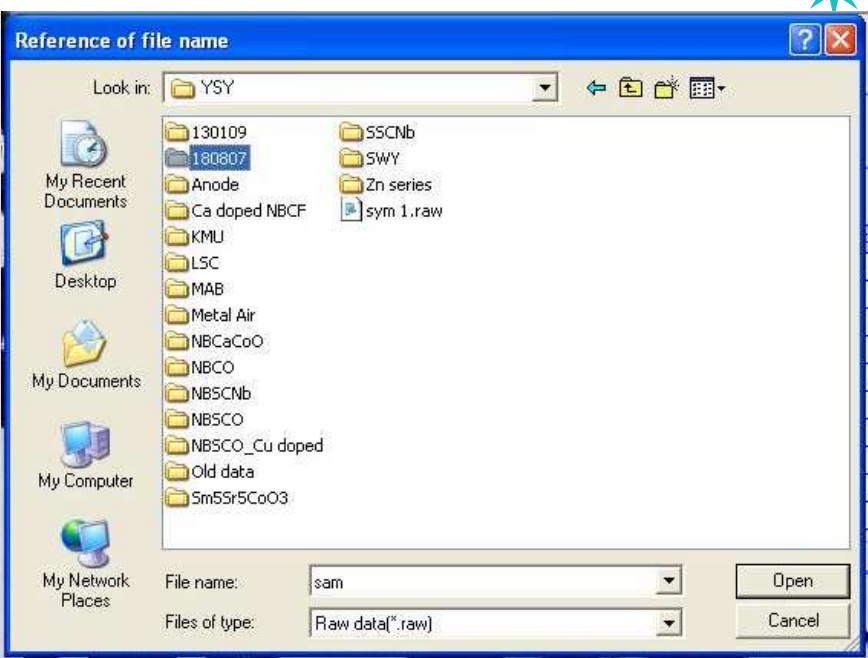
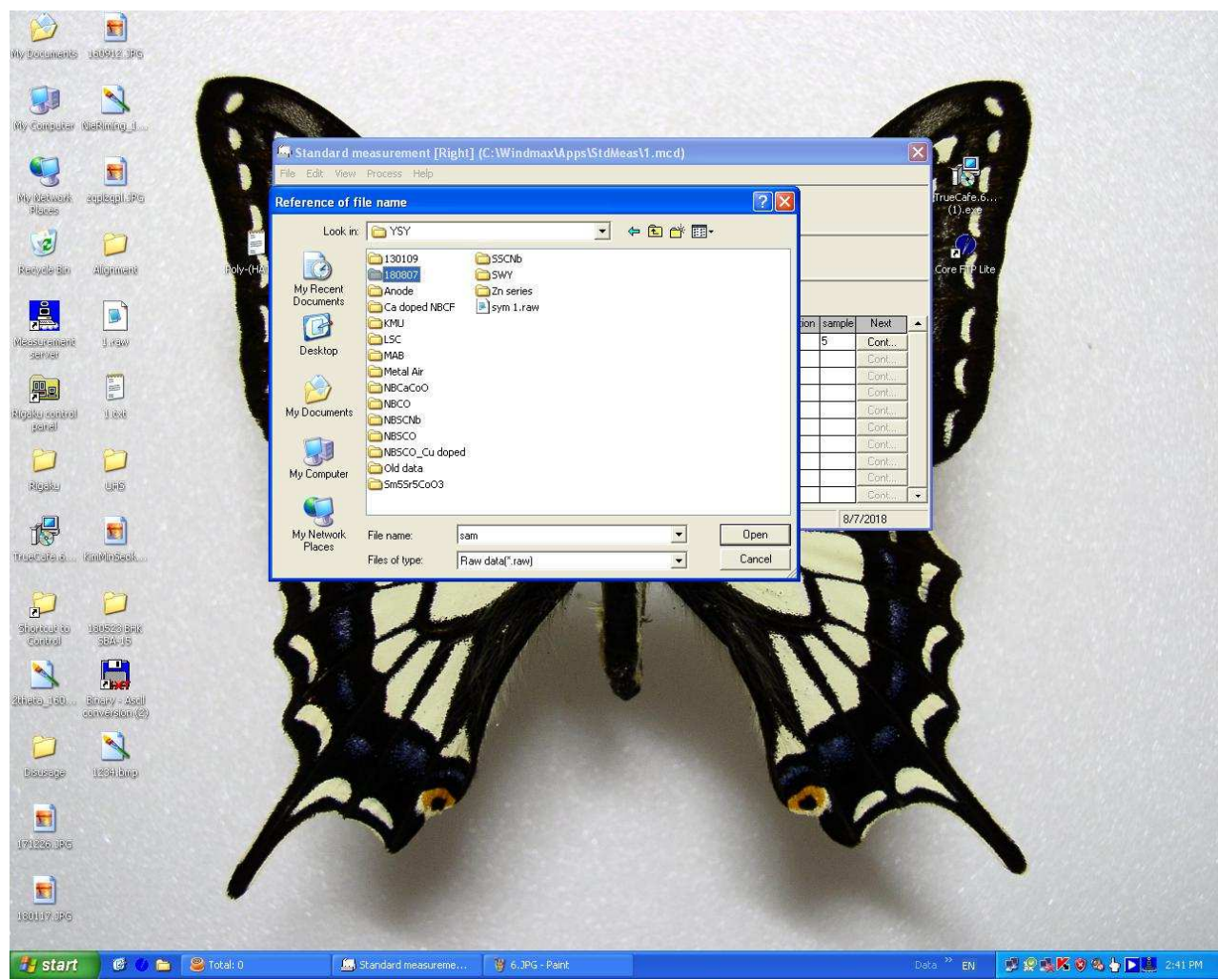
Click 'My Computer' and then click [D Drive > Data].

Standard Measurement (Right)



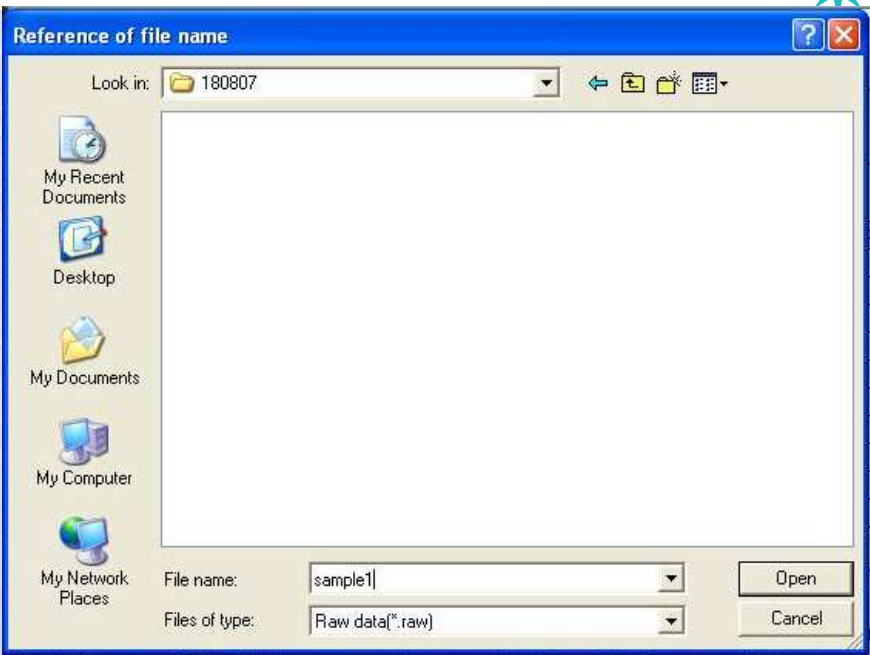
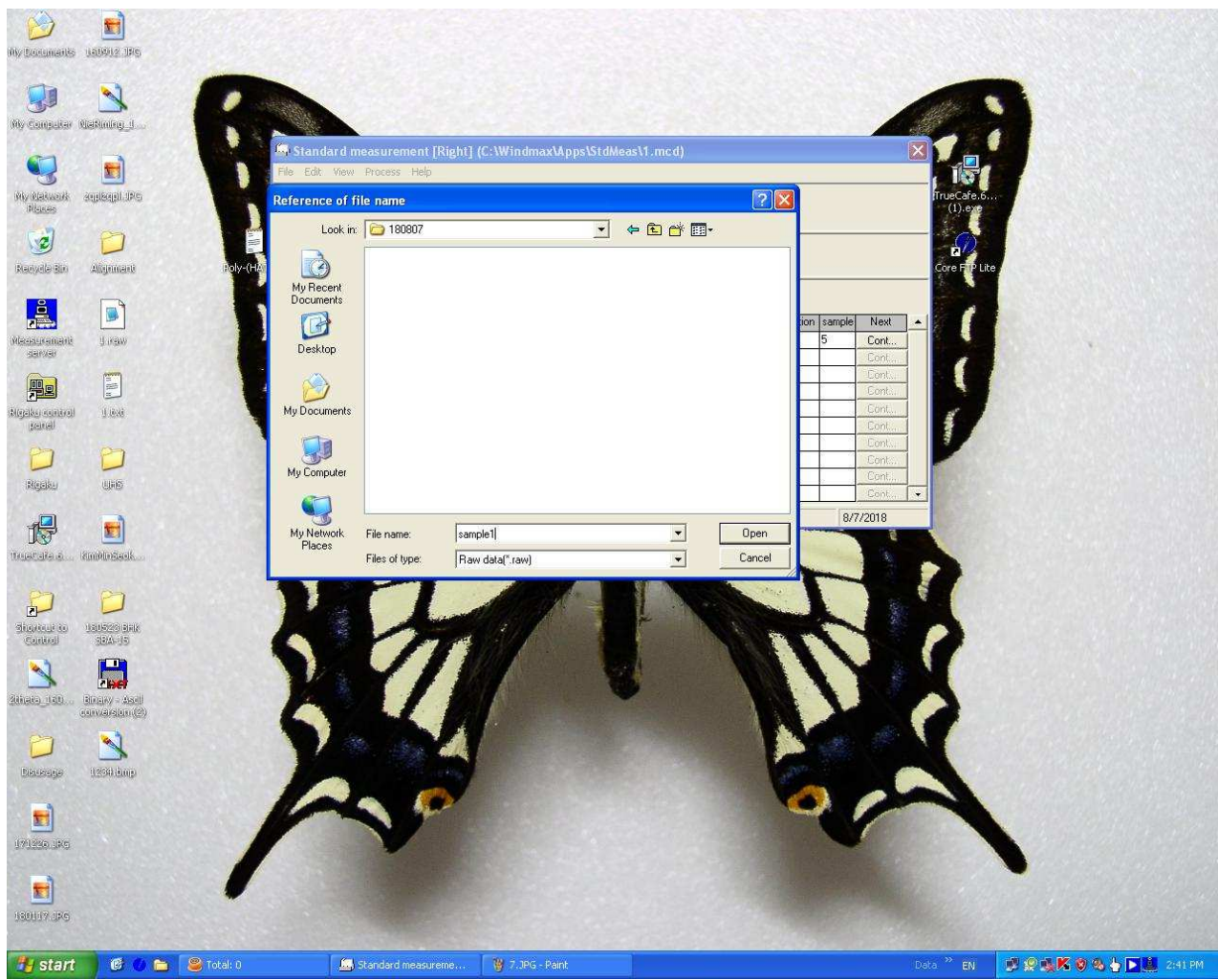
Click [D Drive > Data > **Lab folder**].

Standard Measurement (Right)



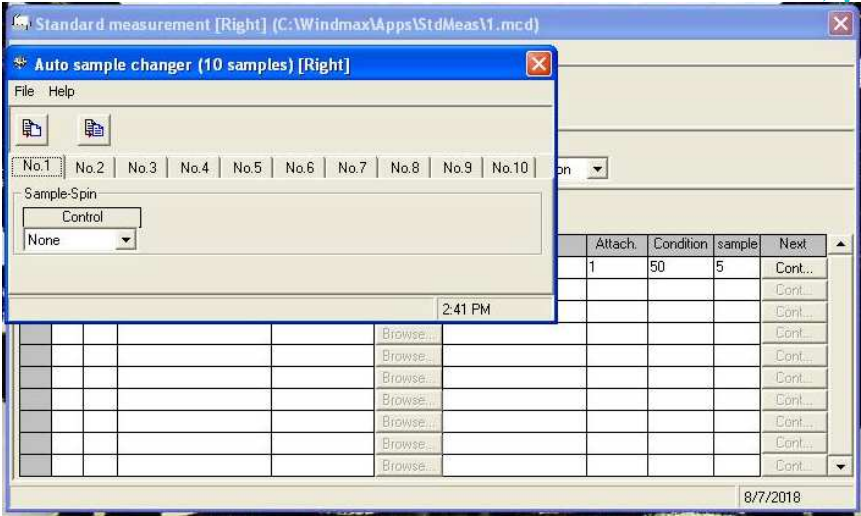
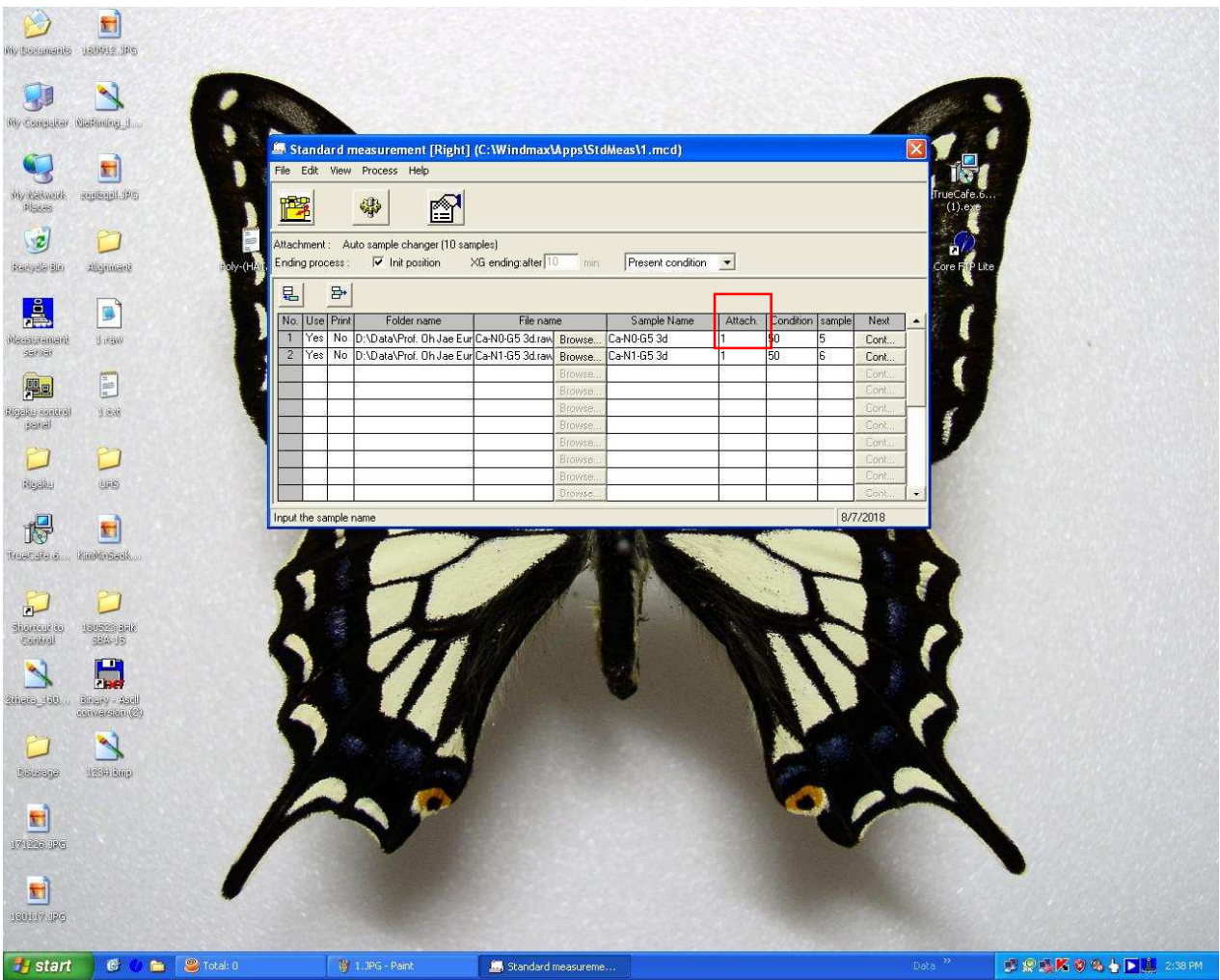
Click [D Drive > Data > Lab folder > **User folder**].

Standard Measurement (Right)



Click [D Drive > Data > Lab folder > User folder > **date**].

Standard Measurement (Right)



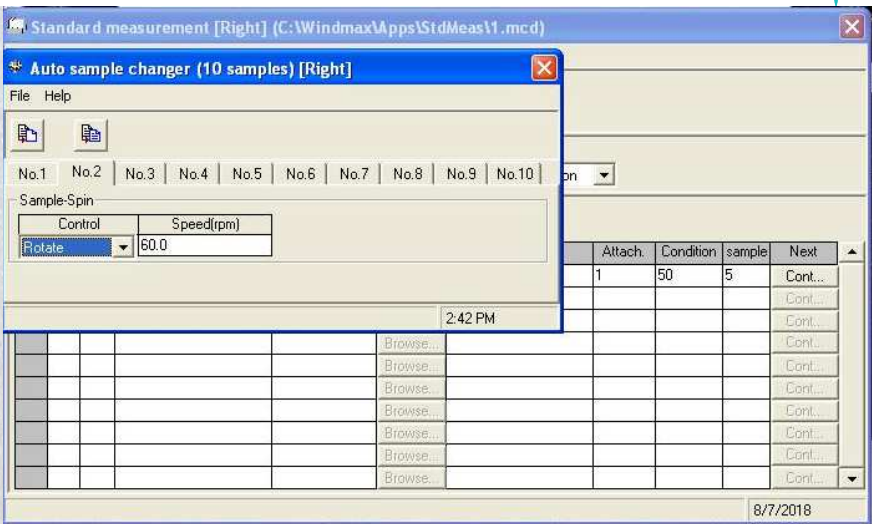
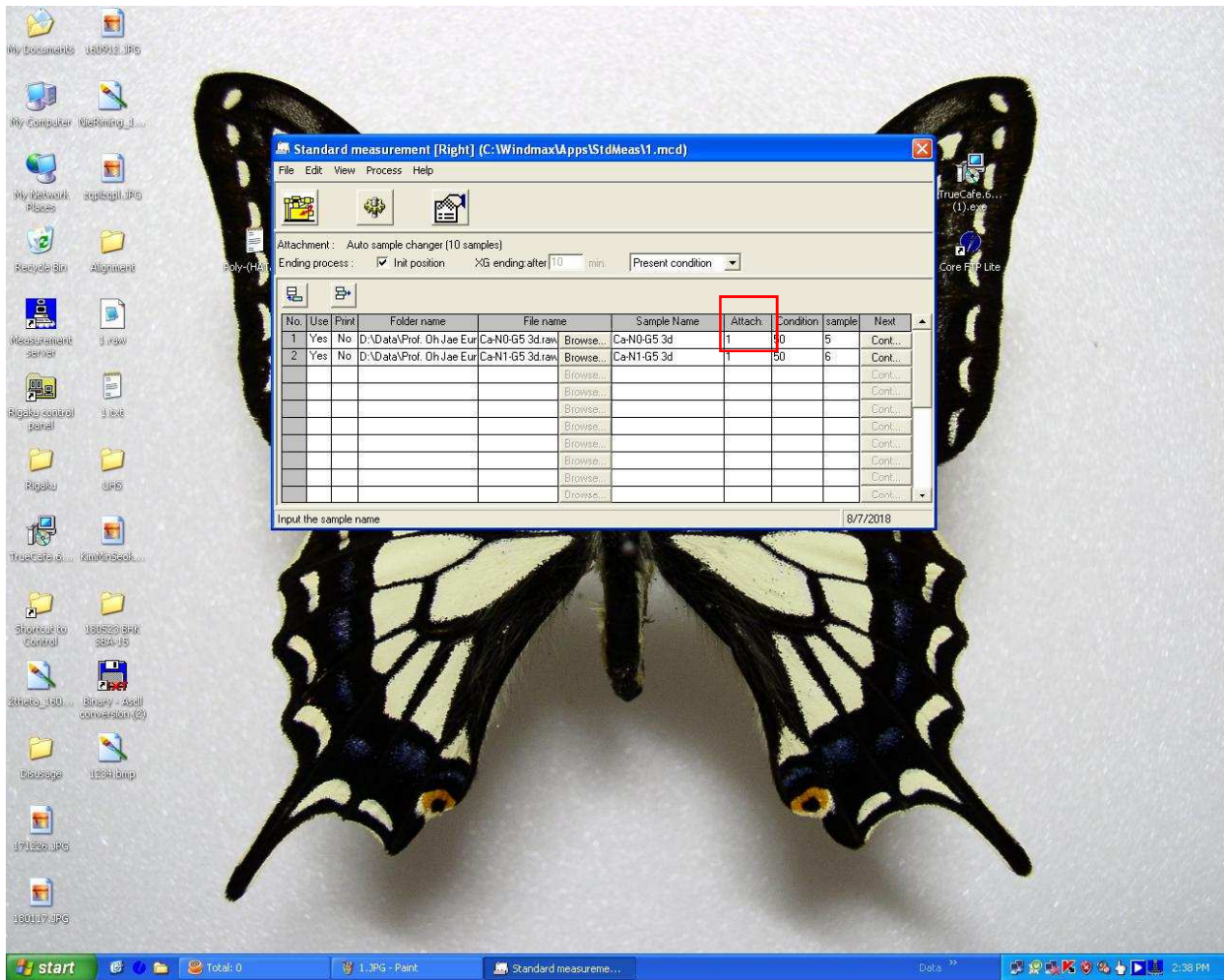
Fill in 'number 1' below Attach and new window opens.

Attach is operation of rotation or non-rotation.

In case of a sample having an orientation property,
It should rotate.

Number 1 is non-rotation.

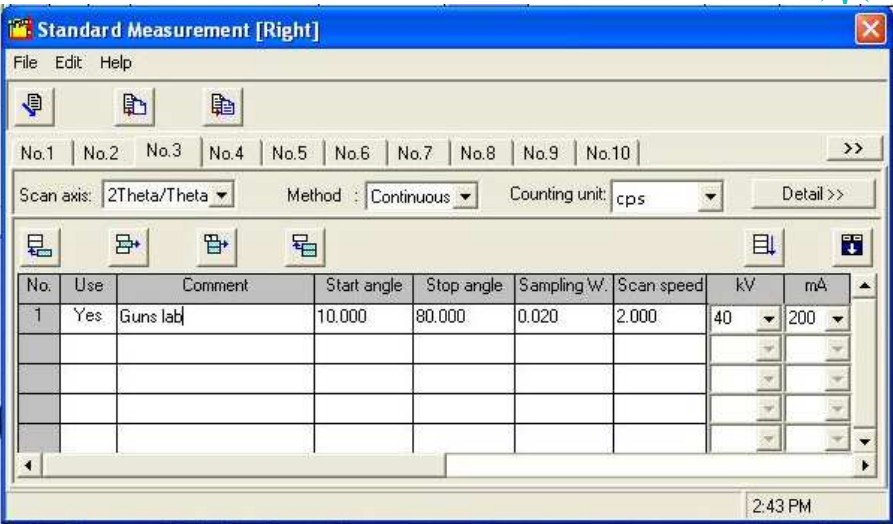
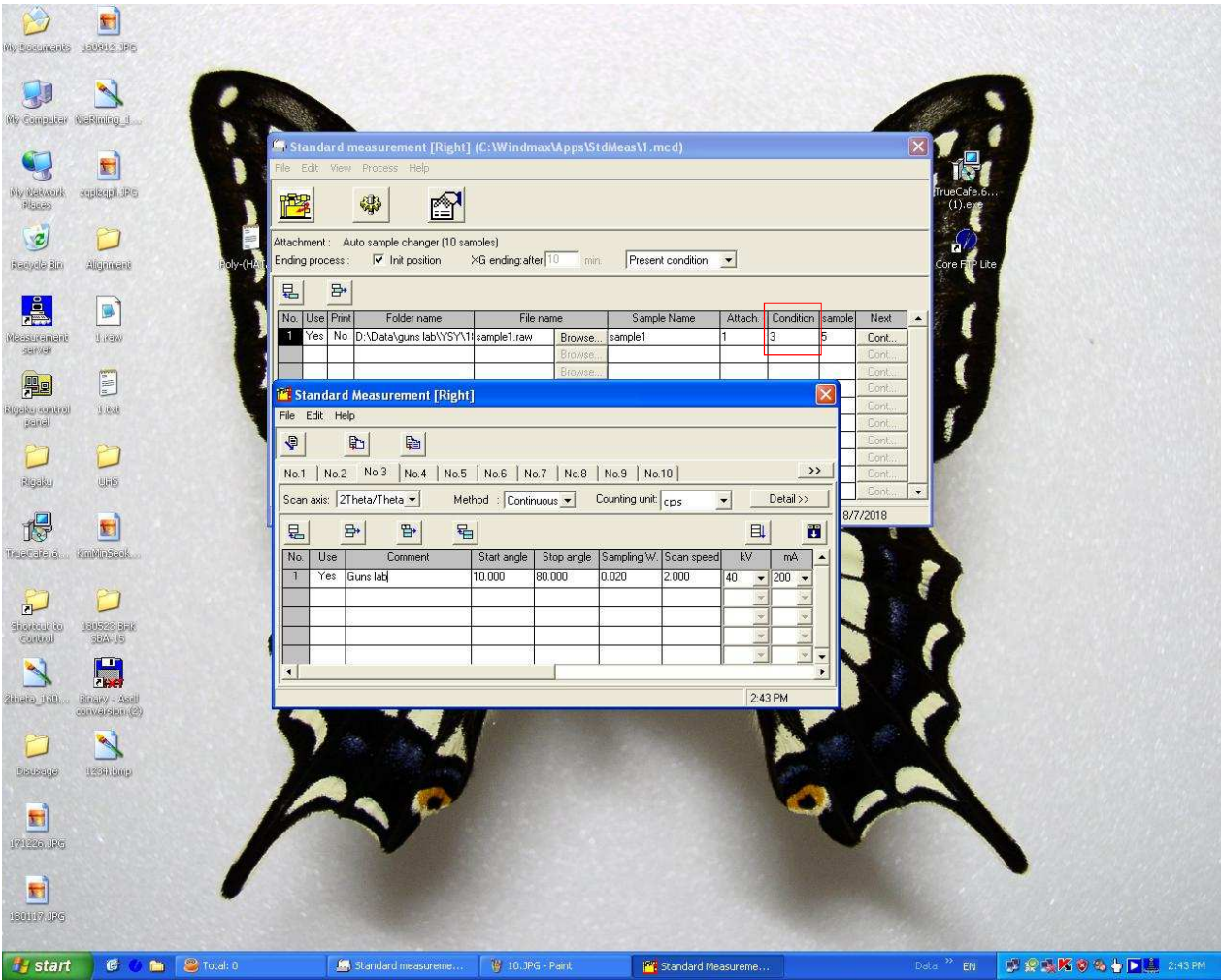
Standard Measurement (Right)



Fill in number 2 below 'Attach' and then,
new window opens.

Number 2 means rotation of 60 RPM.

Standard Measurement (Right)



Next, set the measurement Condition.

Fill in random number of 2~100 except number 1, and then new window opens.

Standard Measurement (Right)



No.	Use	Comment	Start angle	Stop angle	Sampling W.	Scan speed	kV	mA
1	Yes	Guns lab	10.000	80.000	0.020	2.000	40	200

In measurement condition window,

'Use' should be 'Yes'.

Fill in the 'Comment' as easy name to remember.

Fill in Start Angle, Stop Angle, Sampling W(=usually 0.02), Scan Speed(° per minute).

For example, we measure range of 20~ 80° as 2°/min of 0.02° interval, and then

Total measurement time = $80 - 20 = 60^\circ / 2^\circ / \text{min} = 30 \text{ min}$.

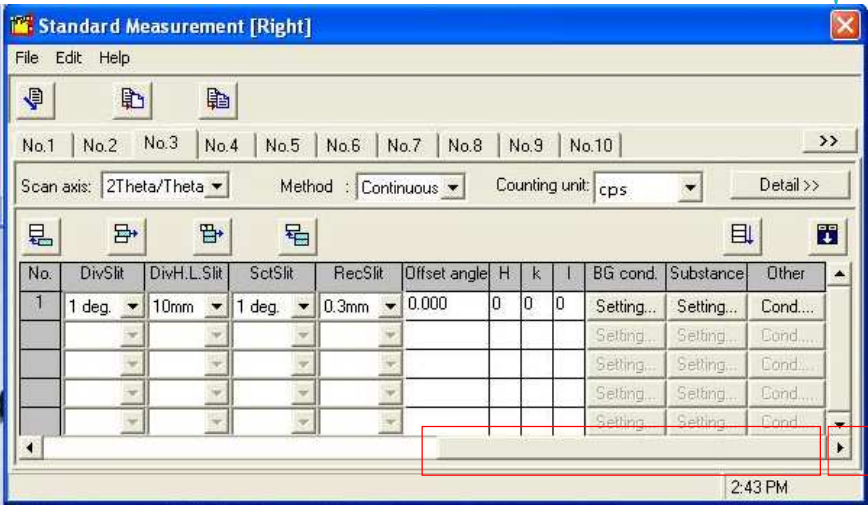
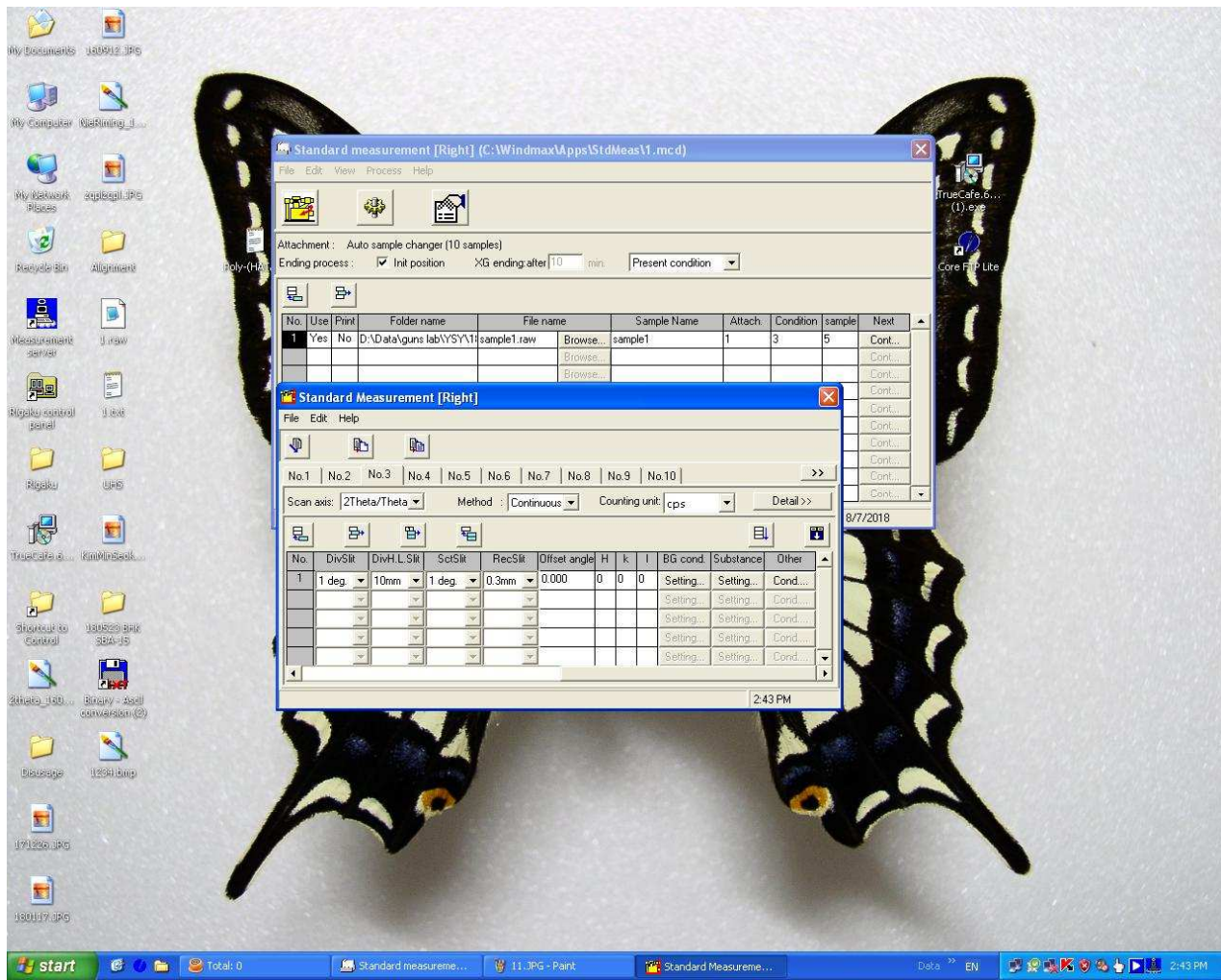
Therefore, self-user can reserve time by calculating measurement time per sample.

Power is regulated by Voltage and Current, and can not be accepted over 40KV, 200mA

In case of exceeding, be careful to occur Filament Short !

As an exception, organic sample can be measured in 40 KV, 30 mA.

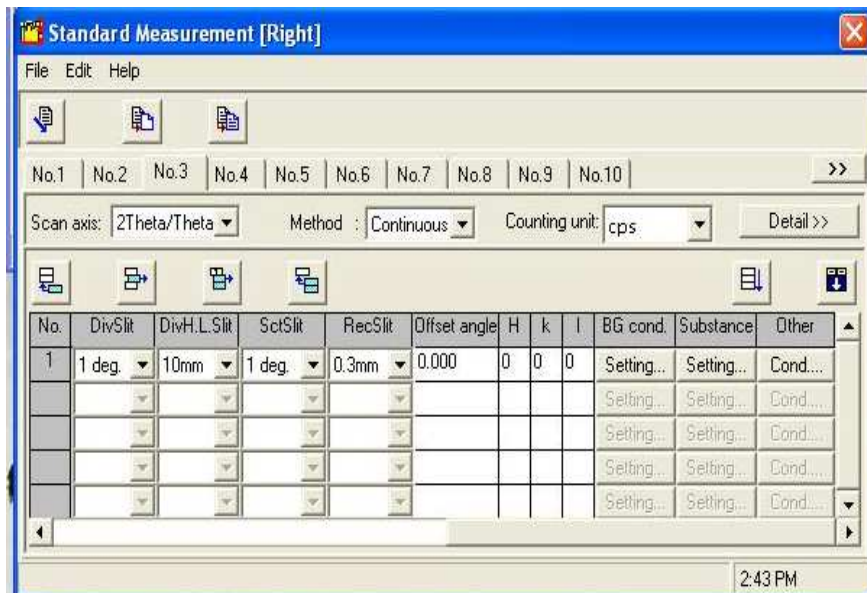
Standard Measurement (Right)



Drag below scroll from left to right.



Standard Measurement (Right)



DivSlit (Divergence Slit) and ScaSlit(Scattering Slit) mean X-ray Beam Depth.

Both Slit have same value.

DivHLSlit(Divergence Height Lenth Slit)means X-ray Beam Height.

Therefore, it could change slit condition by sample holder.

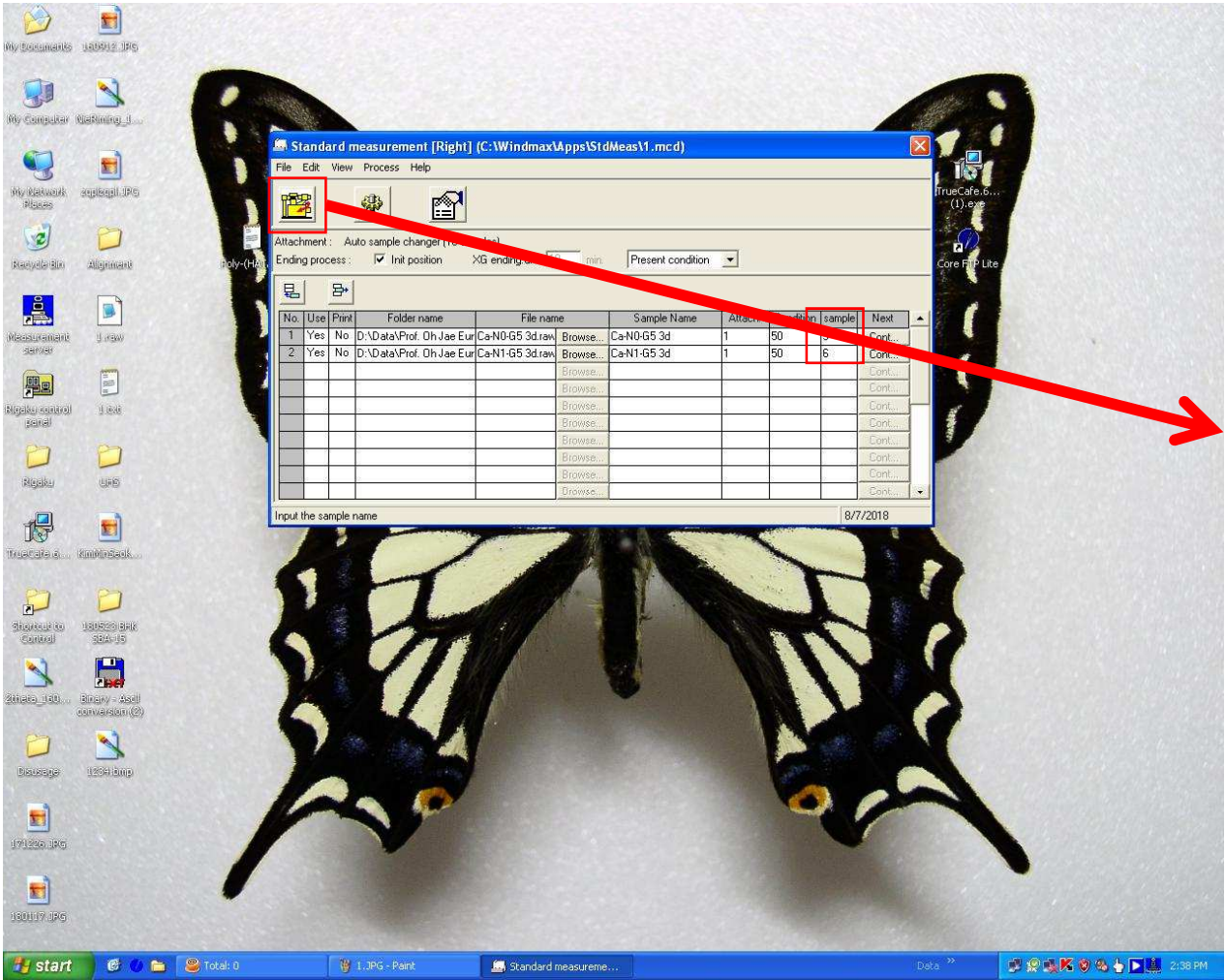
X-ray Beam Width changes widely in small angle and narrowly in high angle according to $\sin\theta$ and control is not possible as Slit.

RecSlit(Receiving Slit) set usually 0.3 mm.

If the peak overlap is severe, it changes smaller value and measure.

Sample Holder	Div Slit	DivHLSlit	Sca Slit	RecSlit
18 mm	1 deg	10 mm	1 deg	0.3 mm
5 mm	½ deg	5 mm	½ deg	0.3 mm

Standard Measurement (Right)



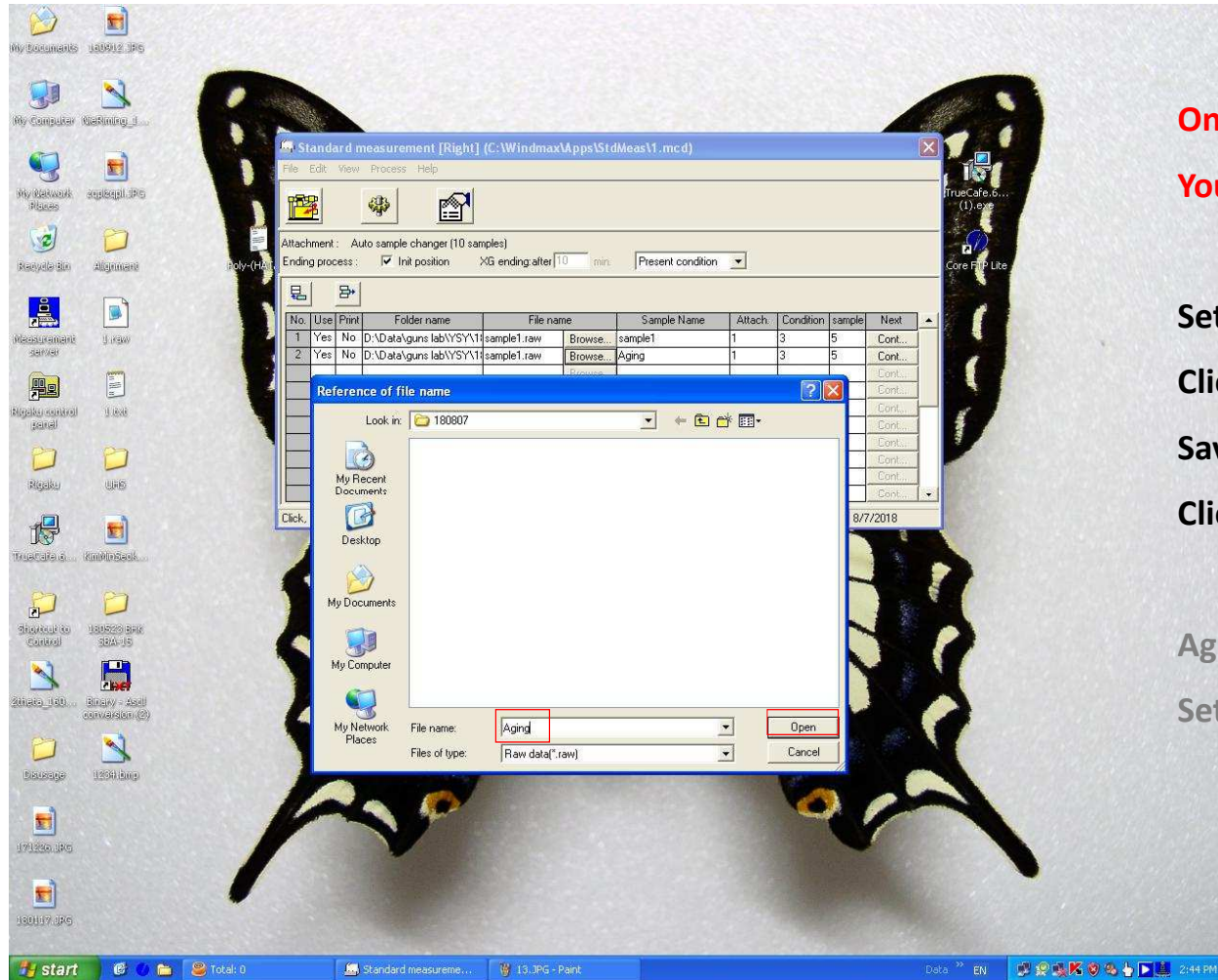
Pos means Sample Position in ASC.

ASC has total 10 Sample Positions.

While Day Time (weekday 9:00~18:00),

It does not need 'Aging Process' so
click 'Execute' button.

Aging Process



On [weekdays 9:00~18:00], it does not need Aging process.

You should set Aging process on a holiday or evening.

Set the measurement condition and Copy condition below field.

Click 'Browse' button and name file 'Aging' and then

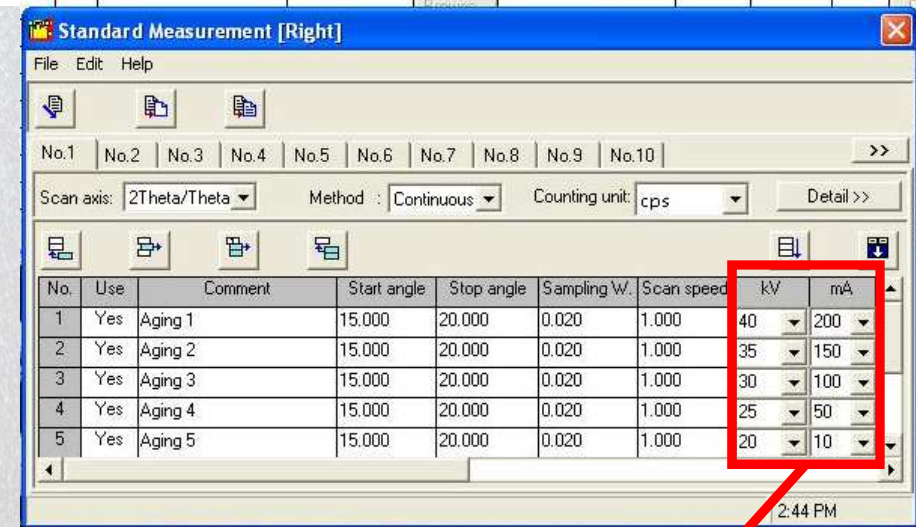
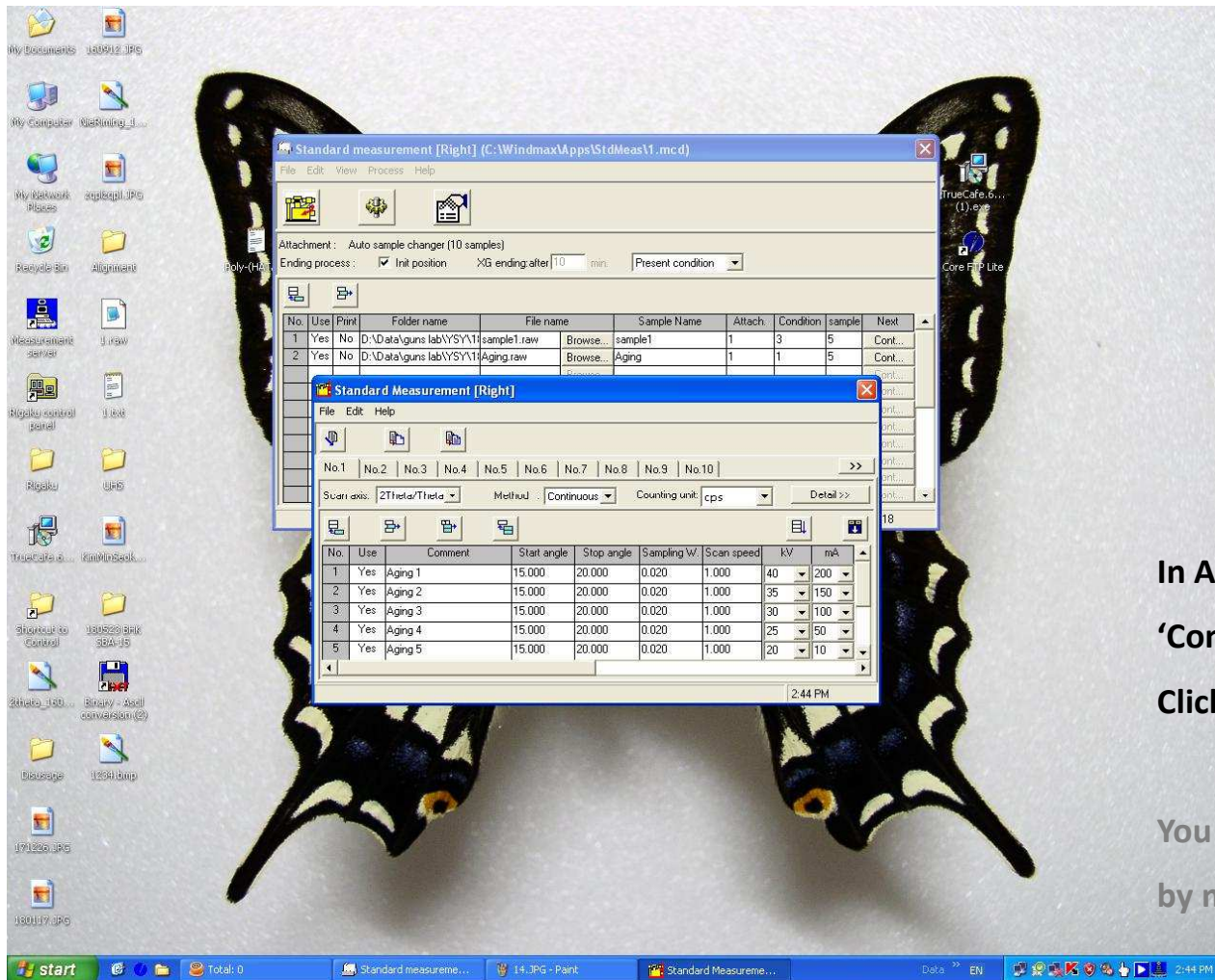
Save as same location with sample file.

Click 'Open' button.

Aging data has no meaning so

Set same 'Attach' and 'Pos' value with sample.

Aging Process



In Aging Process, important thing is !

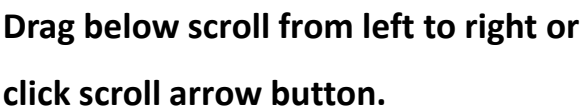
'Condition' value should be '1'.

Click the 'Condition value' and new window opens.

You can see 'No.1' to lower power stably

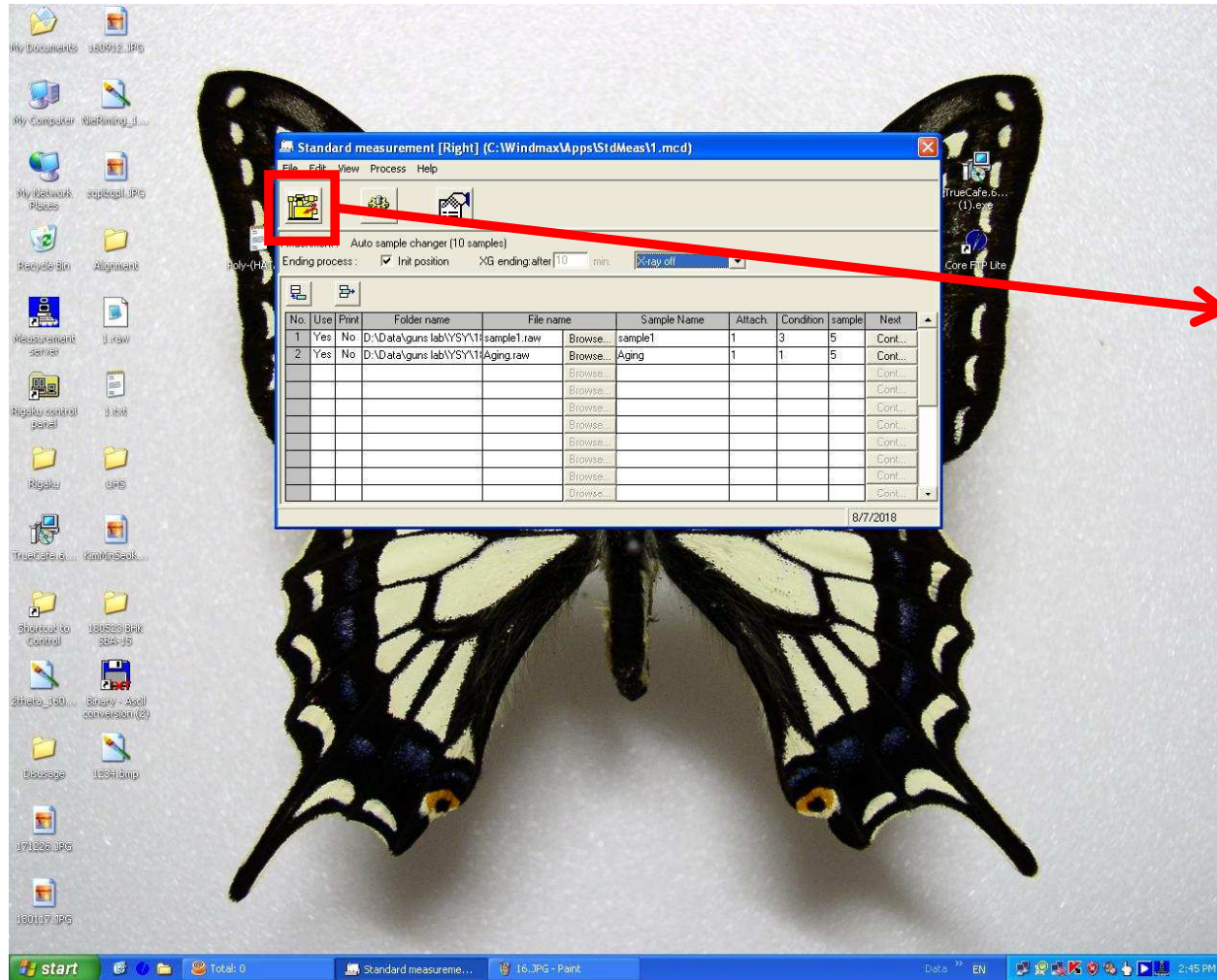
by making low temp. of 15~20 degree (low angle) repeatedly.

The screenshot shows a Windows XP desktop environment. The background is a wallpaper of a butterfly. The desktop is populated with numerous icons, including system folders like 'My Documents', 'My Computer', 'My Network Places', 'Recycle Bin', and 'Alignment'. There are also application icons such as 'Measurement series', 'Rigaku control panel', 'Rigaku', 'TrueCafe 6...', 'Sketch to control', '2theta_180...', 'Dsource', and several image files like '171226.JPG' and '180117.JPG'. Two application windows are open. The first window, titled 'Standard measurement [Right] (C:\Windmax\apps\StdMeasV1.mcd)', has a menu bar with 'File', 'Edit', 'View', 'Process', and 'Help'. It contains a toolbar with icons for file operations and a table with columns: 'No.', 'Use', 'Print', 'Folder name', 'File name', 'Sample Name', 'Attach', 'Condition', 'sample', and 'Next'. The table lists two entries. The second window, titled 'Standard Measurement [Right]', also has a menu bar with 'File', 'Edit', and 'Help'. It features a toolbar, a row of buttons labeled 'No.1' through 'No.10', a 'Scan axis' dropdown set to '2Theta/Theta', a 'Method' dropdown set to 'Continuous', a 'Counting unit' dropdown set to 'cps', and a 'Detail >>' button. Below these is an 'Input' section with a table that has columns: 'No.', 'DivSR', 'DivH.LSR', 'ScSR', 'RecSR', 'Offset angle', 'H', 'k', 'l', 'BG cond', 'Substance', and 'Other'. This table contains five rows of data. The taskbar at the bottom includes the Start button, icons for 'Total: 0', 'Standard measure...', '15.JPG - Paint', and 'Standard Measureme...', along with the system clock showing '2:44 PM'.



Click 'finish button' after checking conditions.

Aging Process



For Aging process condition,

Change 'Present Condition' to '**X-ray off**' located next to 'XG ending after'.

And then, click '**Execution measurement**' button.

After measuring 'No.1 condition',

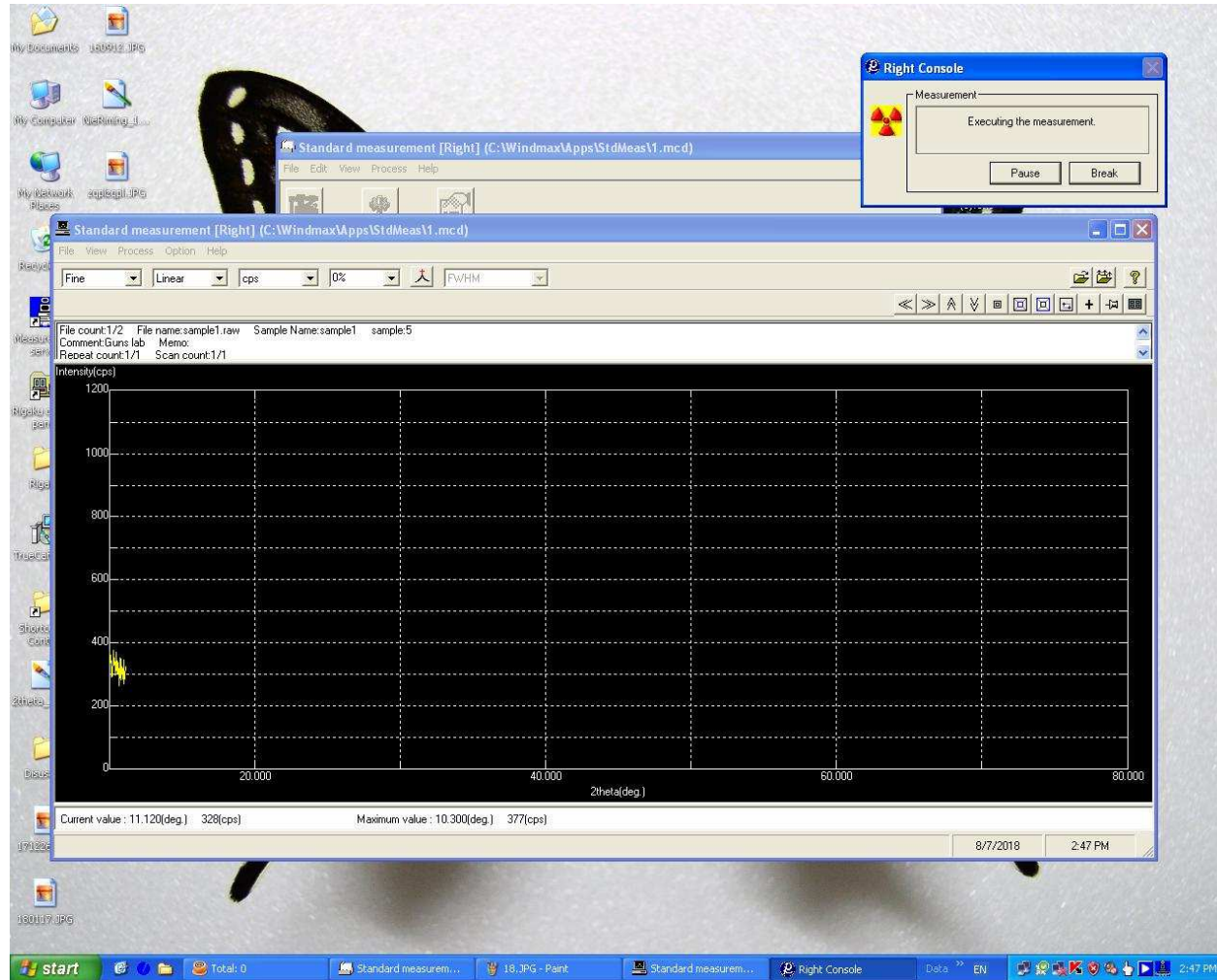
lower power for 10min and then do X-ray off as No.2 condition.

In case of measuring at night or holiday,

It can measure and do X-ray off automatically

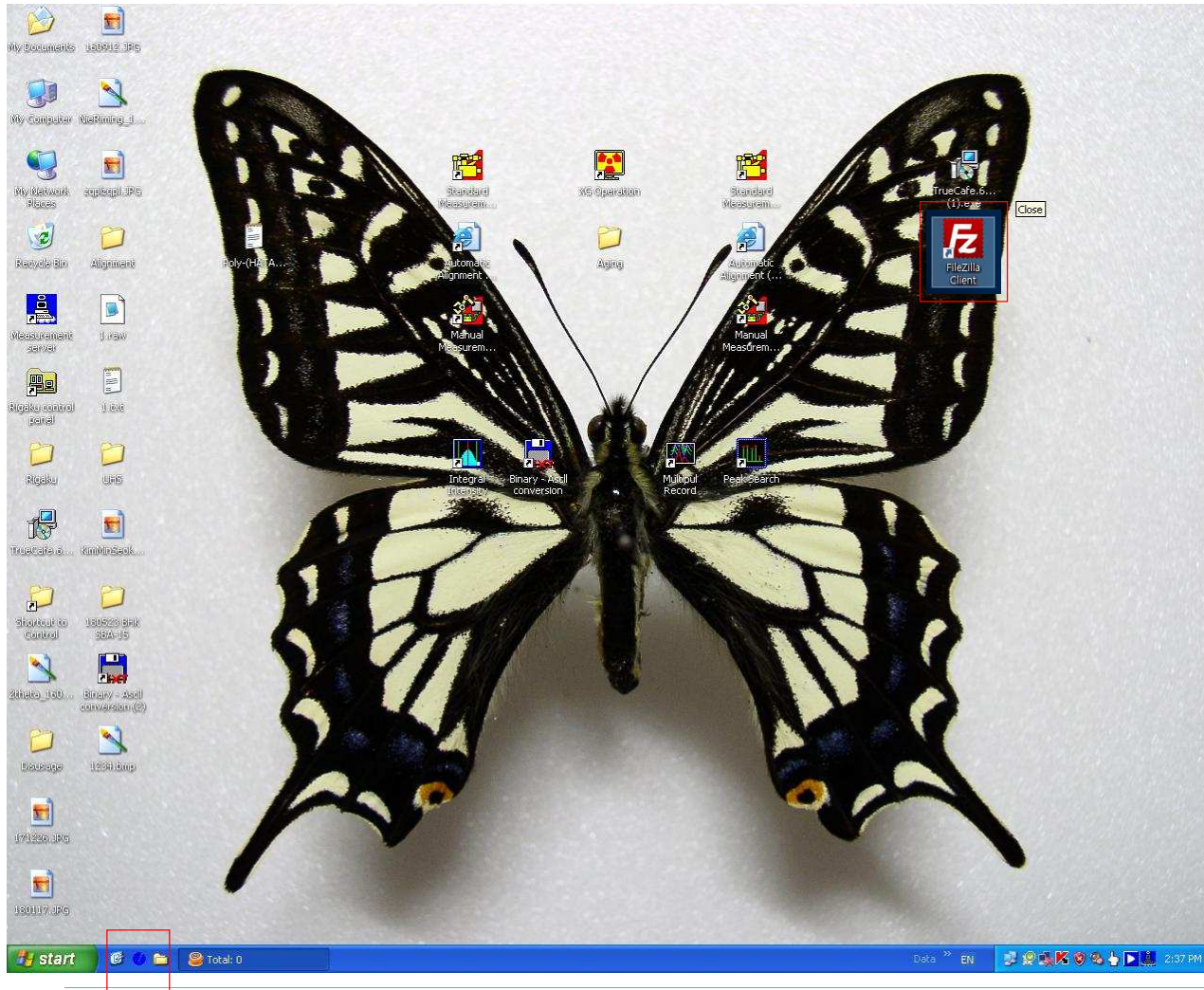
Without setting Aging process.

Measurement



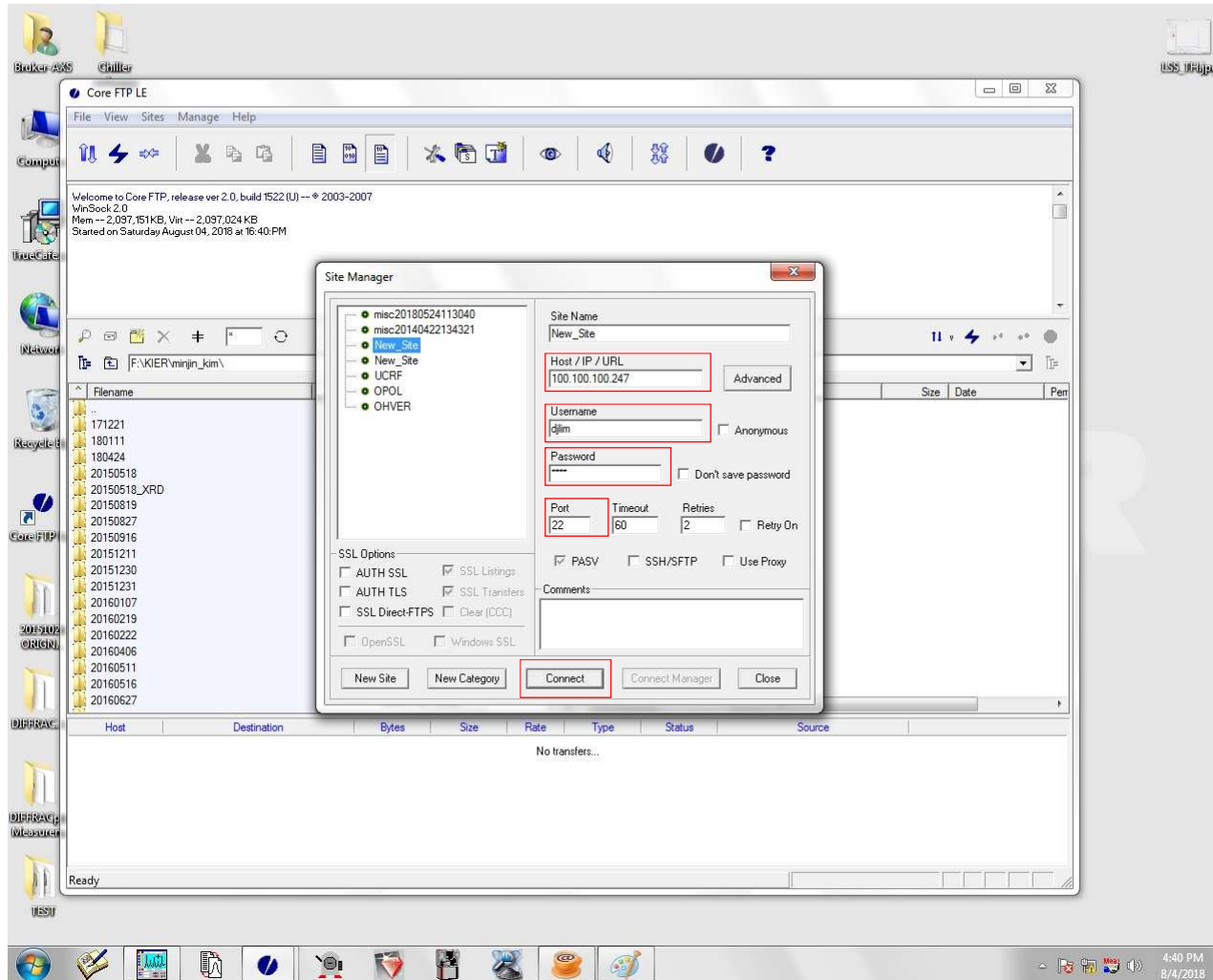
After starting measurement,
It does not matter you to leave XRD room
If data shows good.

Data Upload (FileZilla)



FileZilla is downloadable program without using USB.
Double click one of both.

Data Upload (FileZilla)



IP: 100.100.100.247,

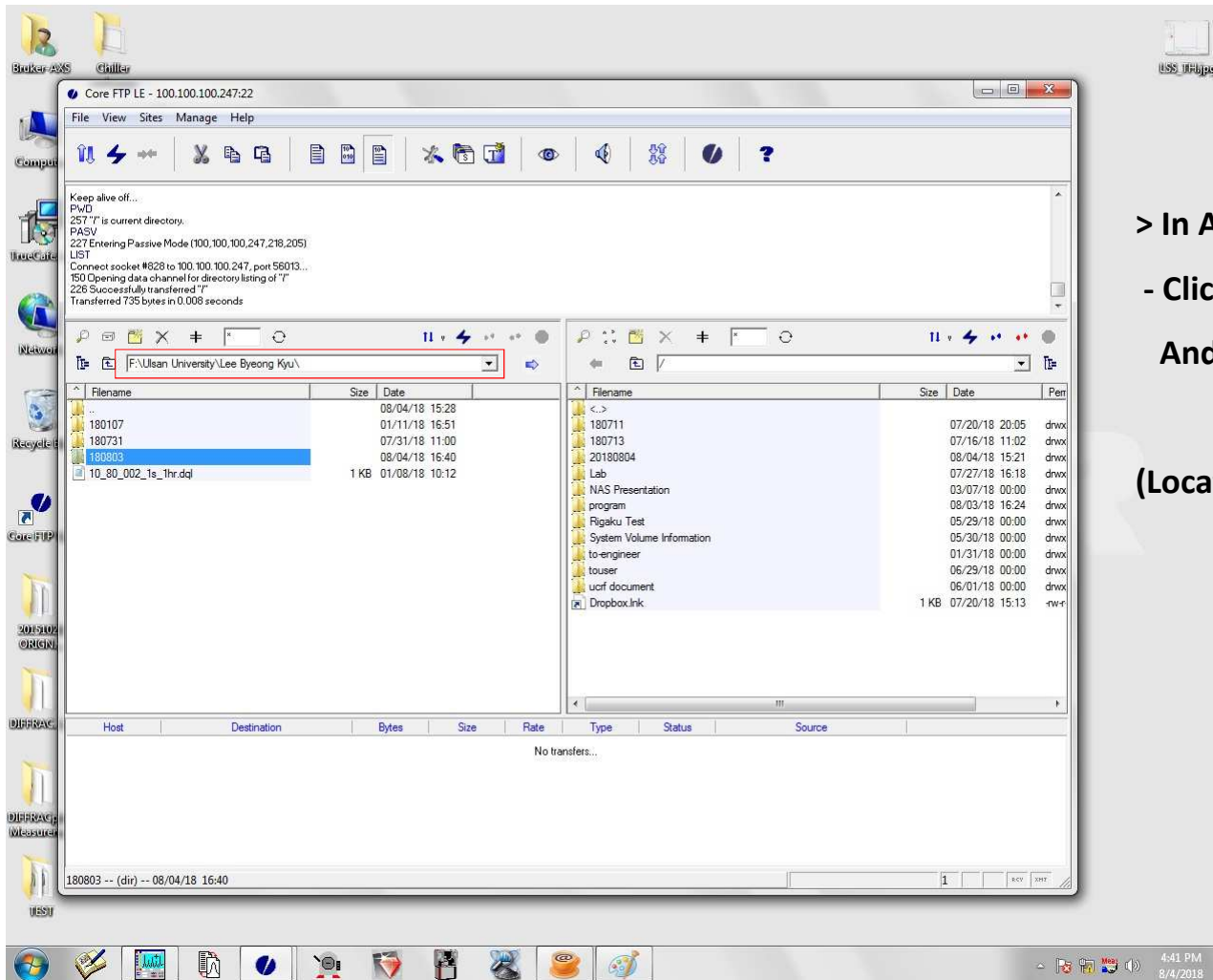
Username: professor name,

Password:

Port: 22

And then, Click 'Connect'

Data Upload (FileZilla)



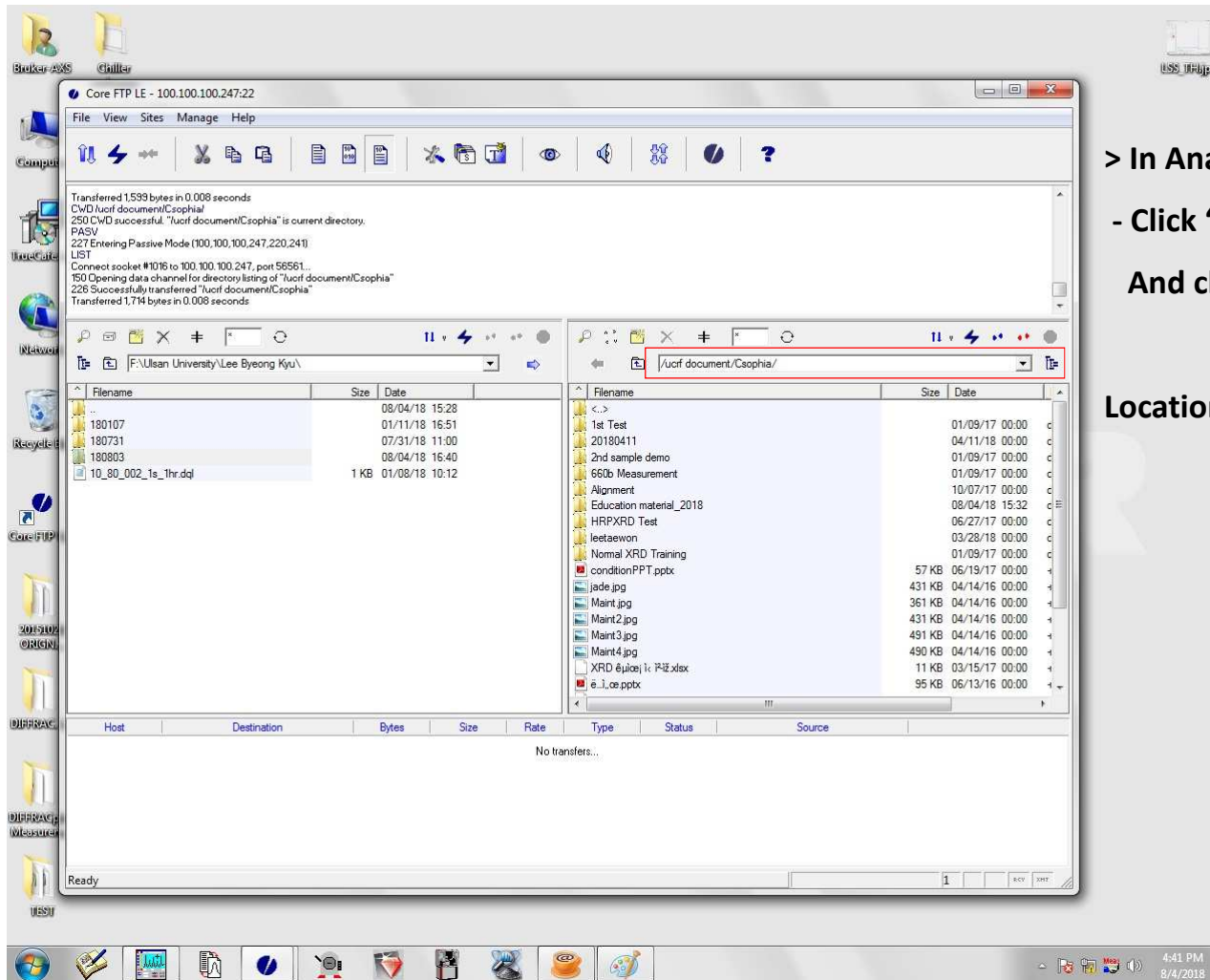
> In Analysis PC

- Click 'Browser' (Left page)

And check file location that you saved.

(Location: F:/Lab name/Requester name/Analysis date)

Data Upload (FileZilla)



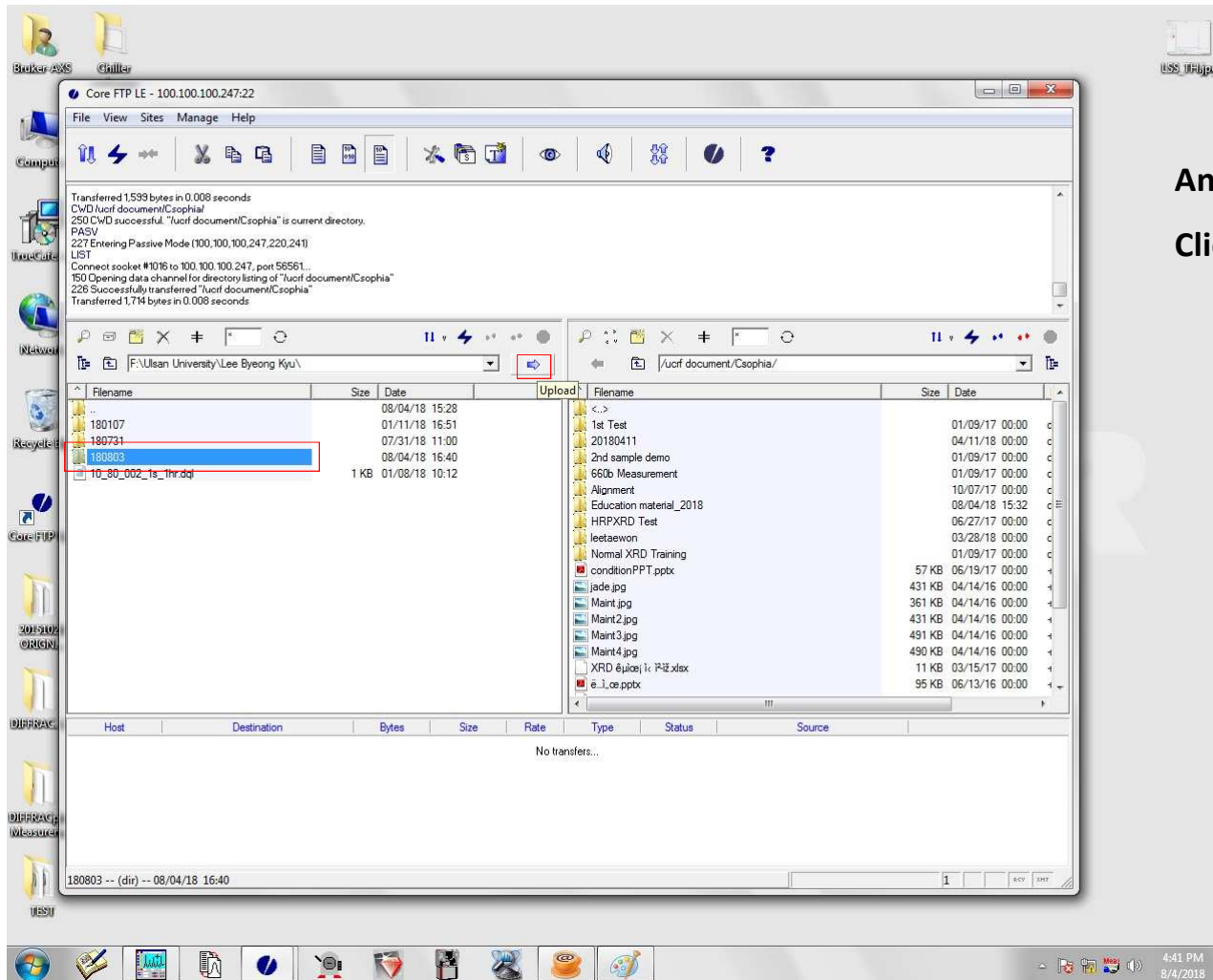
> In Analysis PC

- Click 'Browser' (Right page)

And check file server location that you want to save in Core FTP.

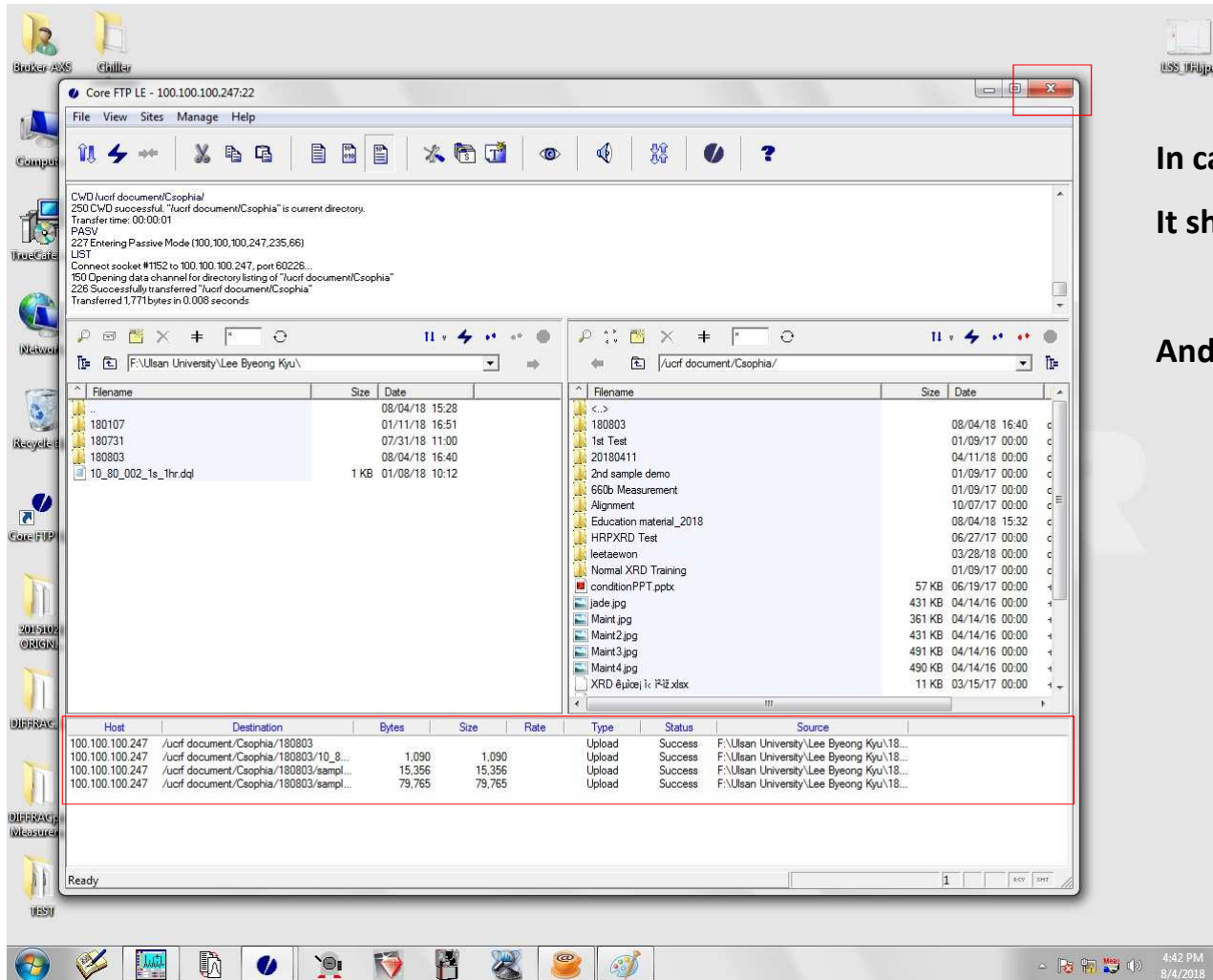
Location: /ucrf document/장비담당자 폴더 ????

Data Upload (FileZilla)



And then, Click file you want to upload in Analysis PC,
Click the 'Upload' button.

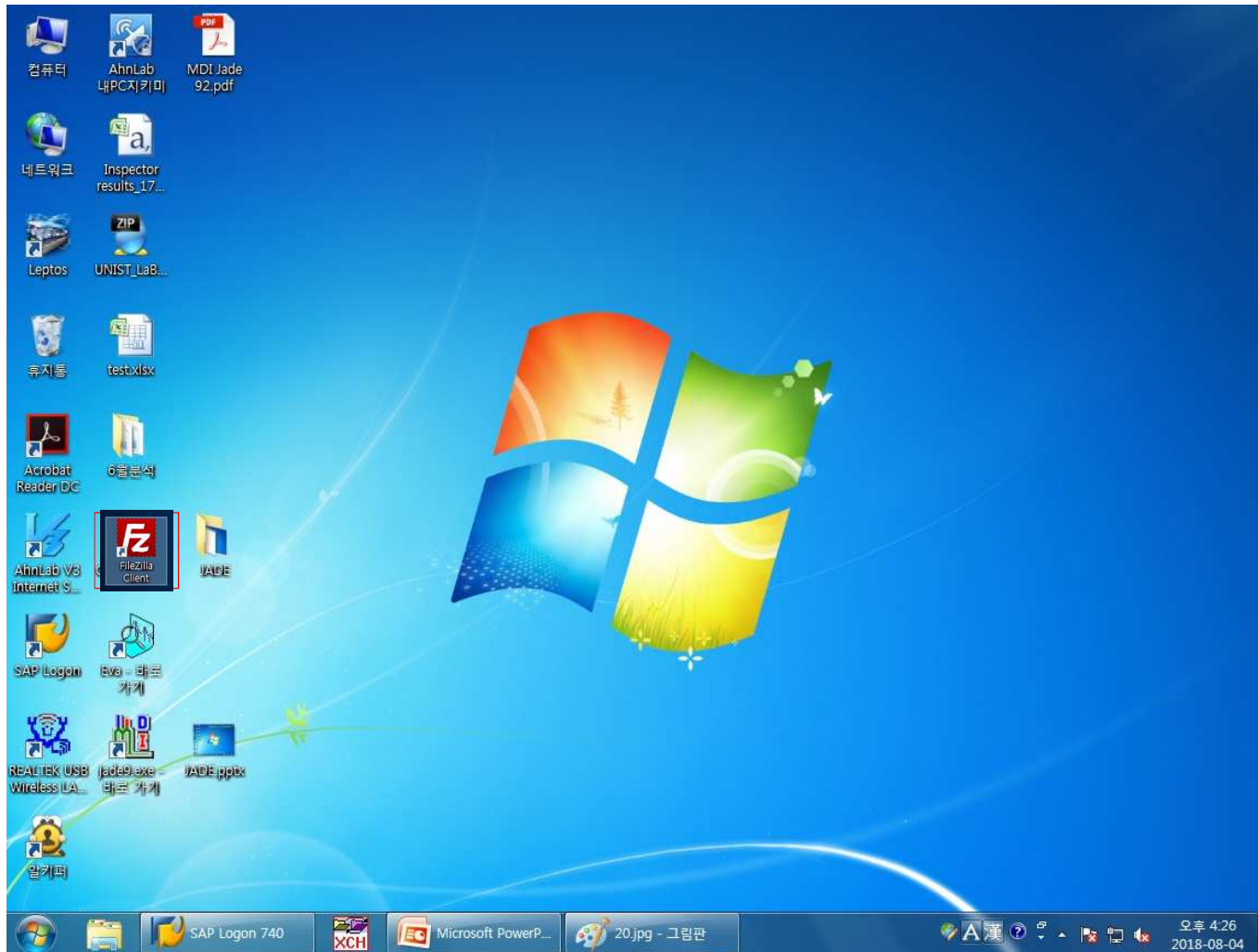
Data Upload (FileZilla)



In case of successful Data upload,
It shows and disappear **success signal** in bottom page.

And then, finish Core FTP program.

Data Download (FileZilla)

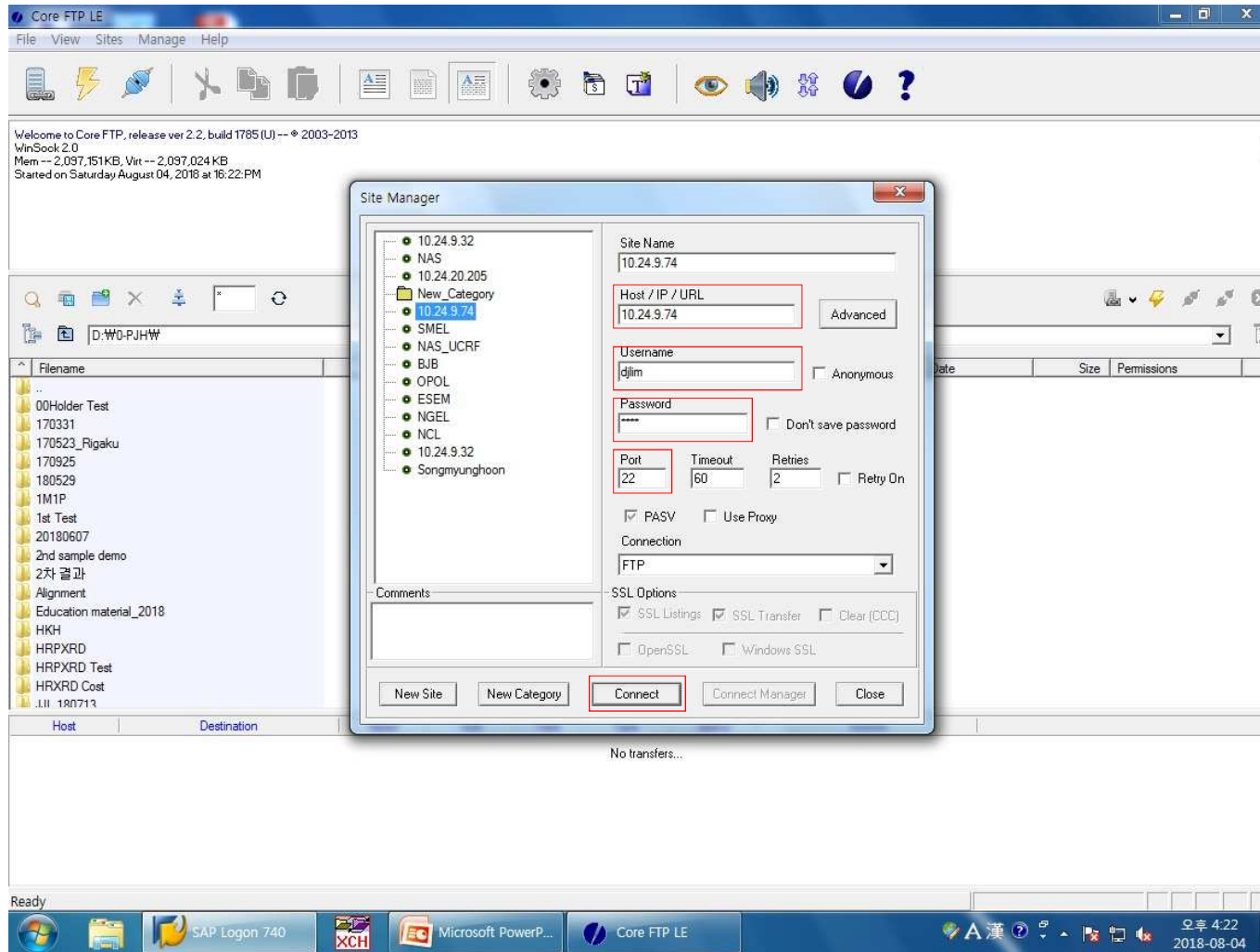


> In PC for **INPUT RESULT**, (center table)

Double click 'FileZilla' in desktop

For Data Download.

Data Download (FileZilla)



IP: 10.24.9.74,

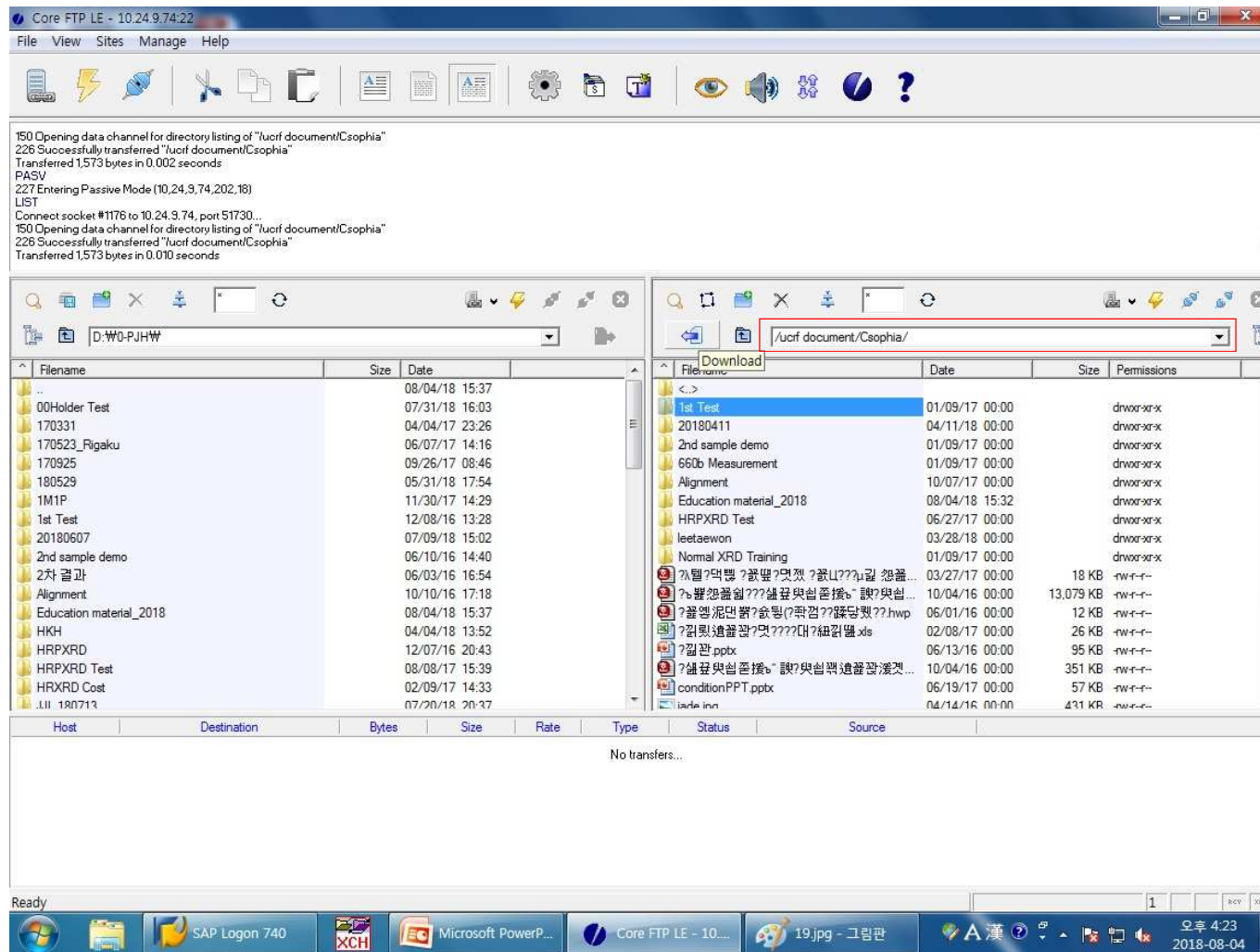
Username: professor name,

Password:;

Port: 22

Click 'Connect'.

Data Download (FileZilla)



➤ Move Left to Right folder

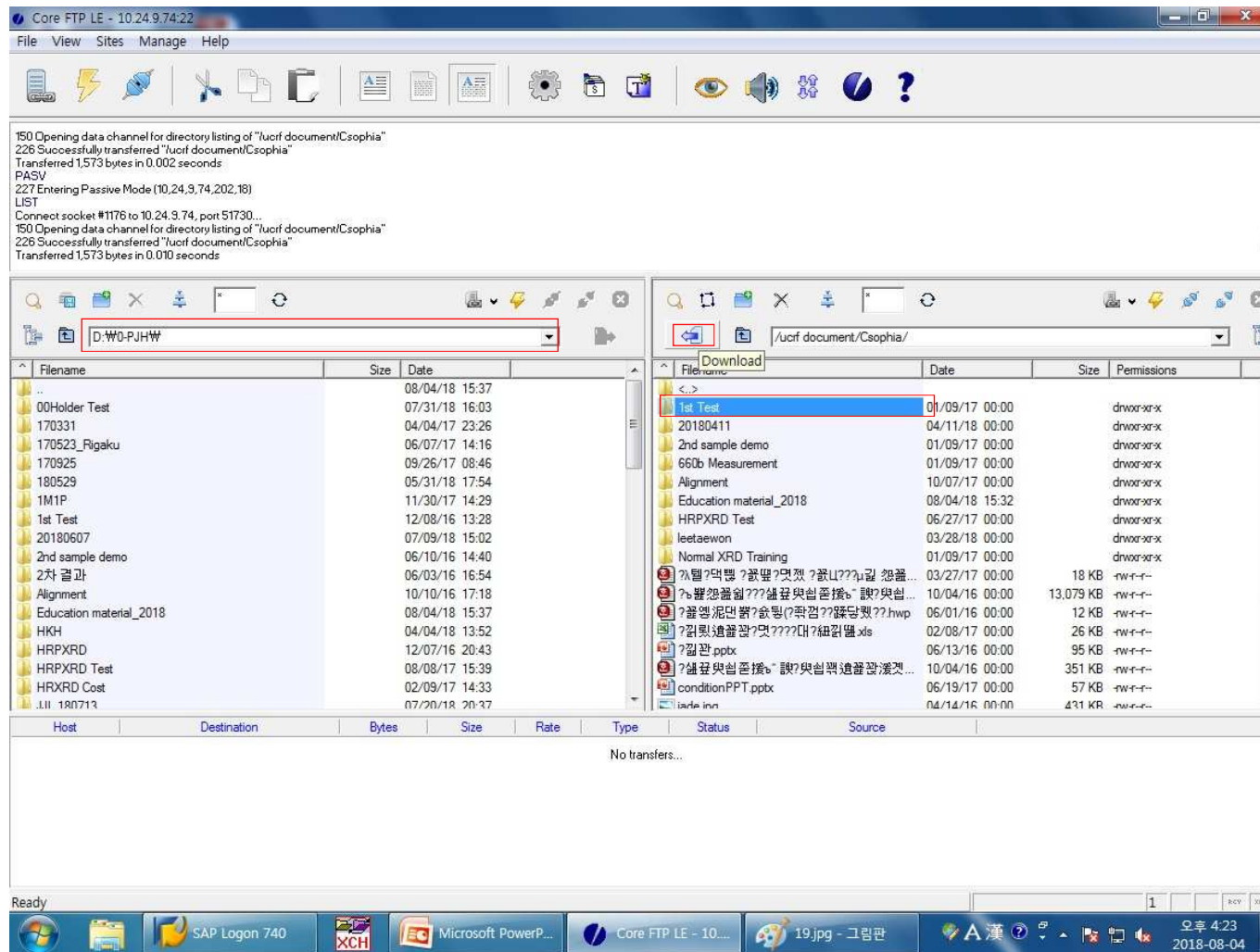
(opposite to Analysis PC move path)

Click 'Browse' button in Right page.

Check file location that download files exist.

Location: /ucrf document/담당자 이름 폴더??

Data Download (FileZilla)



Click 'Browse' button in Left page.

Check file location that you want to save.

위치: /ucrf document/담당자 폴더???

Click 'Download' button after clicking folder.