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

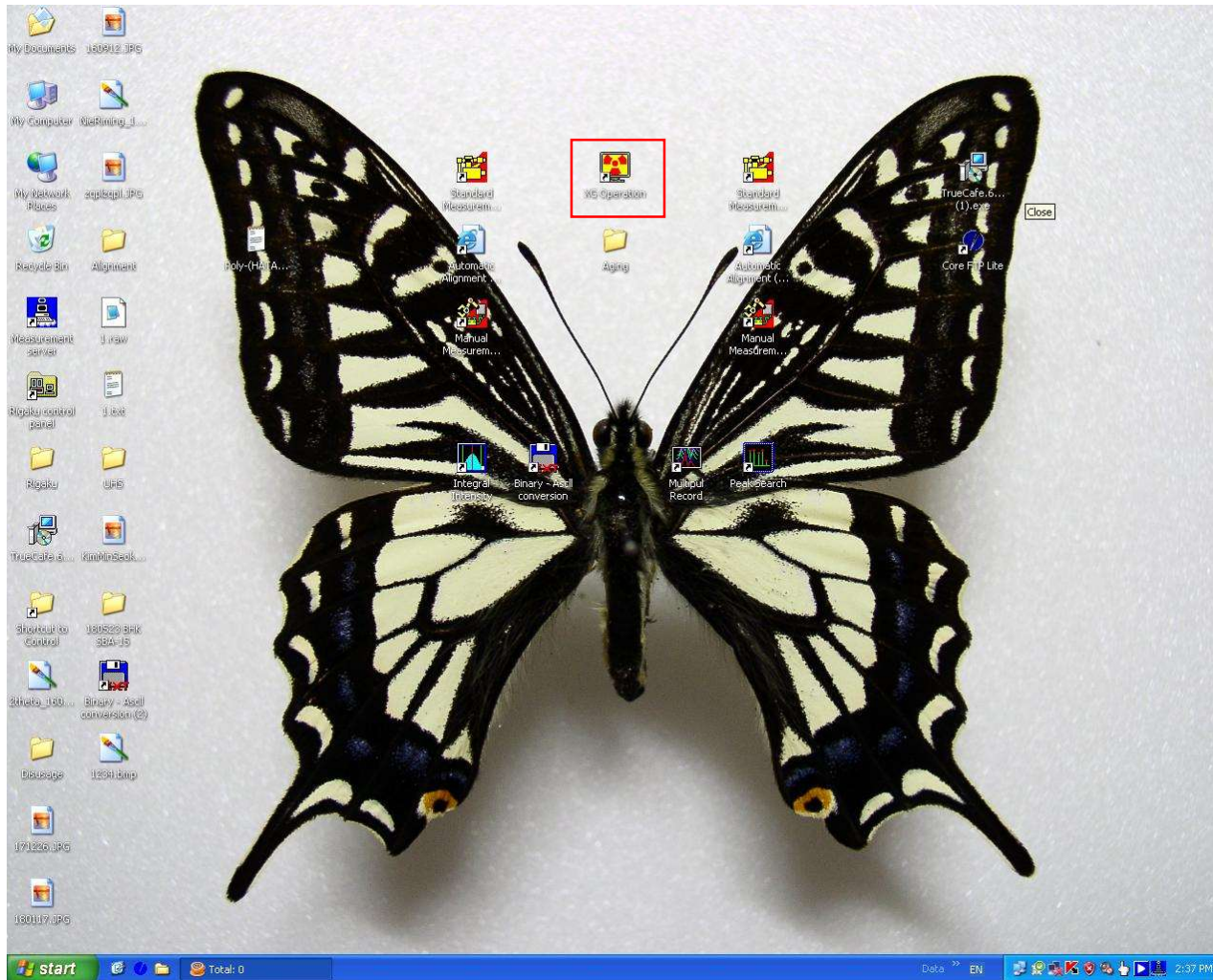which located at bottom of HPXRD.

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

X-ray Operation

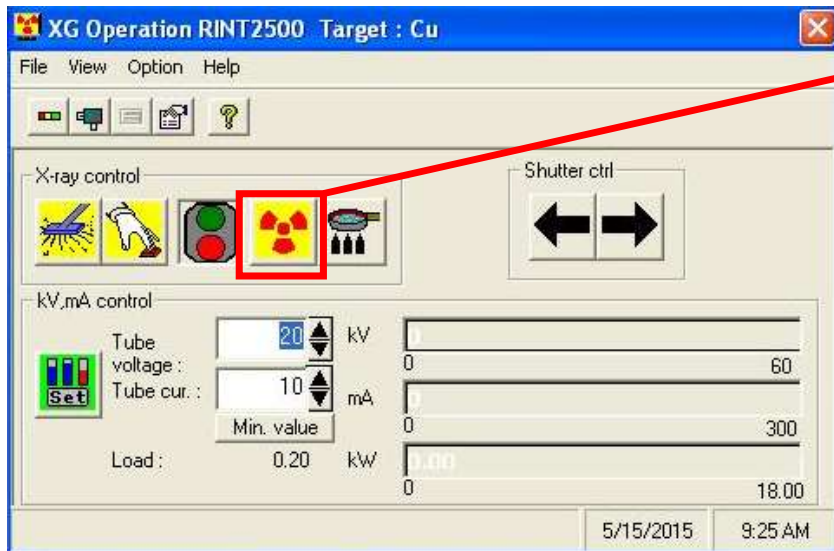XG Operation is,

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 [HOLYDAY use],
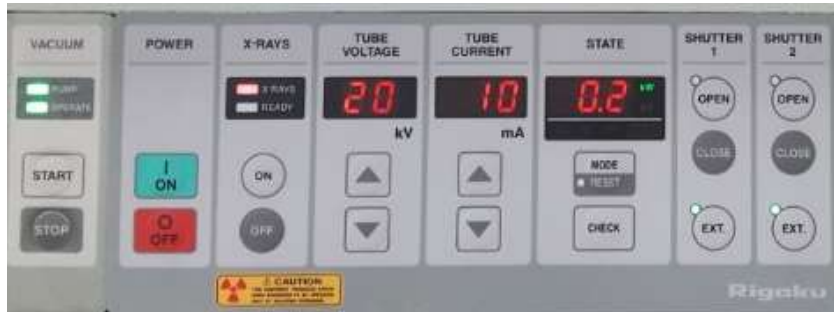
You should do 'Aging Process'.

> 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.
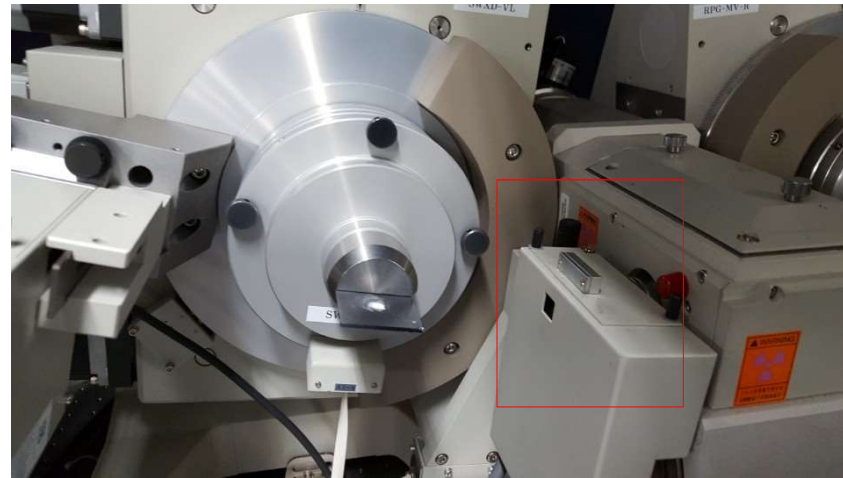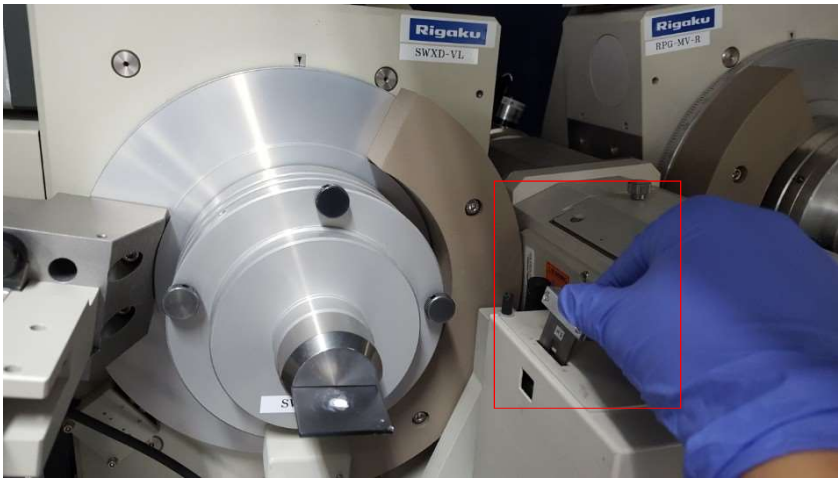
Standard Measurement (Left)

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.

FIRST IN CHANGE
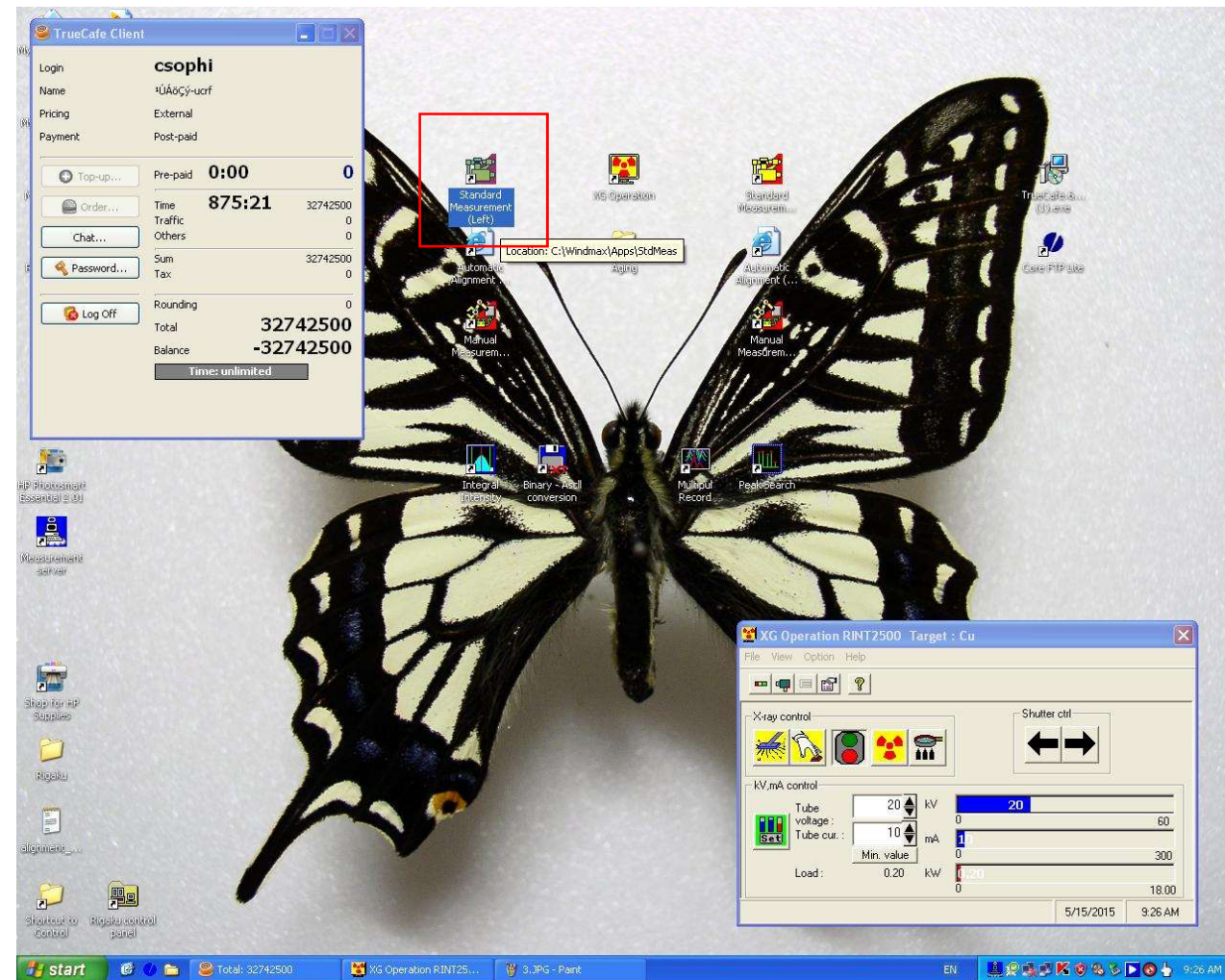
Standard Measurement (Left)
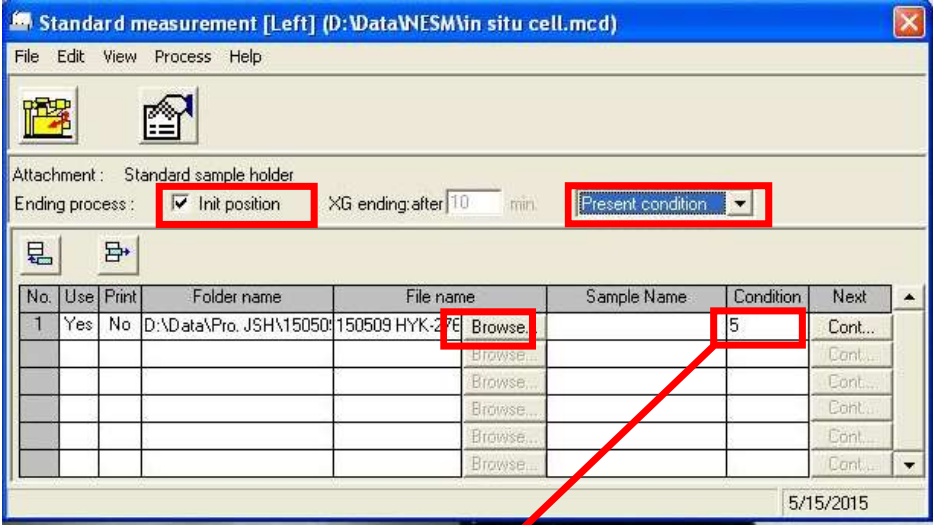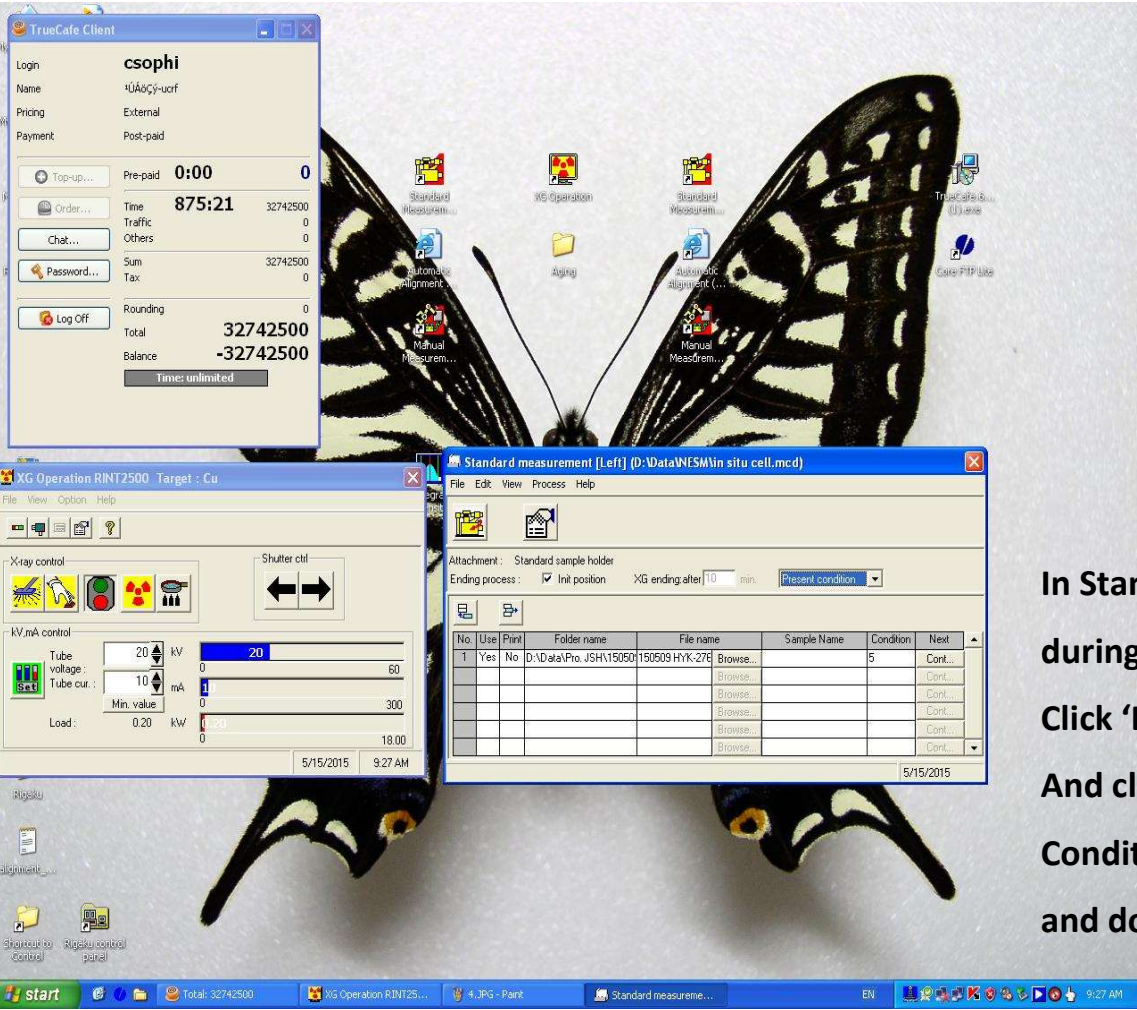
In Standard Measurement(Left),

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)
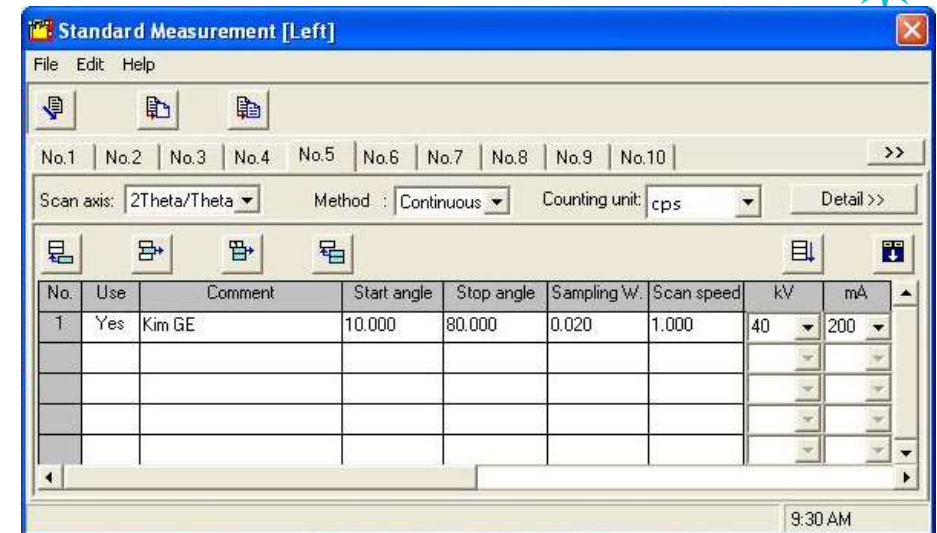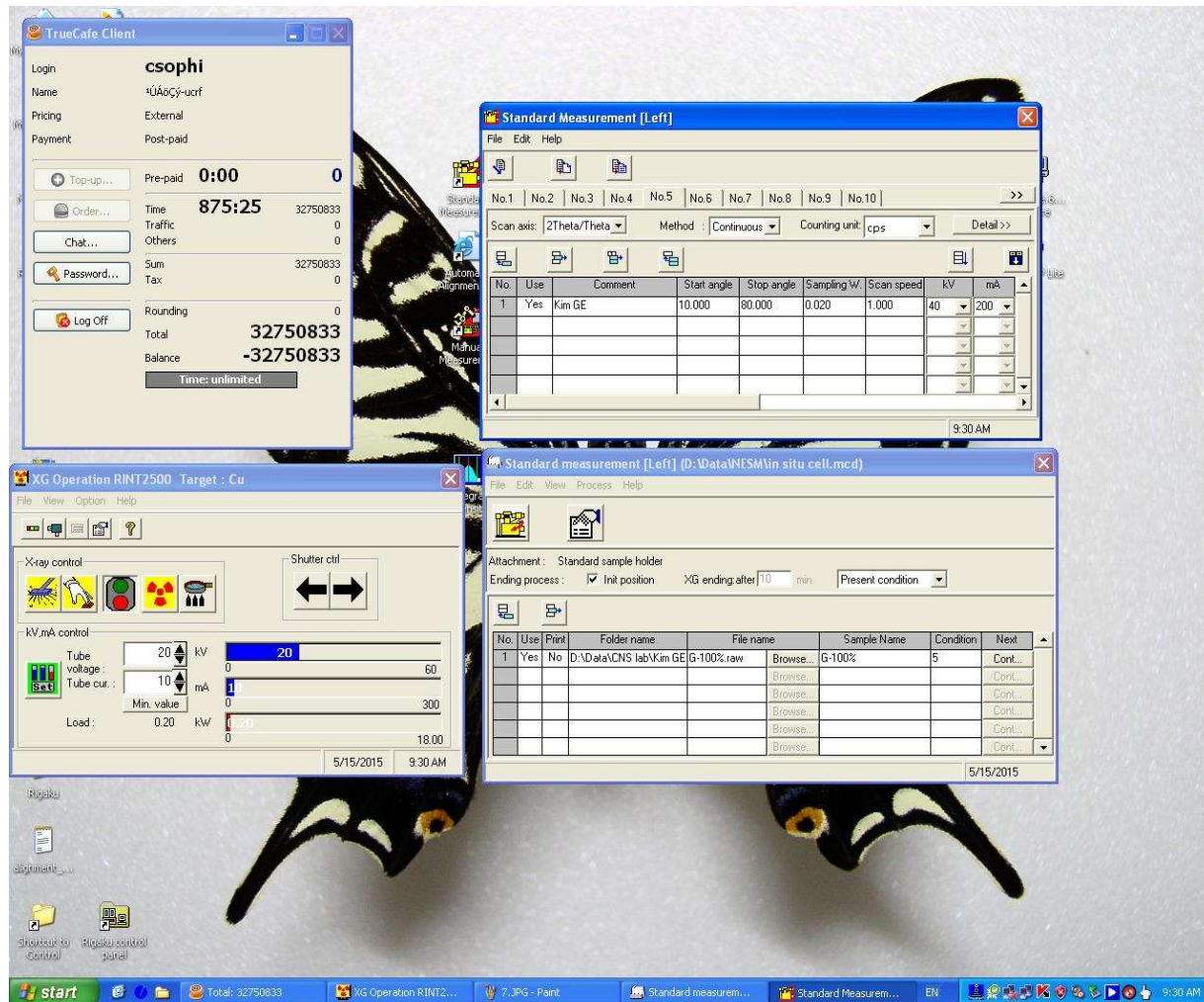
In condition pop-up,

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 <u>quit button </u>and that means Autosave.

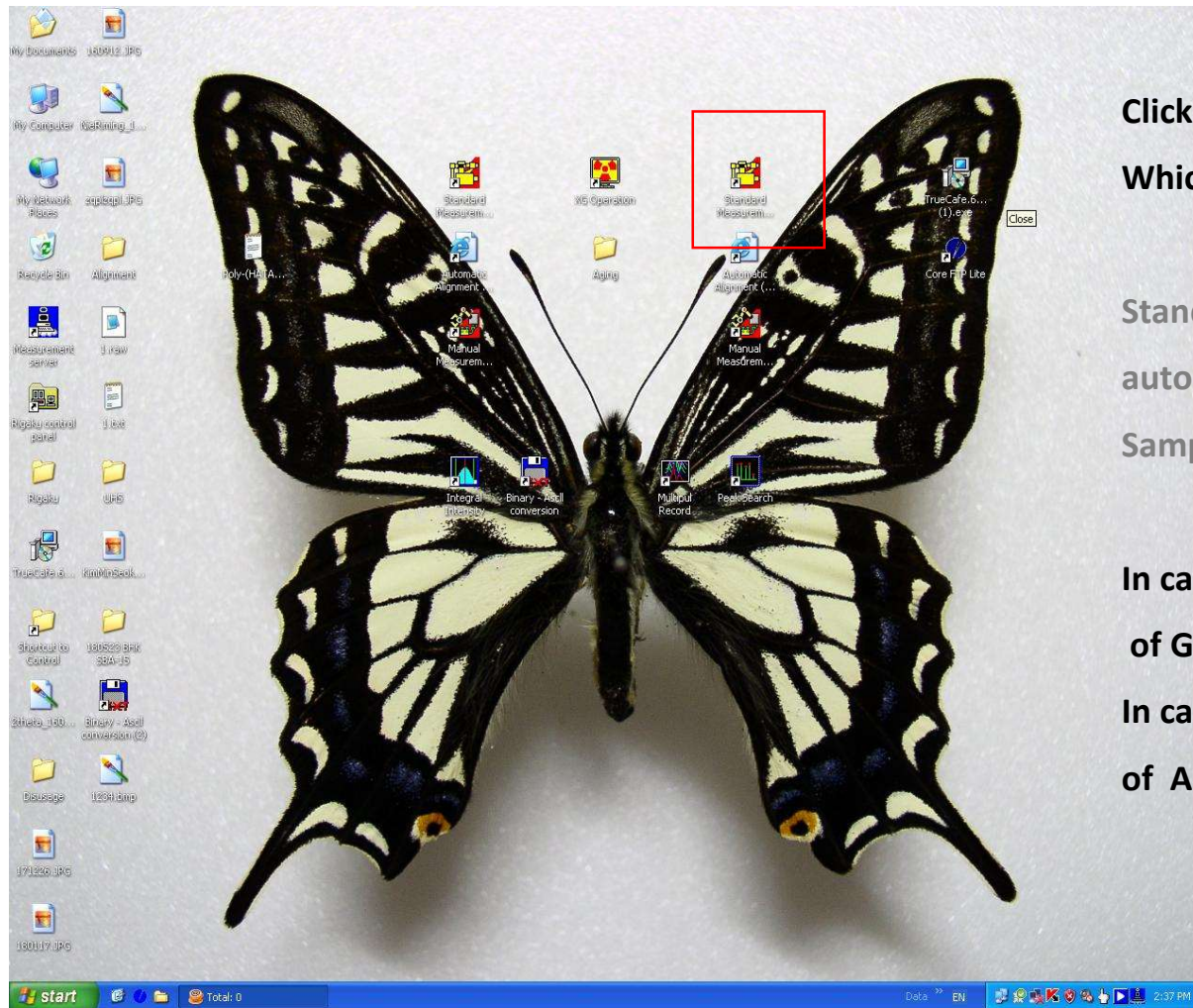# Standard Measurement (Left)

<Tip! For using the same measurement condition>
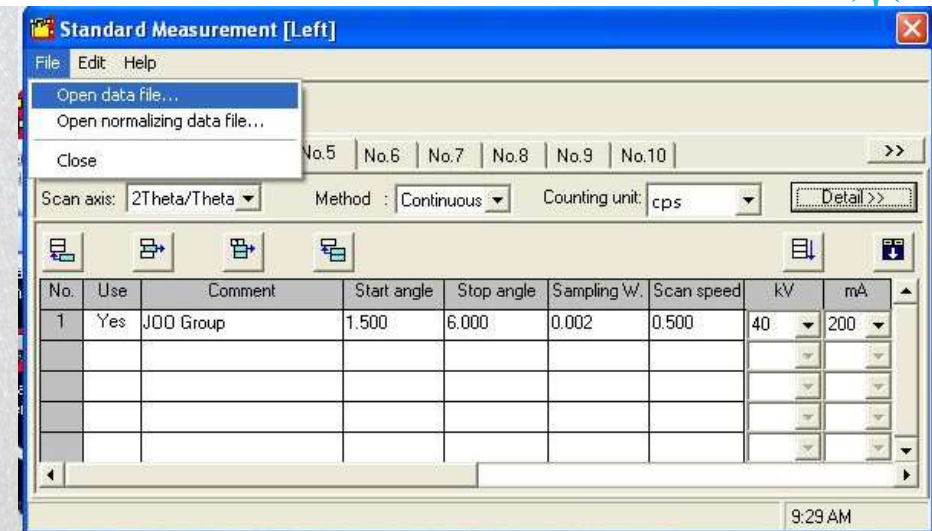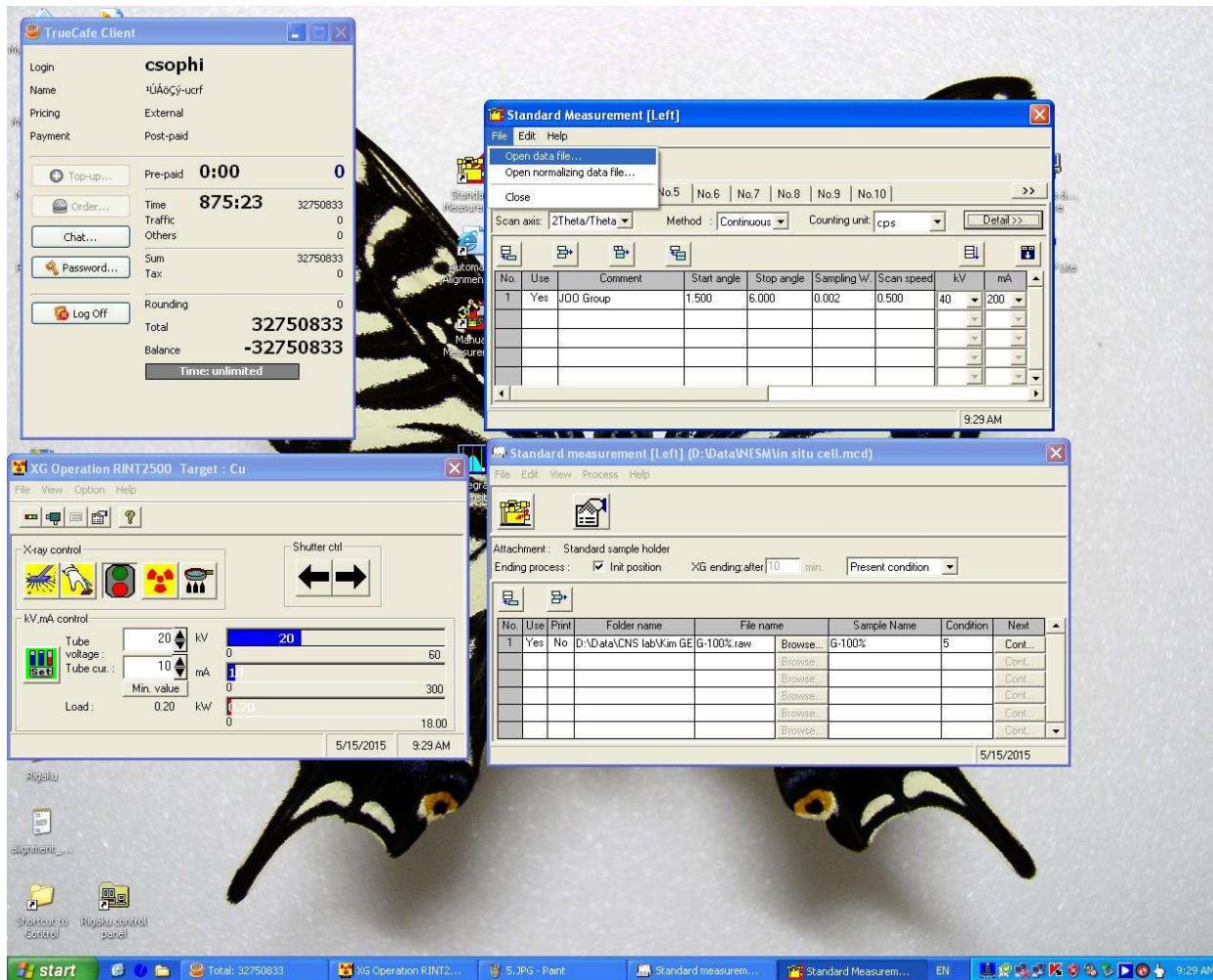
After clicking the number of 'Condition', it shows

new window of Standard Measurement and then,

Click 'Open Data File' in 'File'.

In new window, click the most recent measurement file.

FIRST IN CHANGE

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.

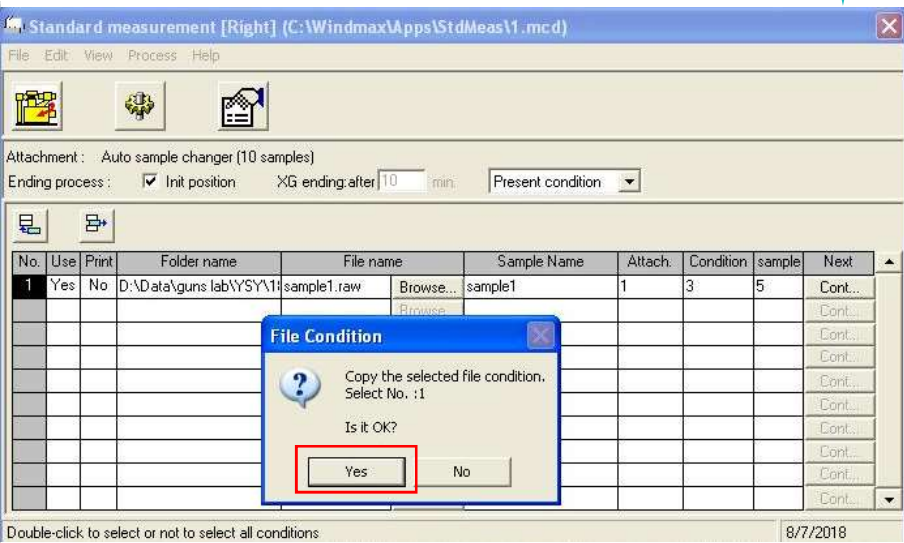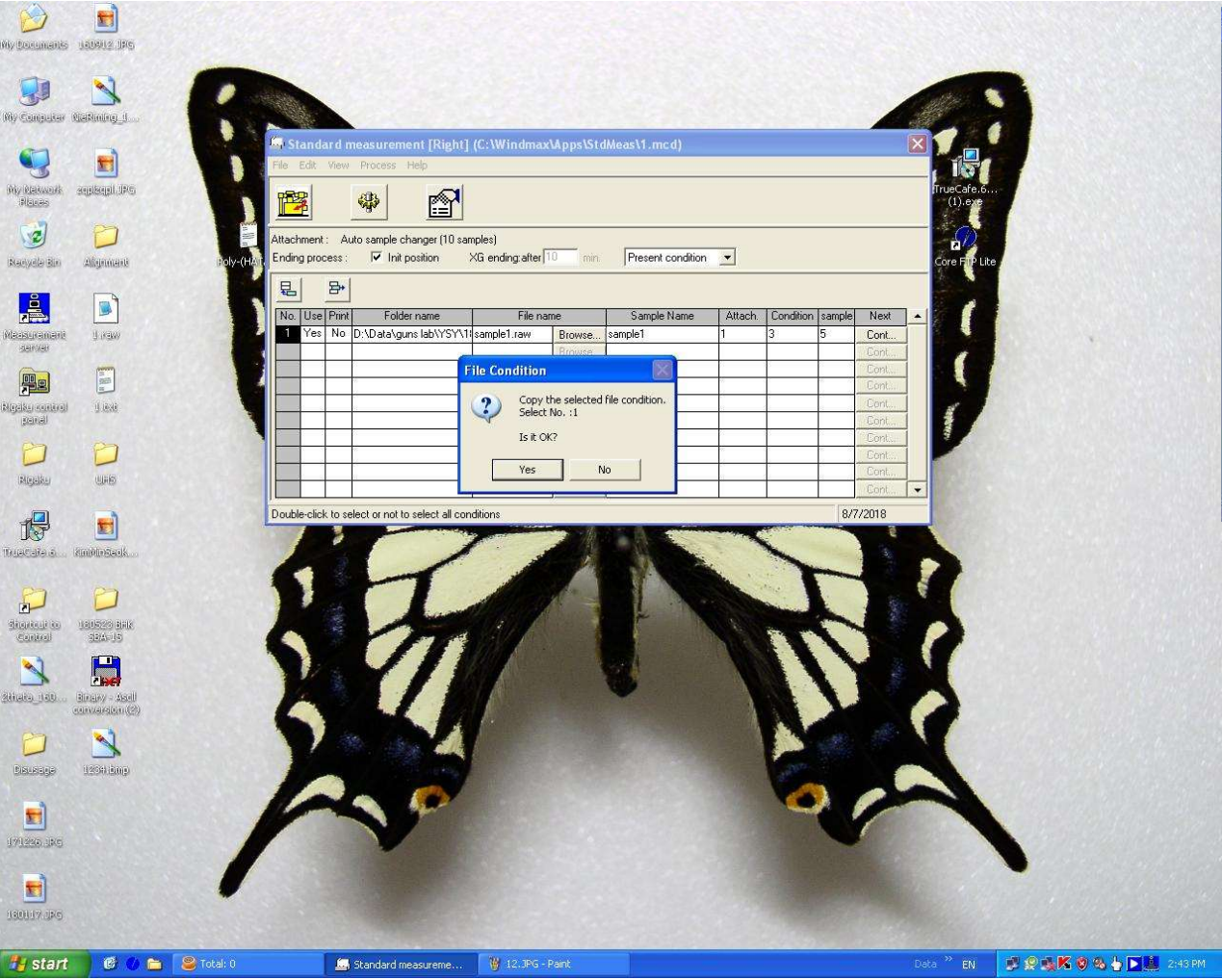**In case of thin film or bulk sample, it has 2 types of bottom presence**

of Al Holder.

Standard Measurement (Right)
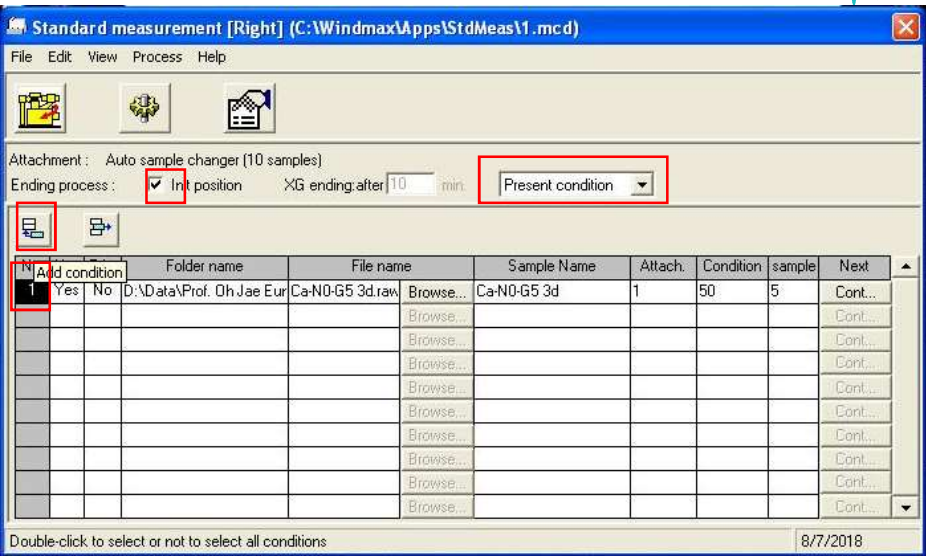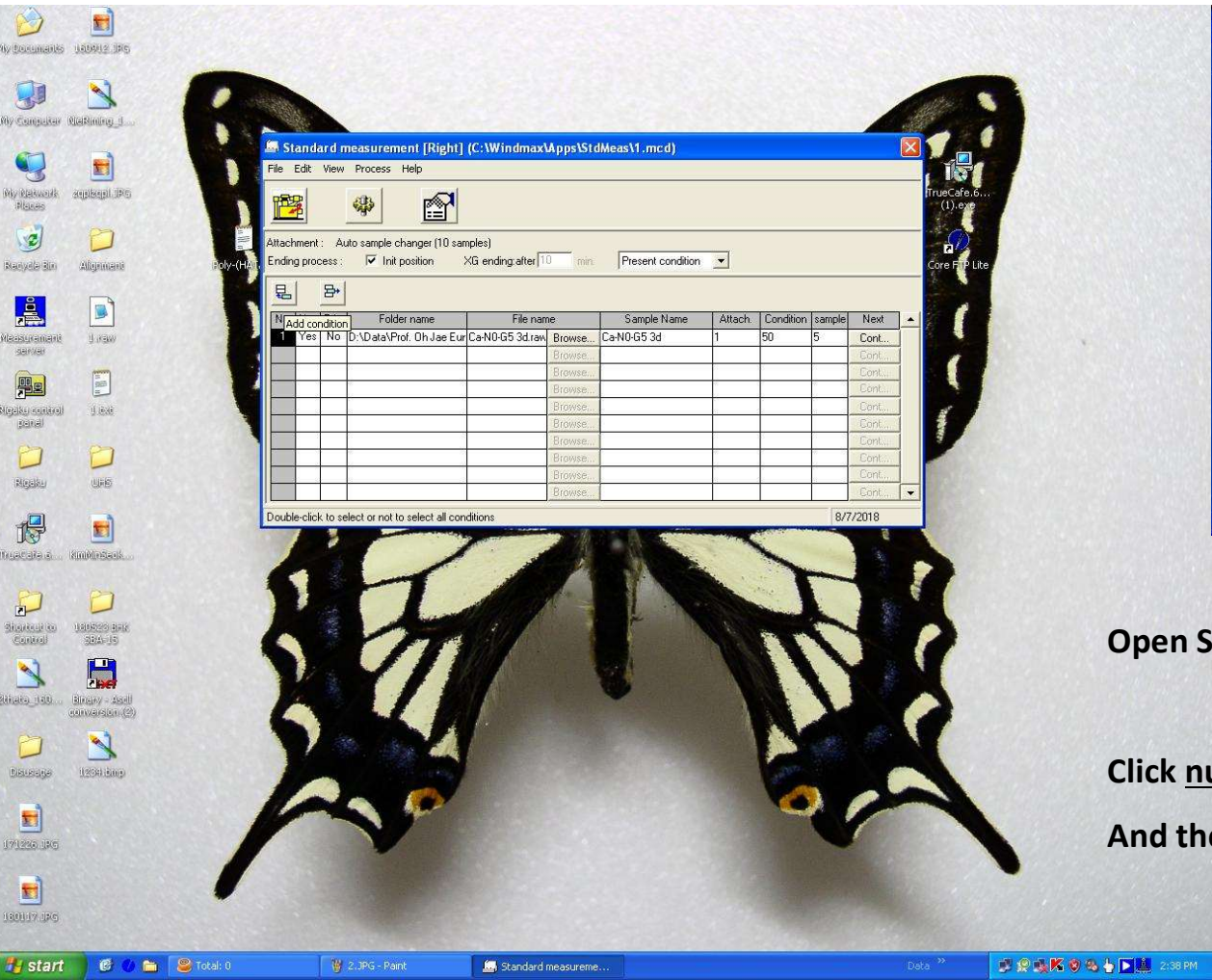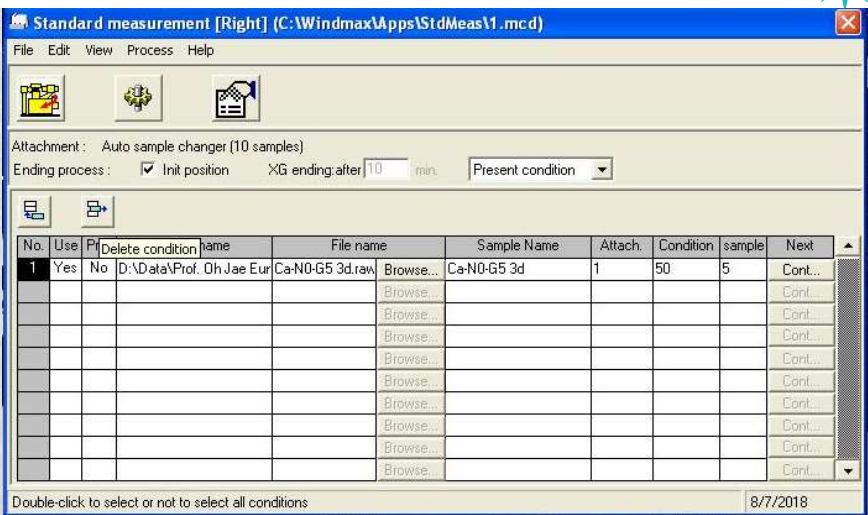

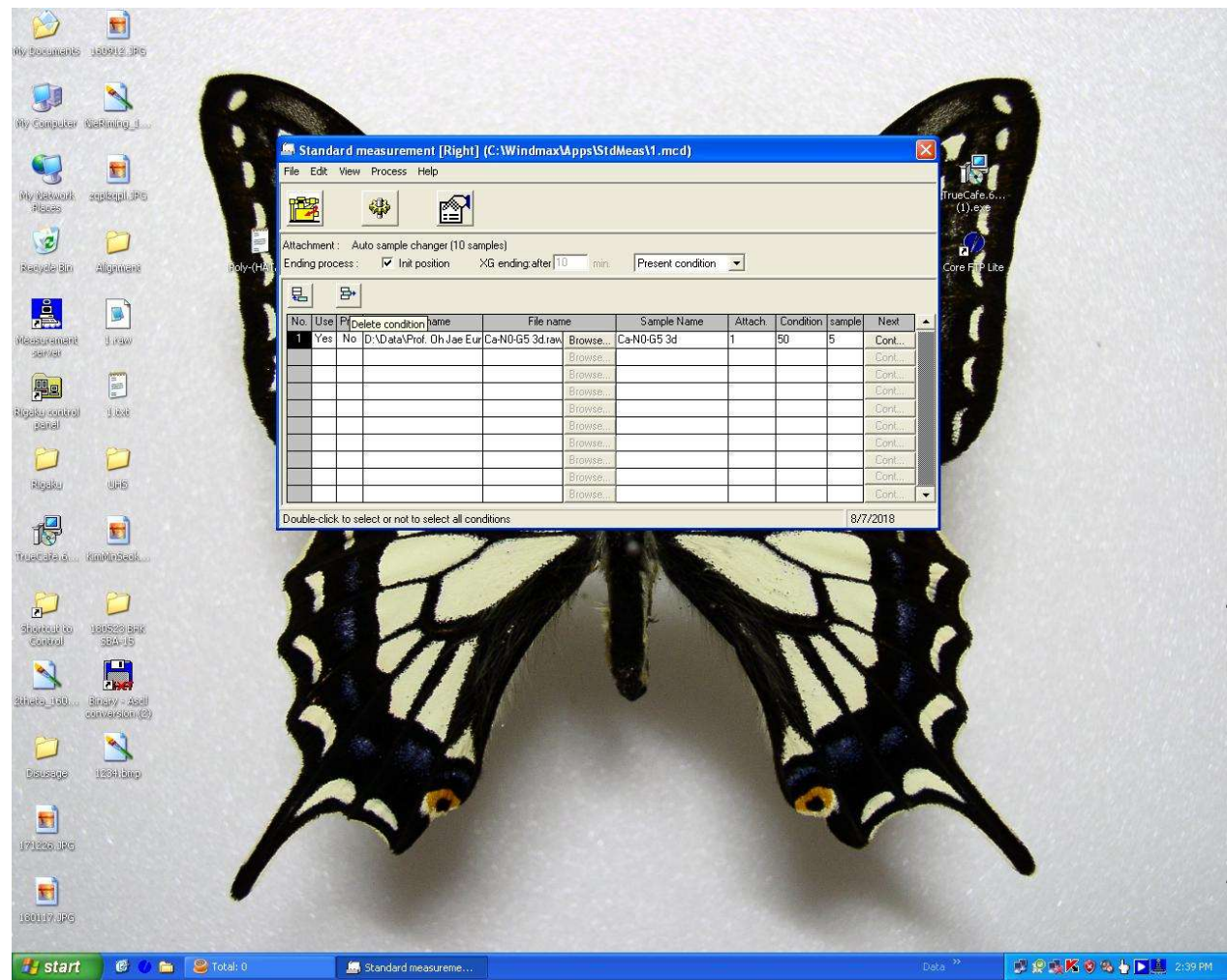**Open Standard Measurement(Right) Window.**

Click <u>number 1</u> **when you measure several samples automatically,**

And then click 'Add condition' button.

Standard Measurement (Right)

After opening 'File Condition Window', click 'yes'.
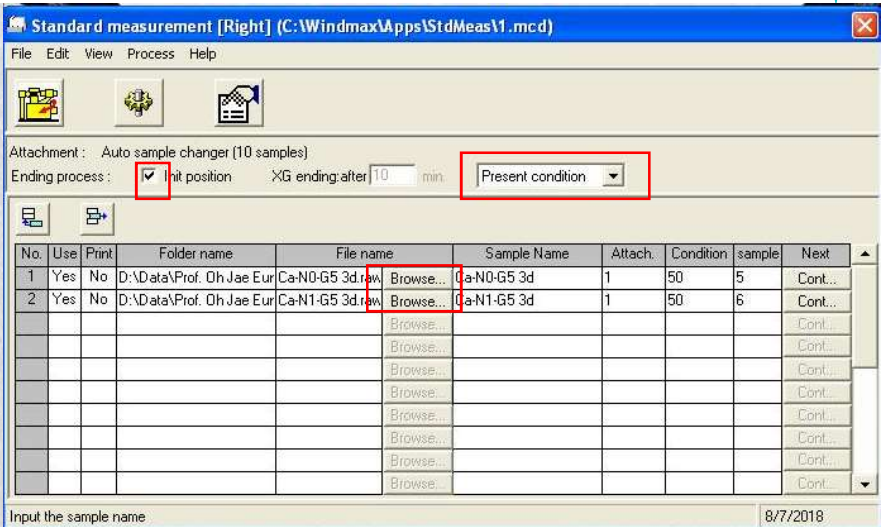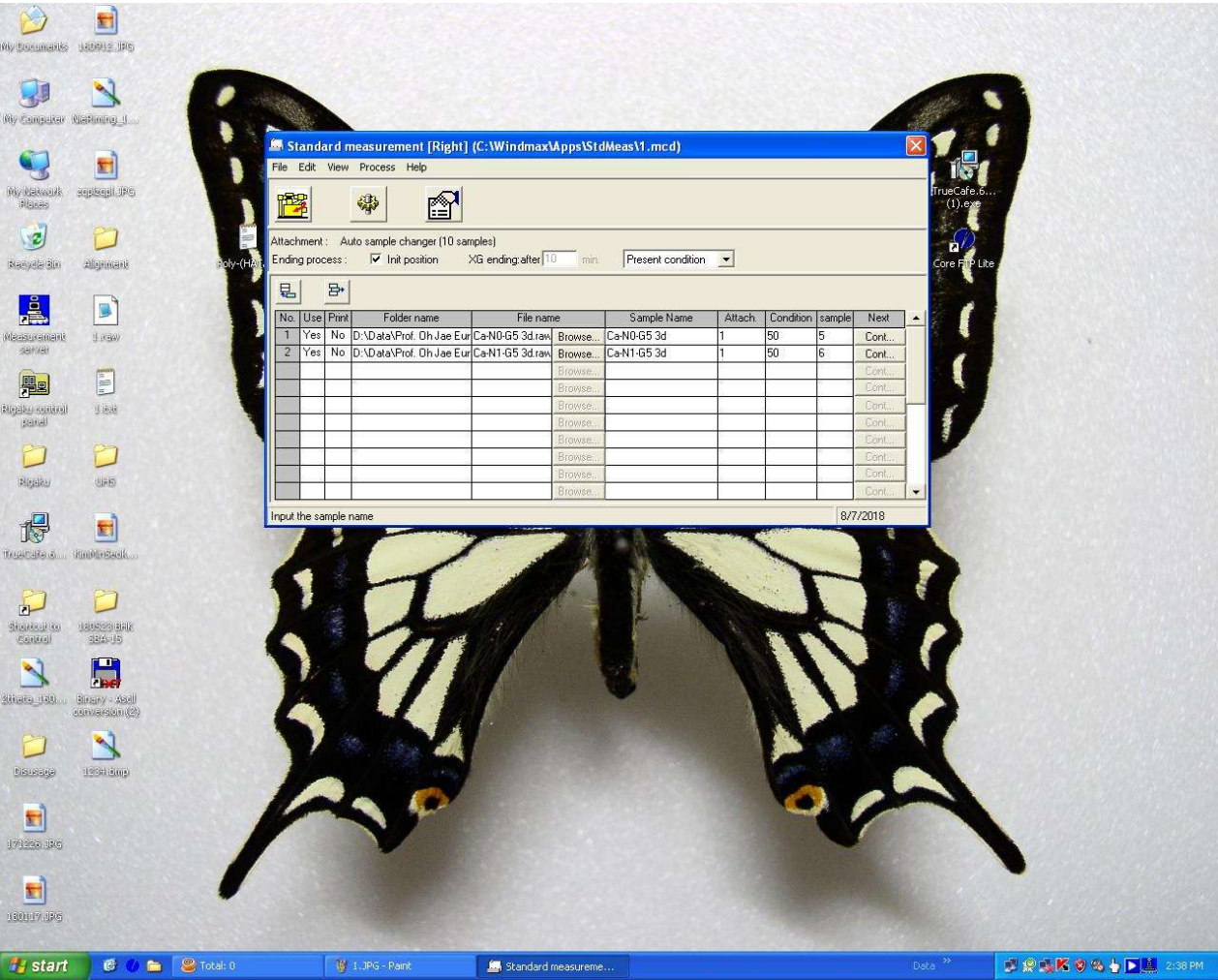
Standard Measurement (Right)

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

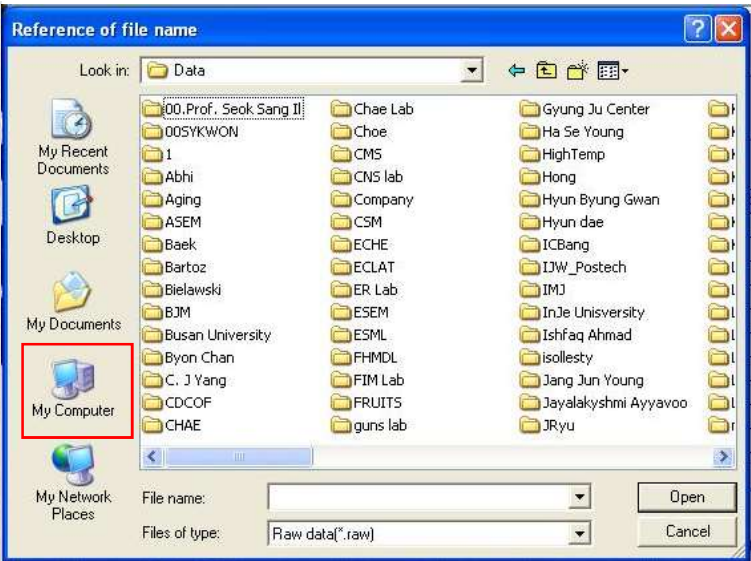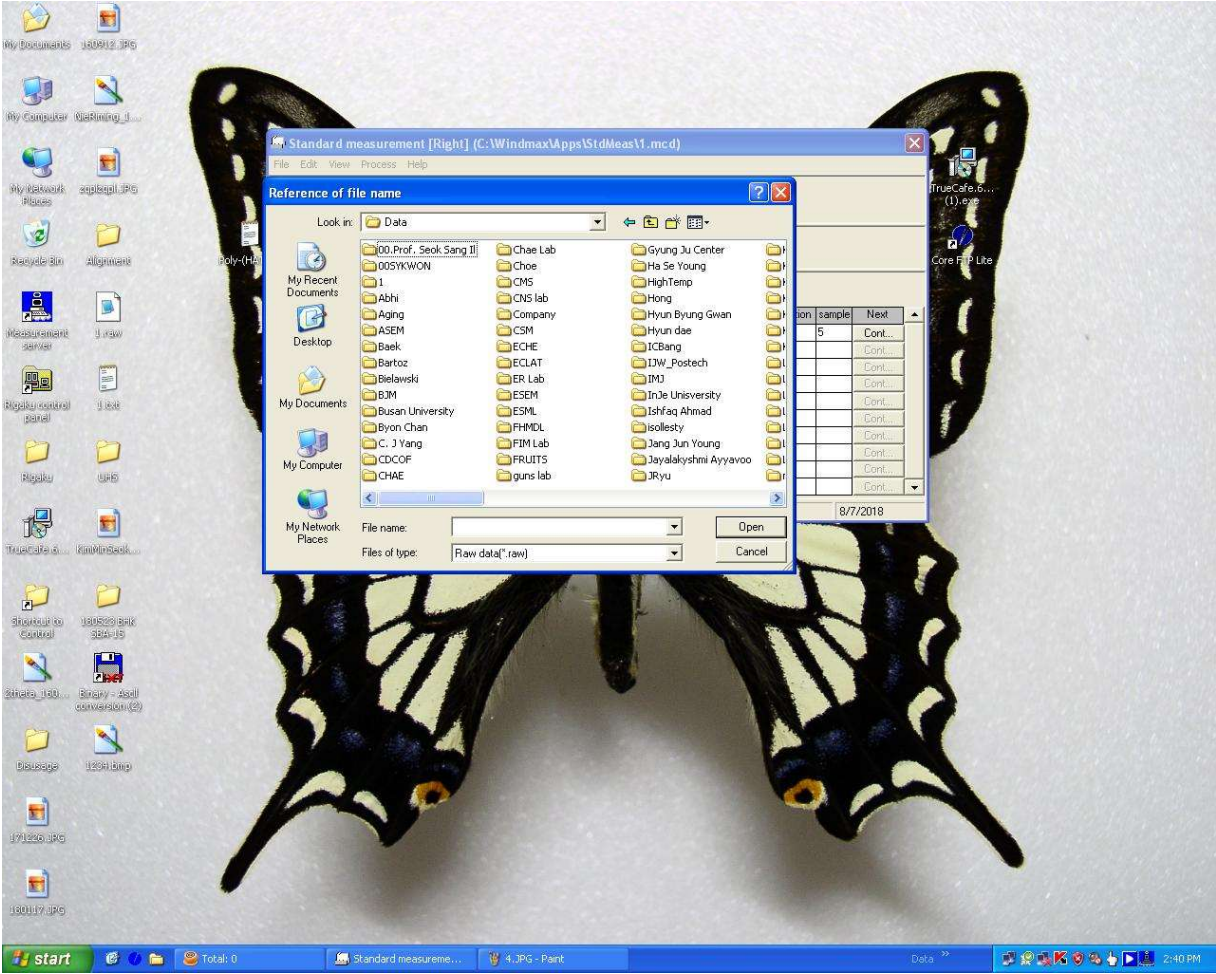In black status of number field, click 'Delete Condition' button

After opening new window, click 'yes'.

Standard Measurement (Right)

While weekday 9:00~18:00,

Check 'Int. position' and 'Present Condition'.
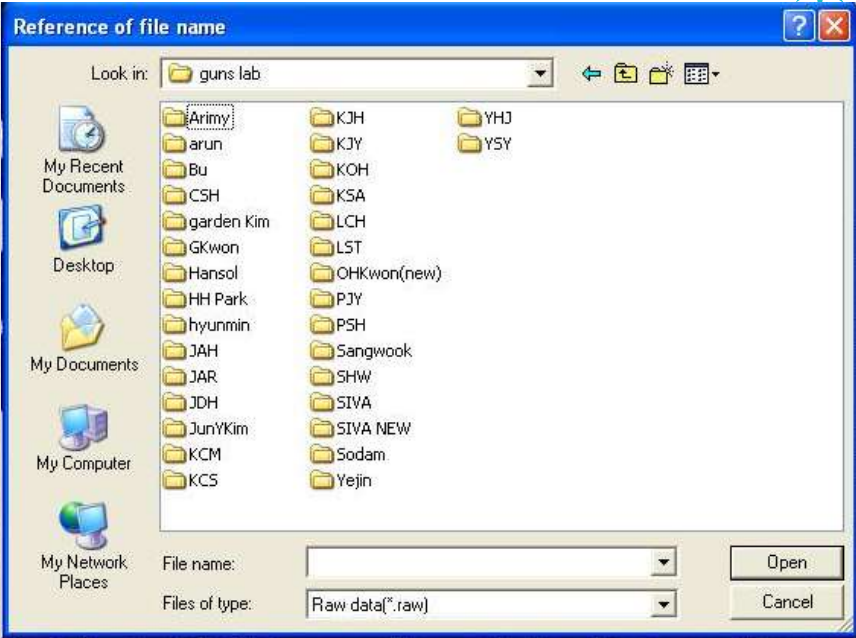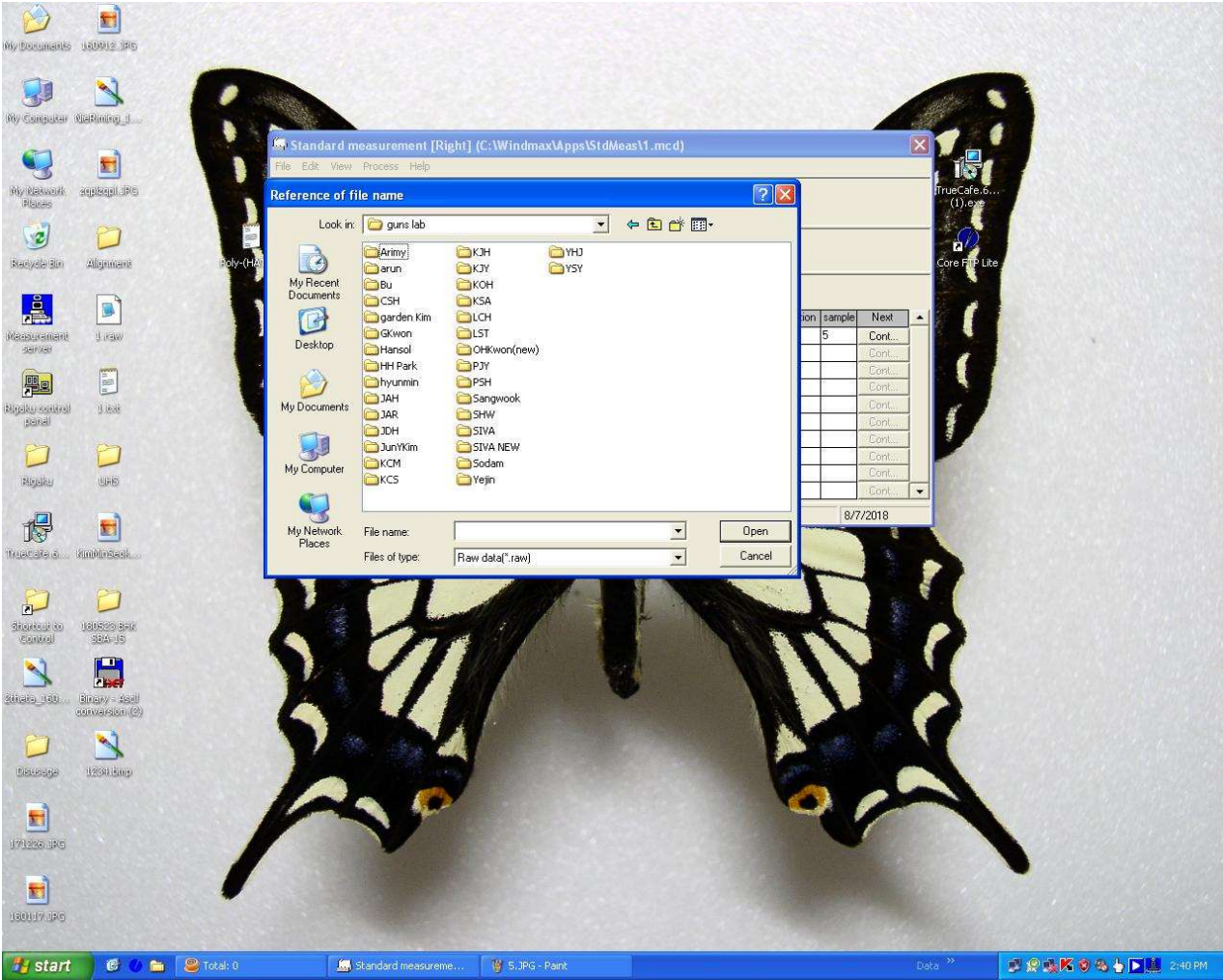
Click 'Browse' button.

Standard Measurement (Right)
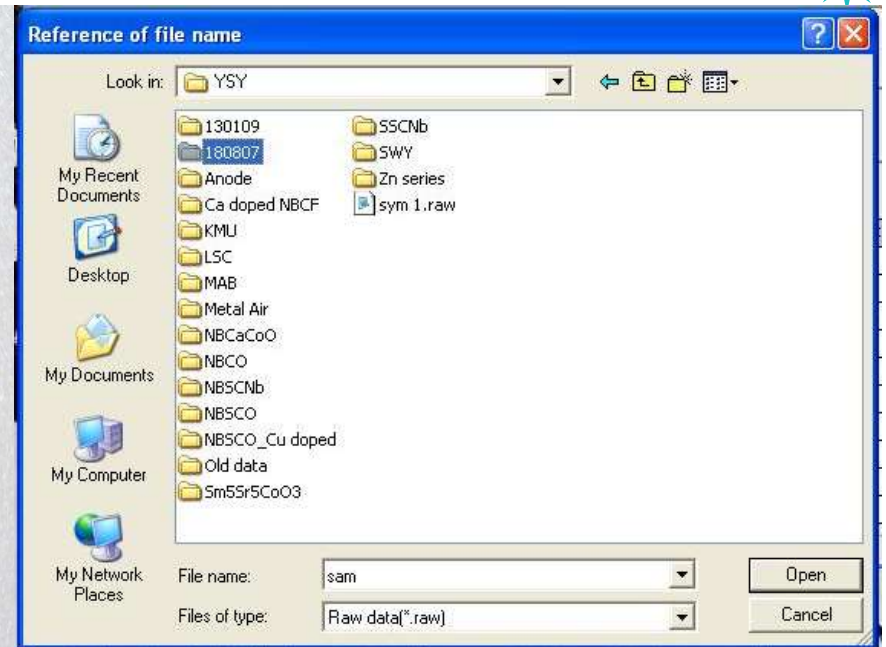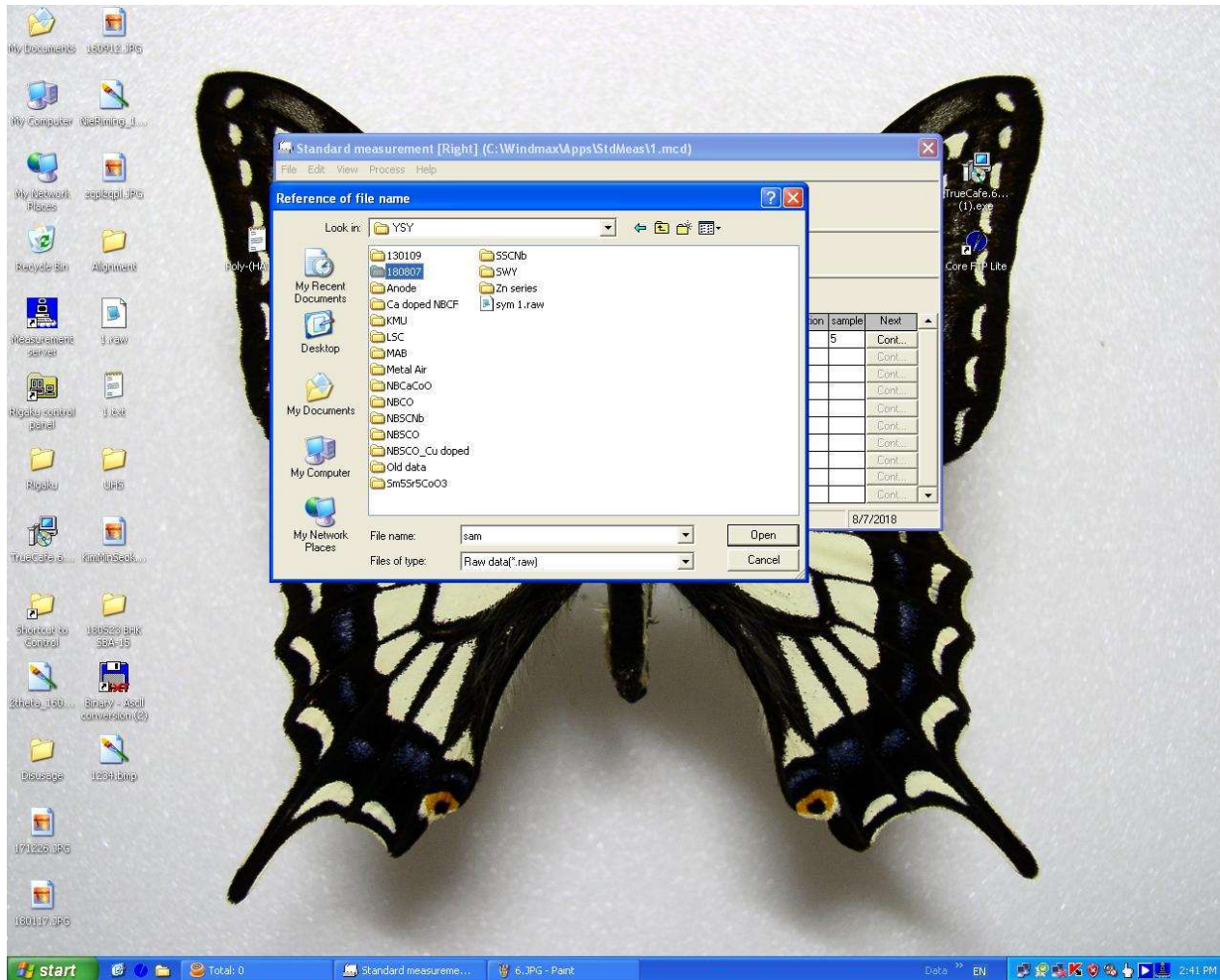

New window opens.

Click 'My Computer' and then click [D Drive > Data].
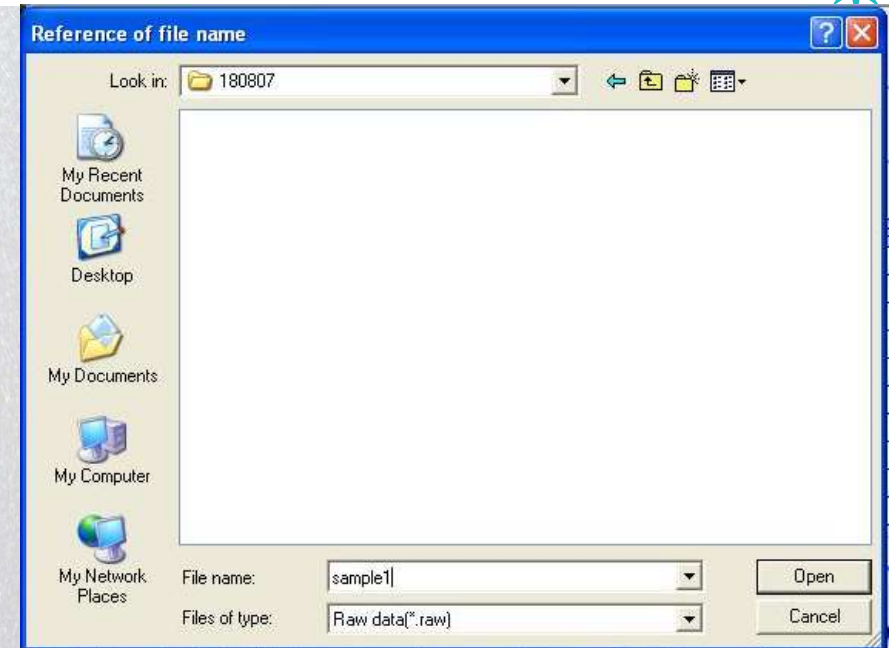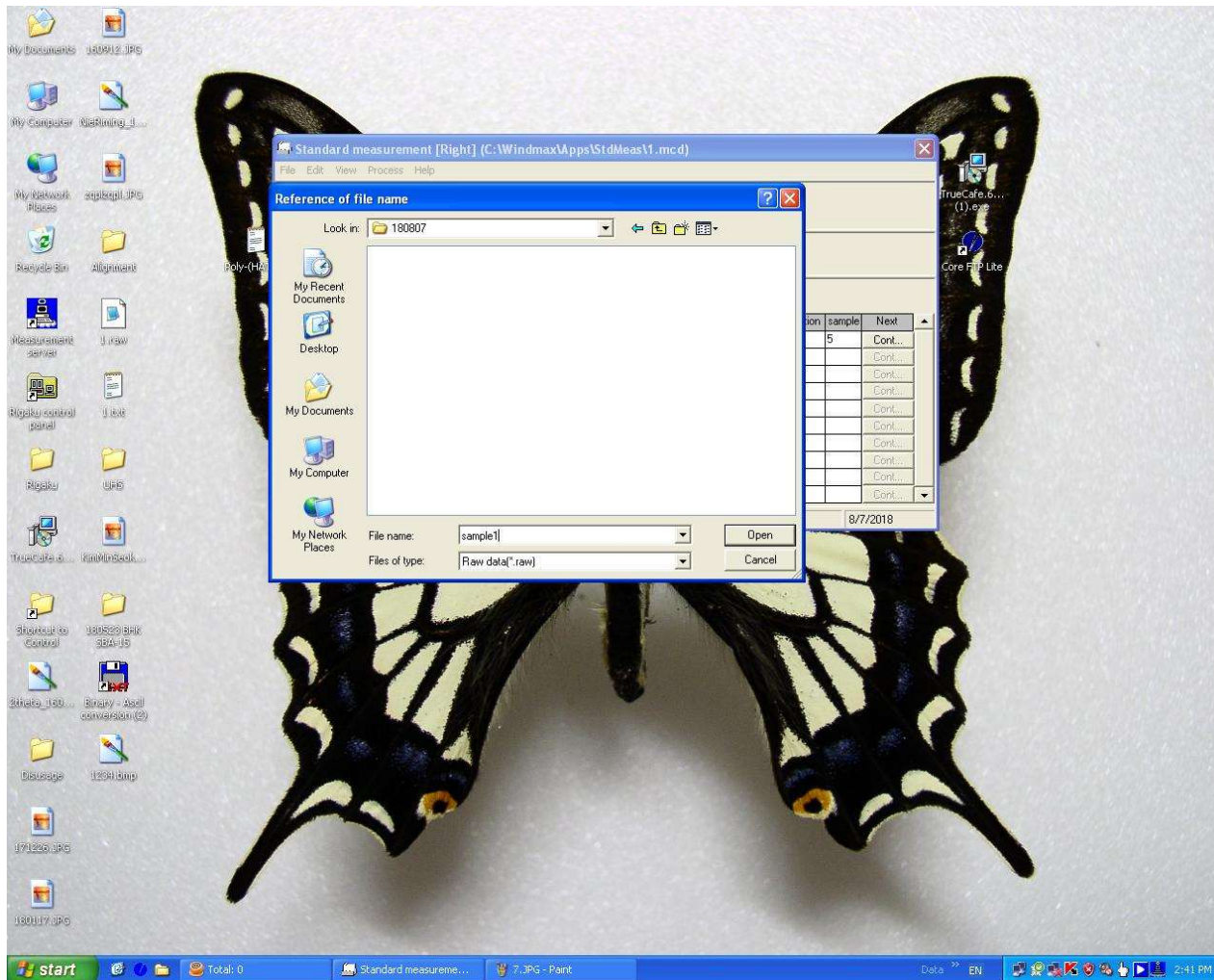
Standard Measurement (Right)

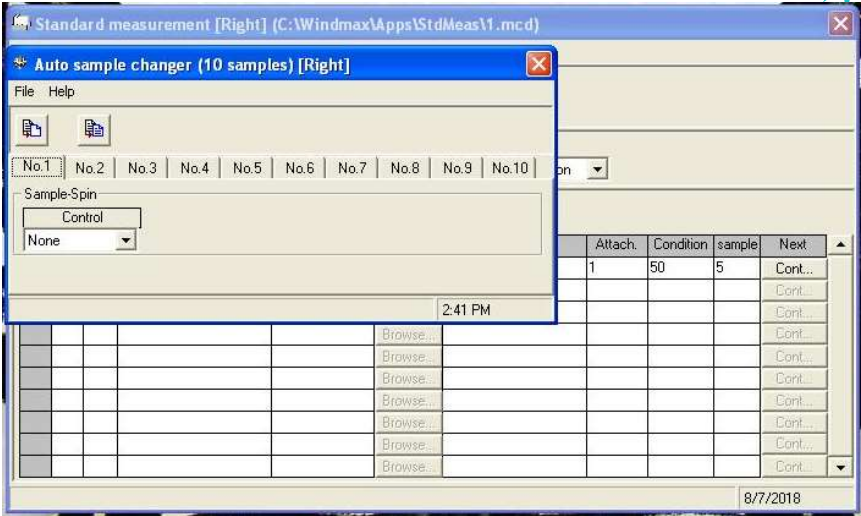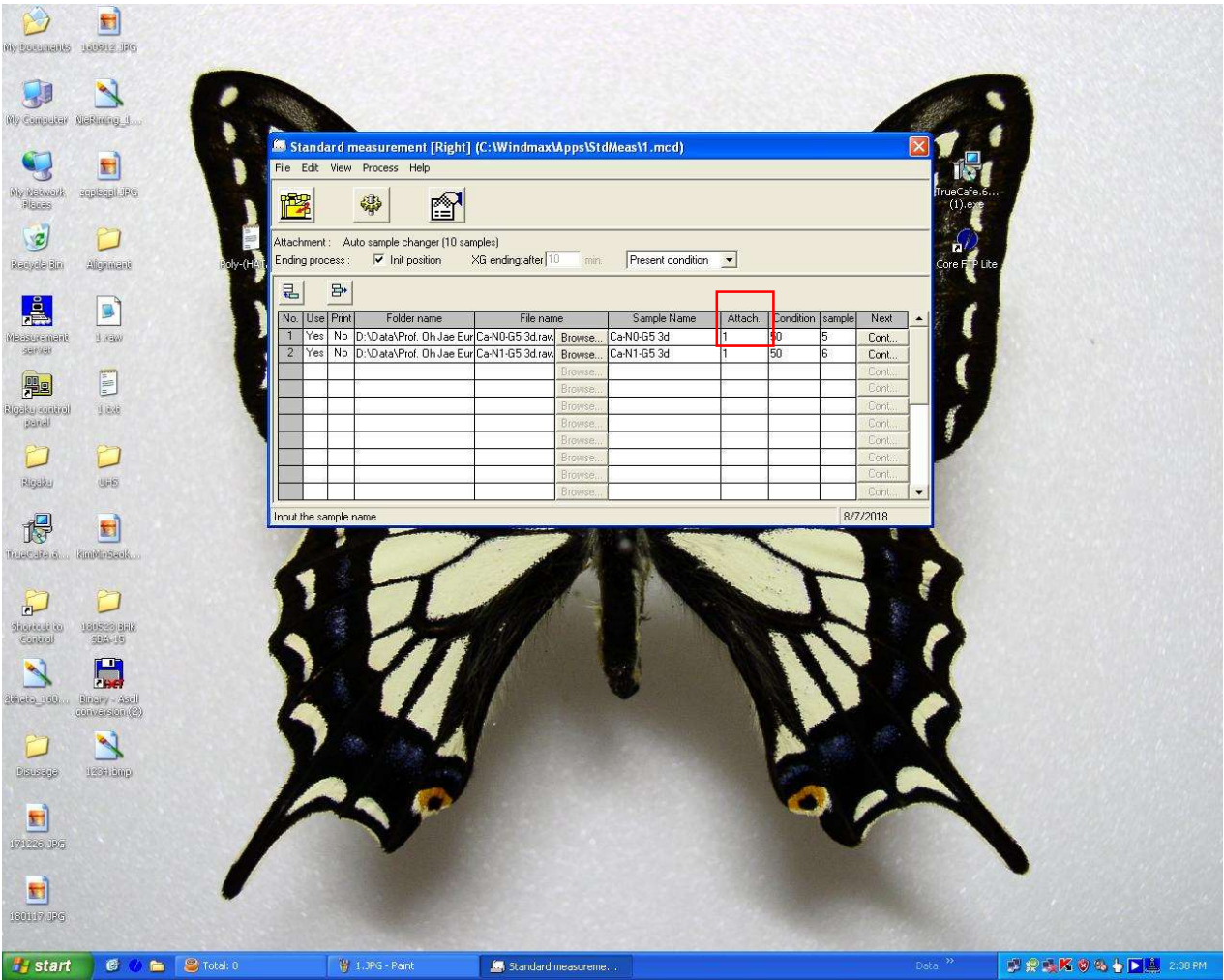**Click [D Drive > Data > Lab folder].**

Standard Measurement (Right)

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

Standard Measurement (Right)

Click [D Drive > Data > Lab folder > User folder > date].
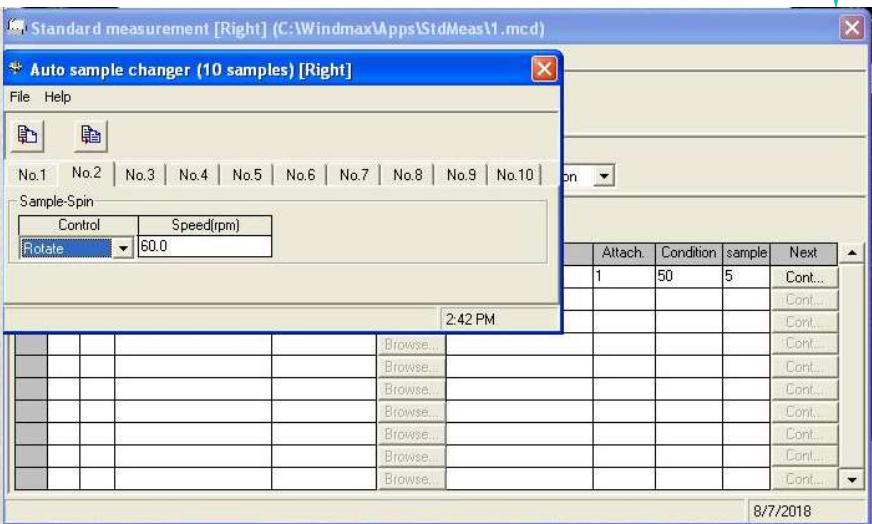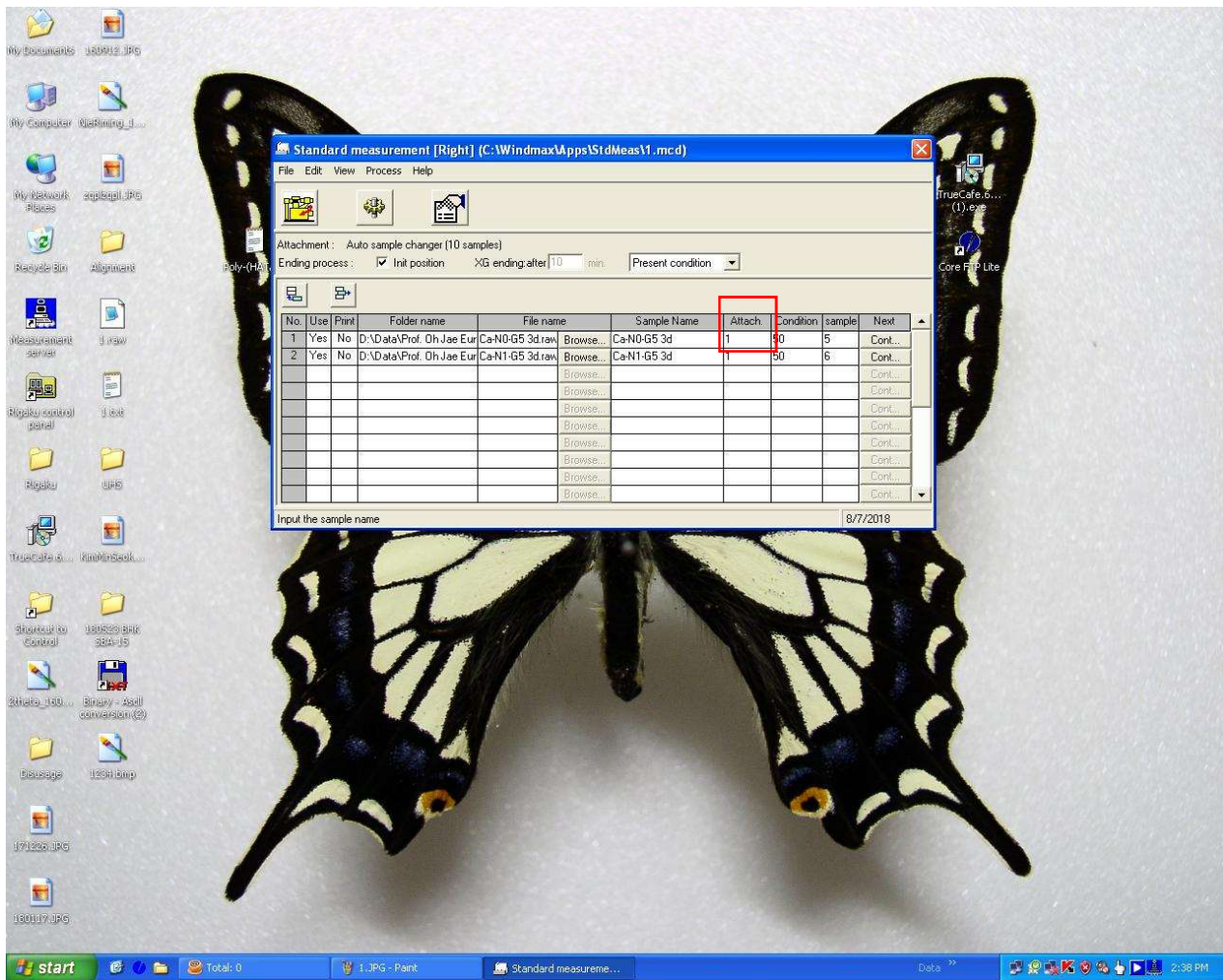
Standard Measurement (Right)

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,
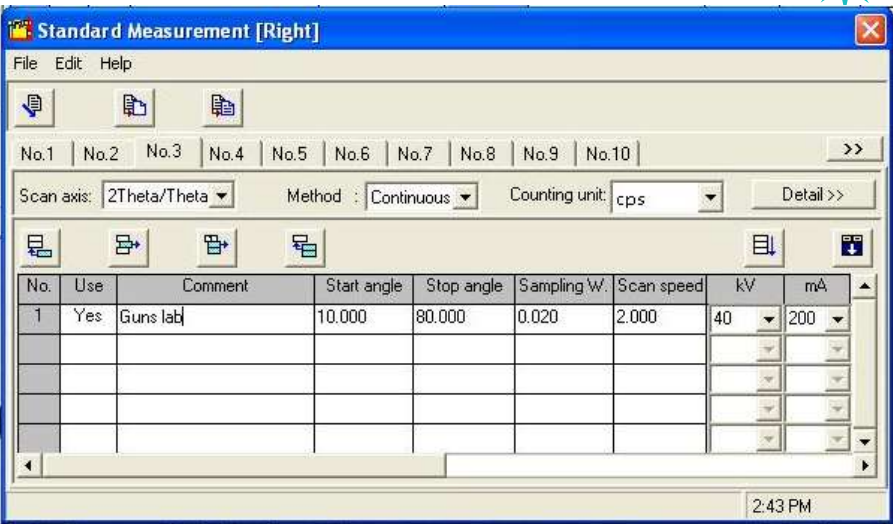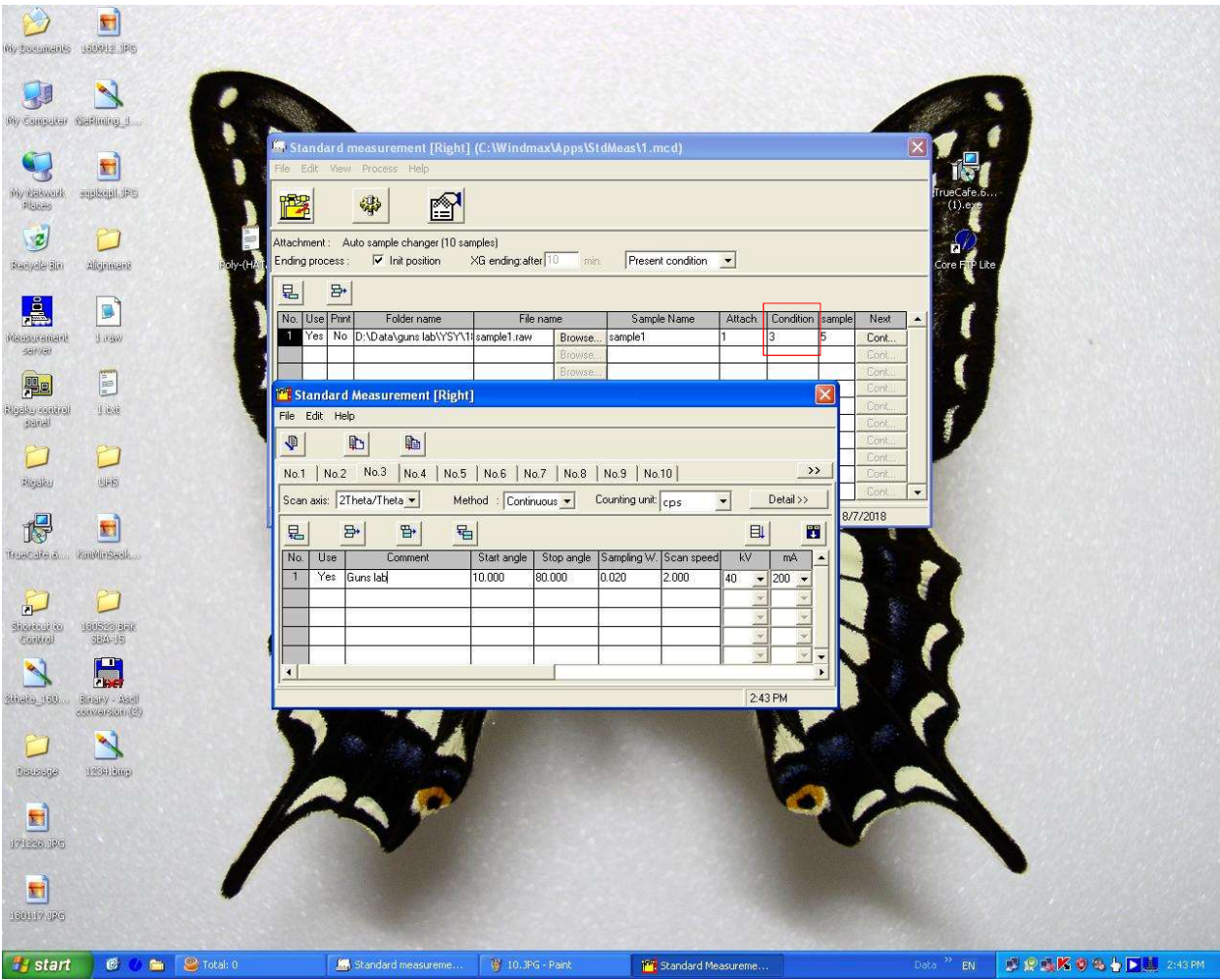
It should rotate.

Number 1 is non-rotation.

FIRST IN CHANGE

Standard Measurement (Right)

Fill in number 2 below 'Attach' and then,

new window opens.

Number 2 means rotation of 60 RPM.

FIRST IN CHANGE

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)

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

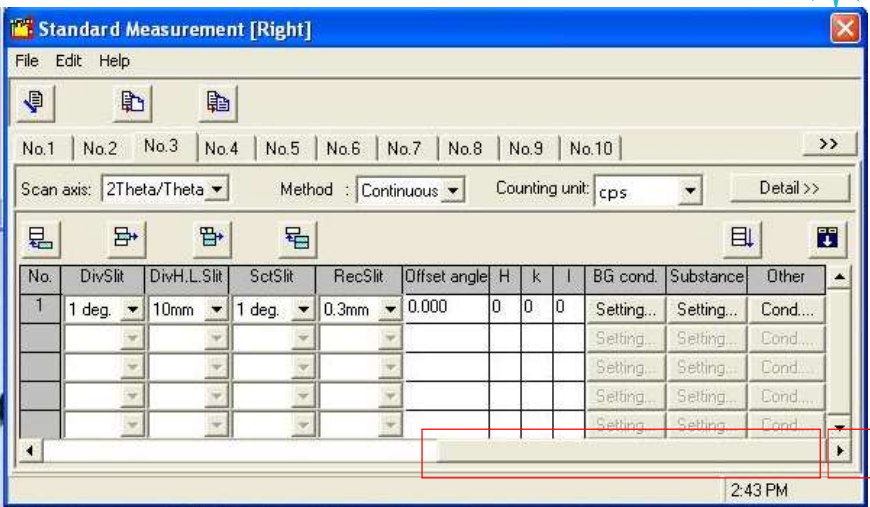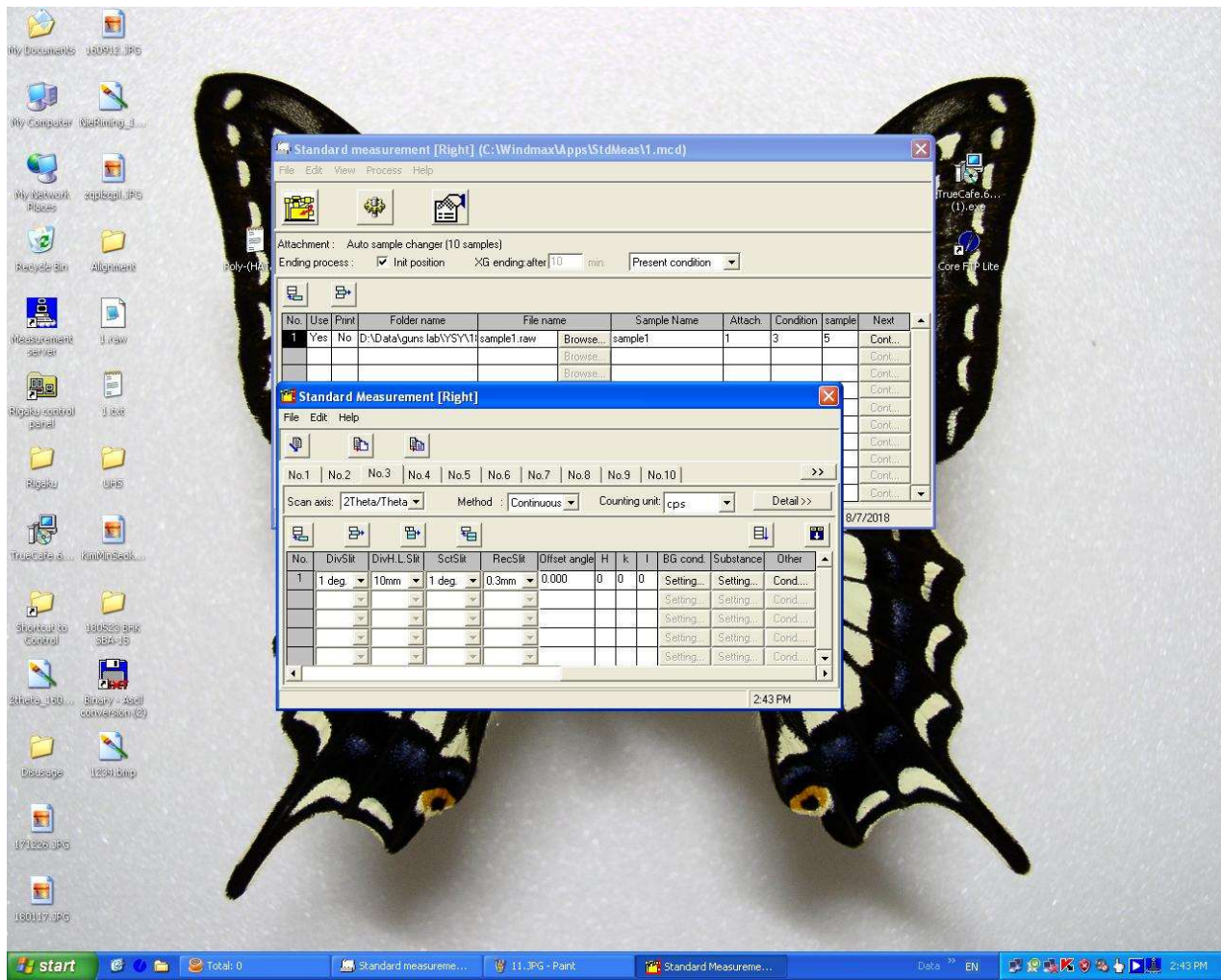Total measurement time = 80-20 = 60° /2° /min = 30min.

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

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

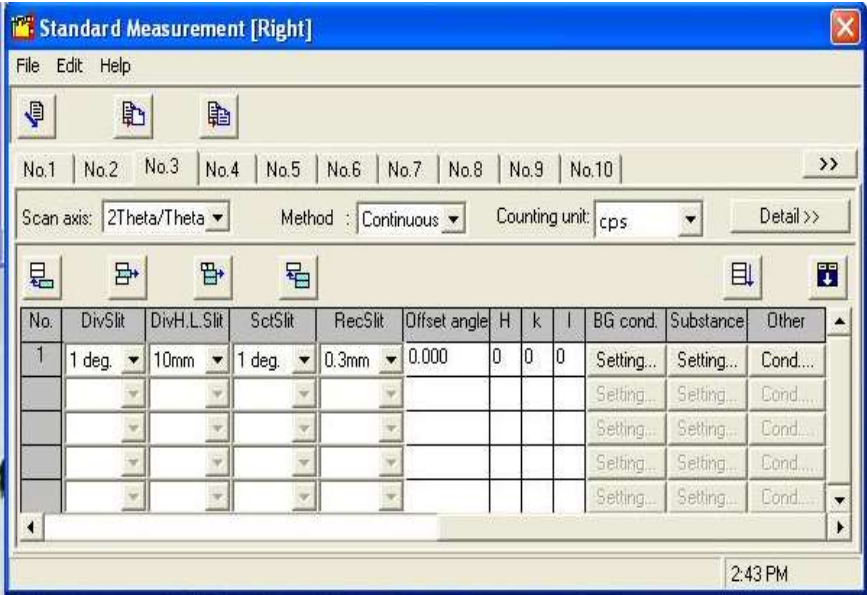In case of exceeding, be careful to occur Filament Short !

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

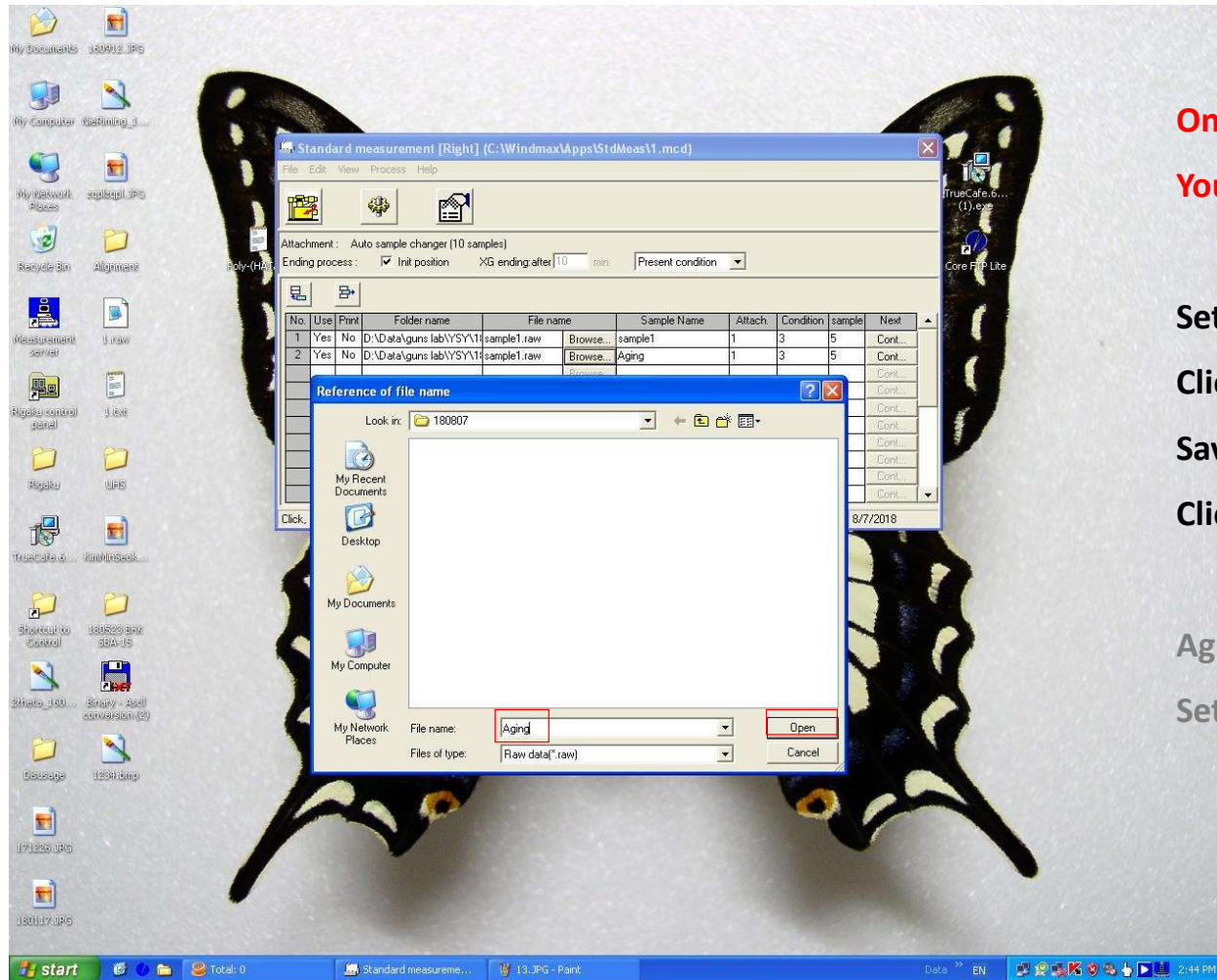# Standard Measurement (Right)
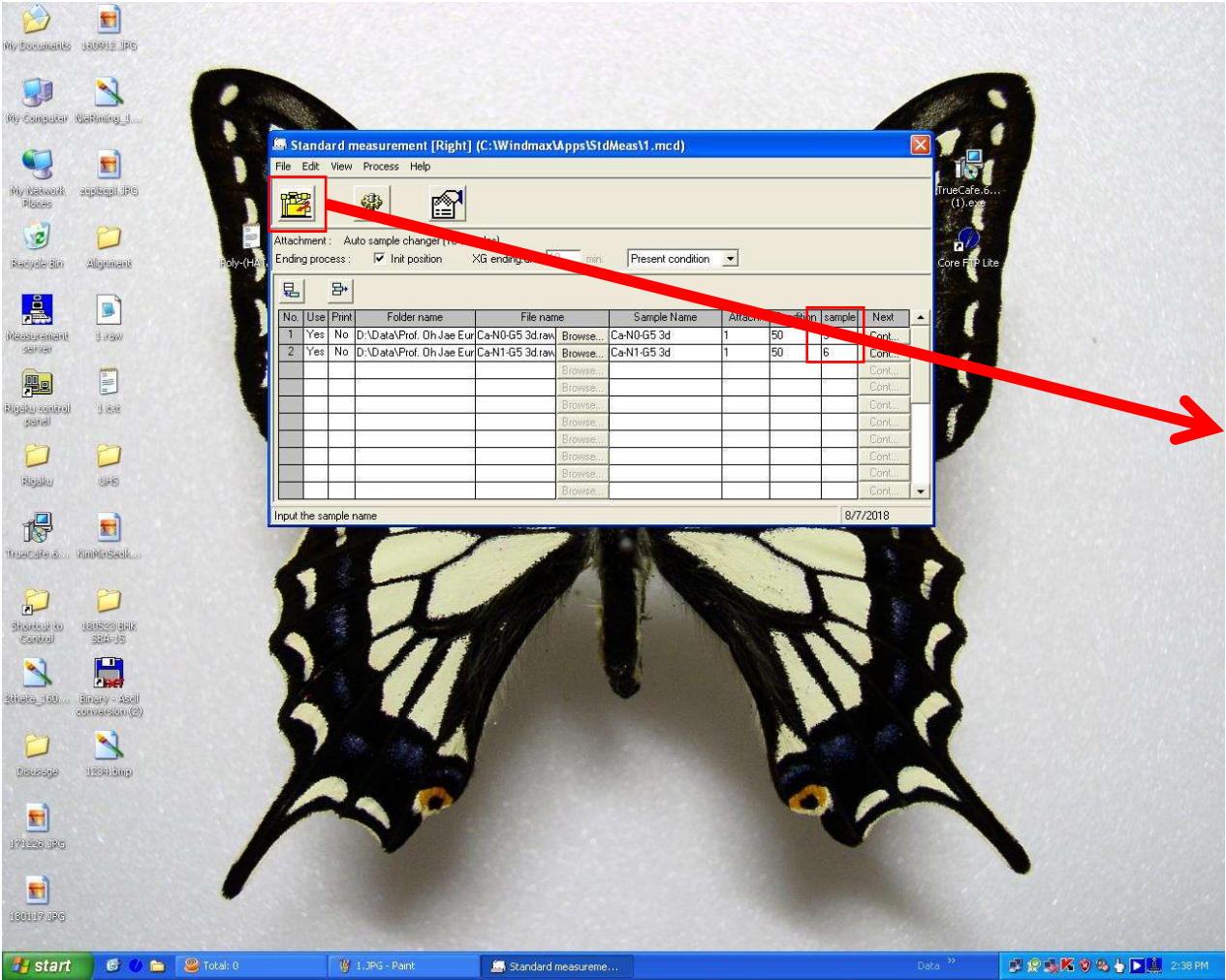
Pos means Sample Position in ASC.

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.

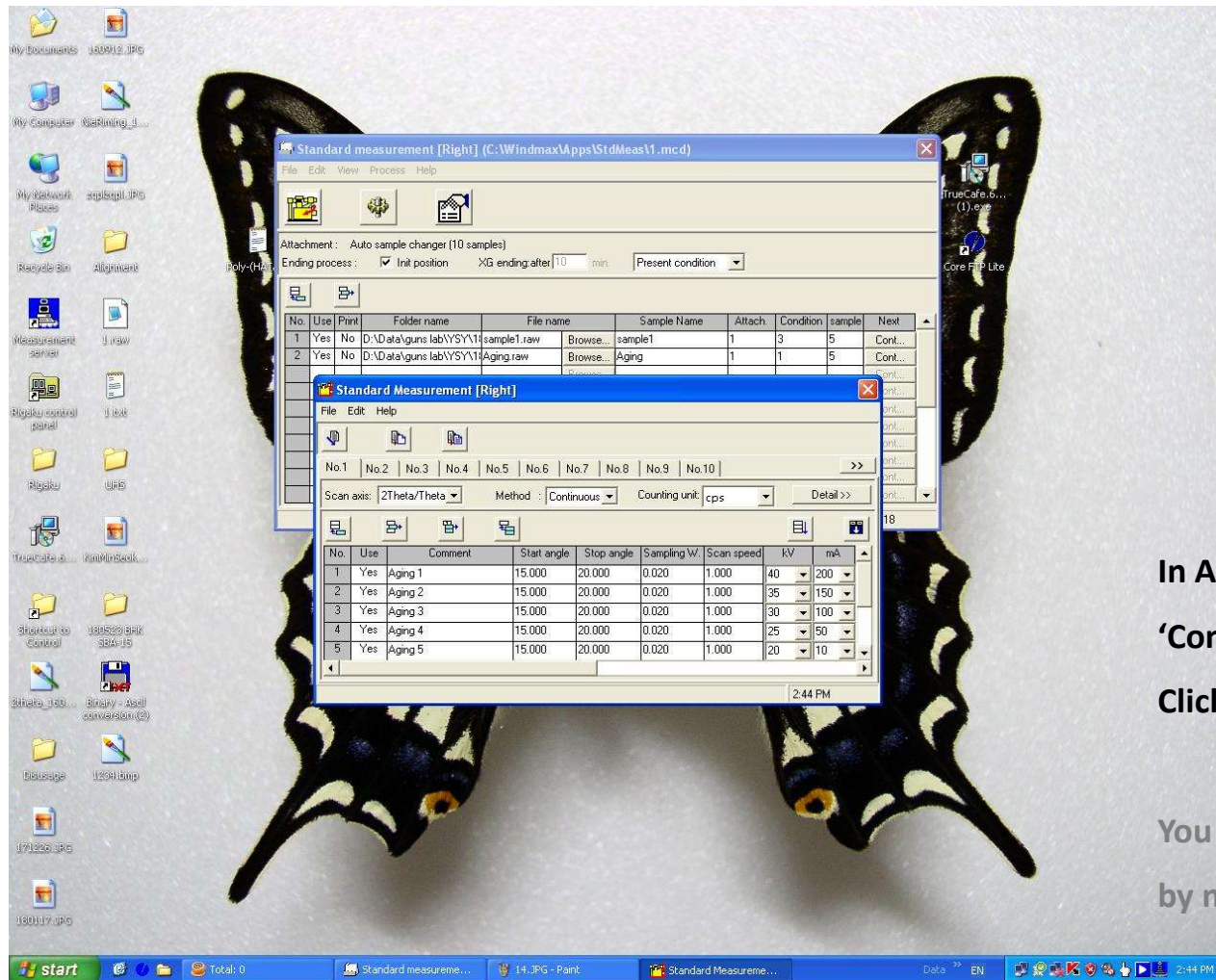Click 'Browse' button and name file 'Aging' and then

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'.

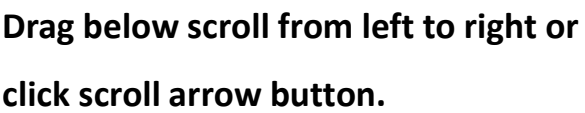Click the 'Condition value' and new window opens.

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

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

FIRST IN CHANGE

Aging Process

Drag below scroll from left to right or click scroll arrow button.

In Aging Slit condition, set 'DivHLSlit' to same condition with measurement condition and

Never change any other condition.

Click 'finish button' after checking conditions.

FIRST IN CHANGE
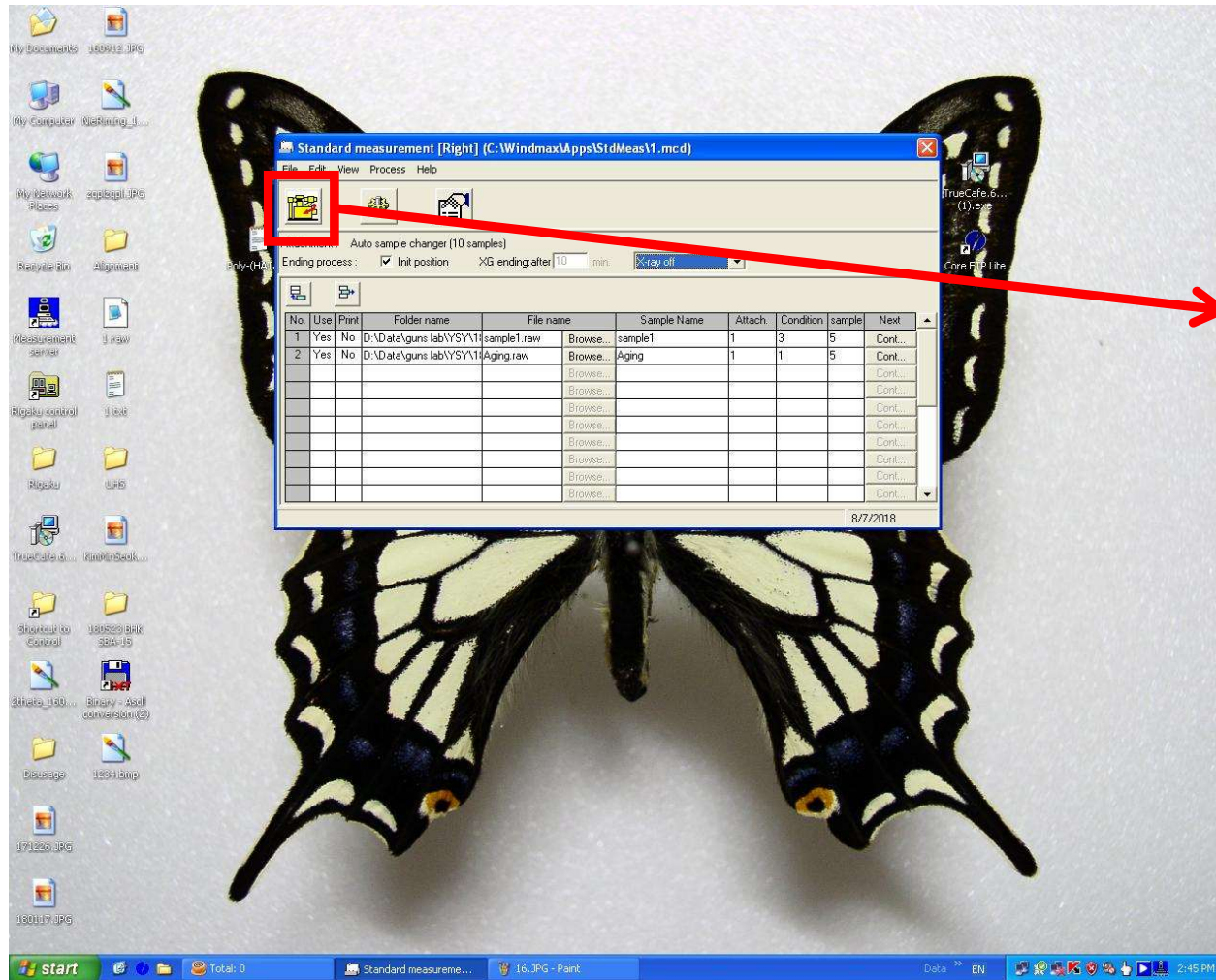
Aging Process

For Aging process condition,

Change 'Present Condition' to 'X-ray off' located next to

'XG ending after'.

And then, click 'Execution measurement' button.

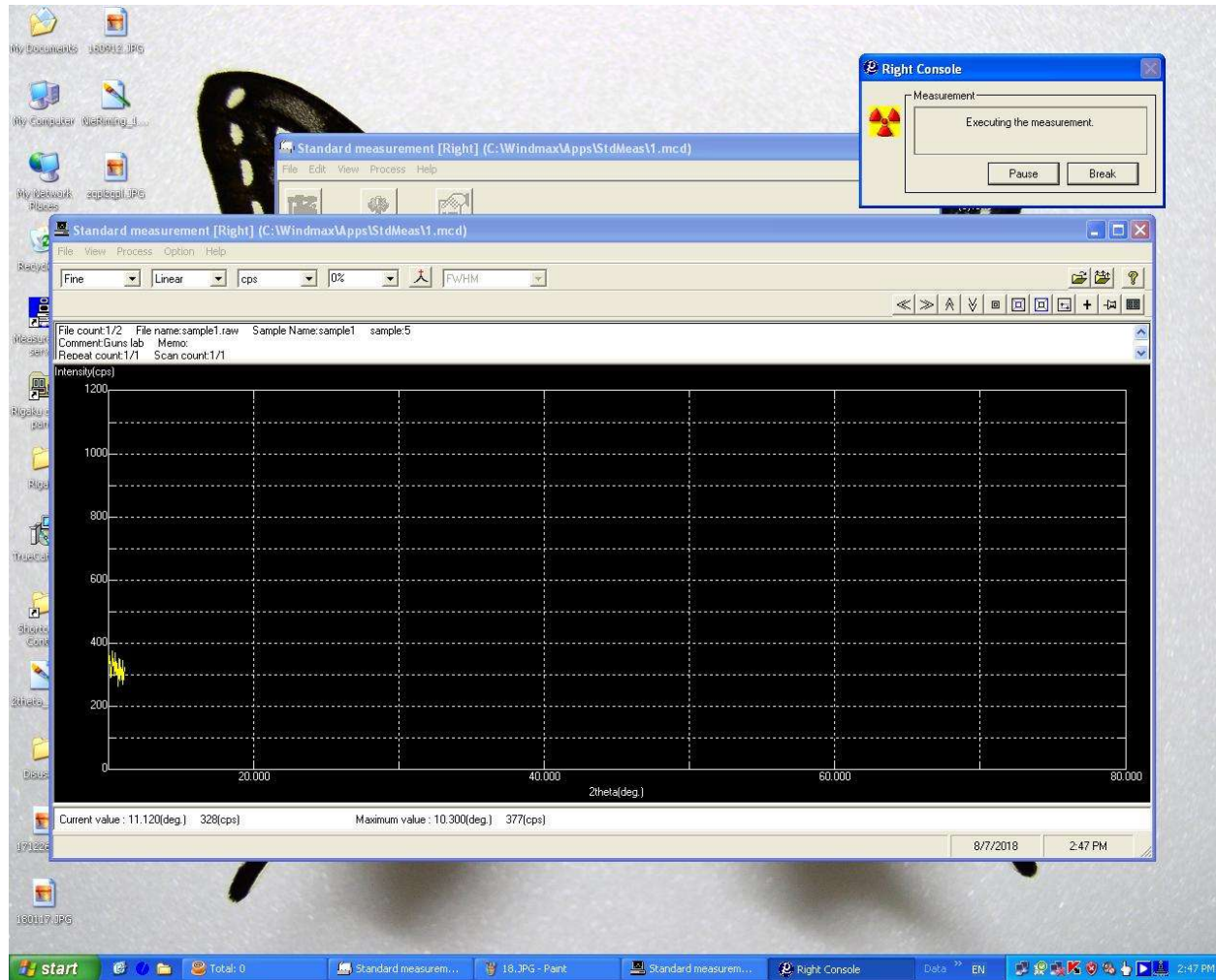After measuring 'No.1 condition',

lower power for 10min and then do X-ray off

as No.2 condition.

In case of measuring at night or holiday,
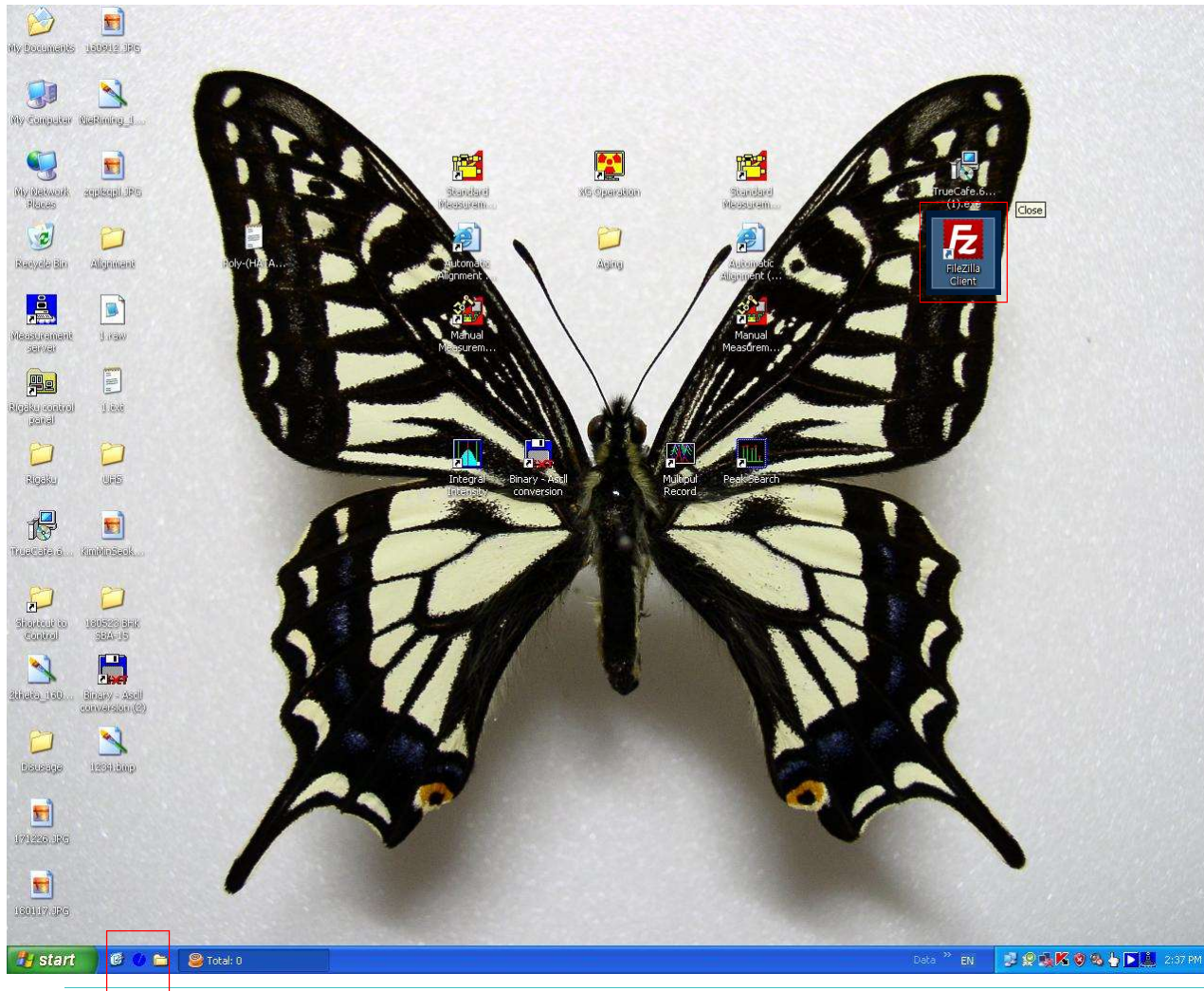
It can measure and do X-ray off automatically

Without setting Aging process.

FIRST IN CHANGE

Measurement

After starting measurement,

It does not matter you to leave XRD room
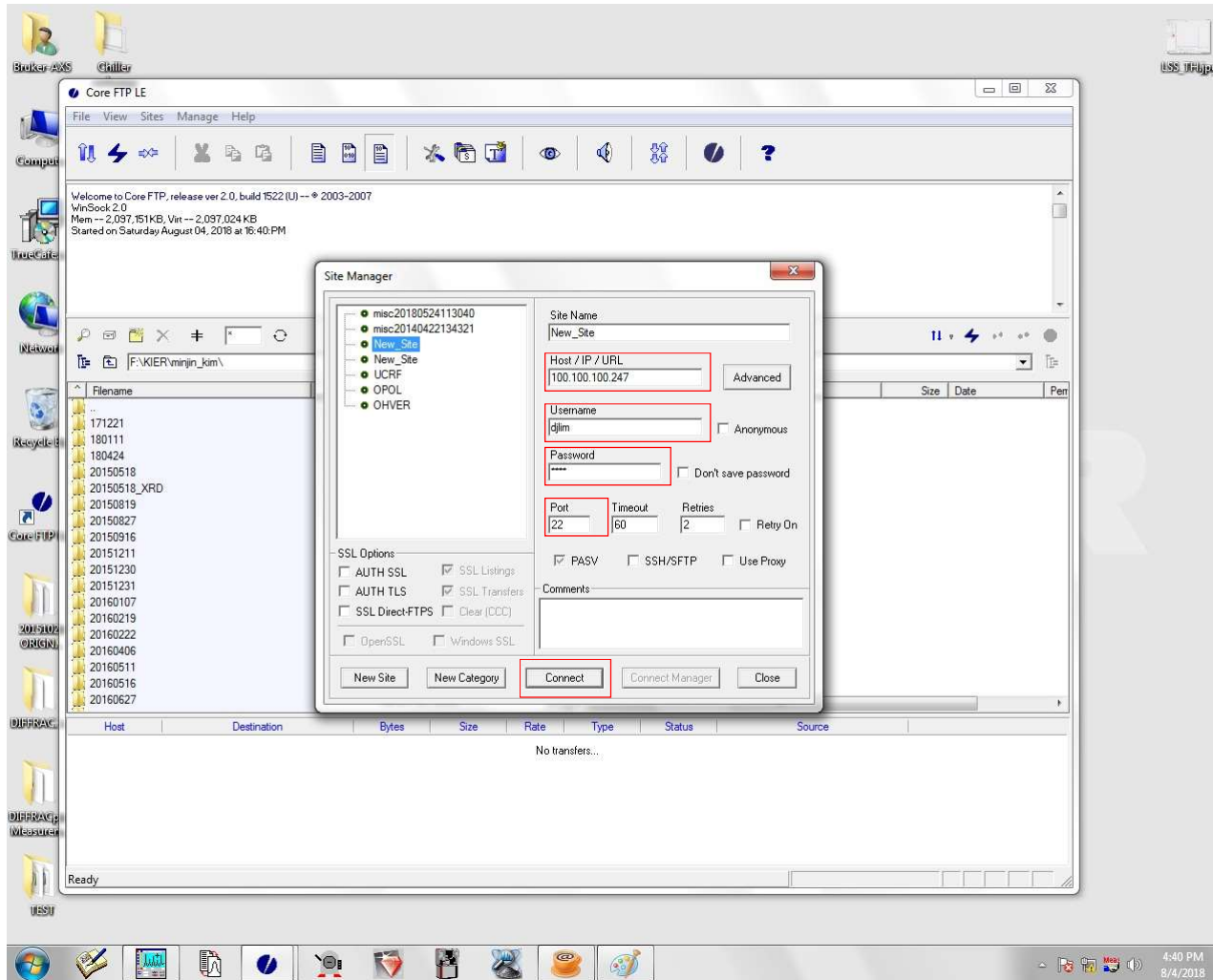
If data shows good.

Data Upload (FileZilla)

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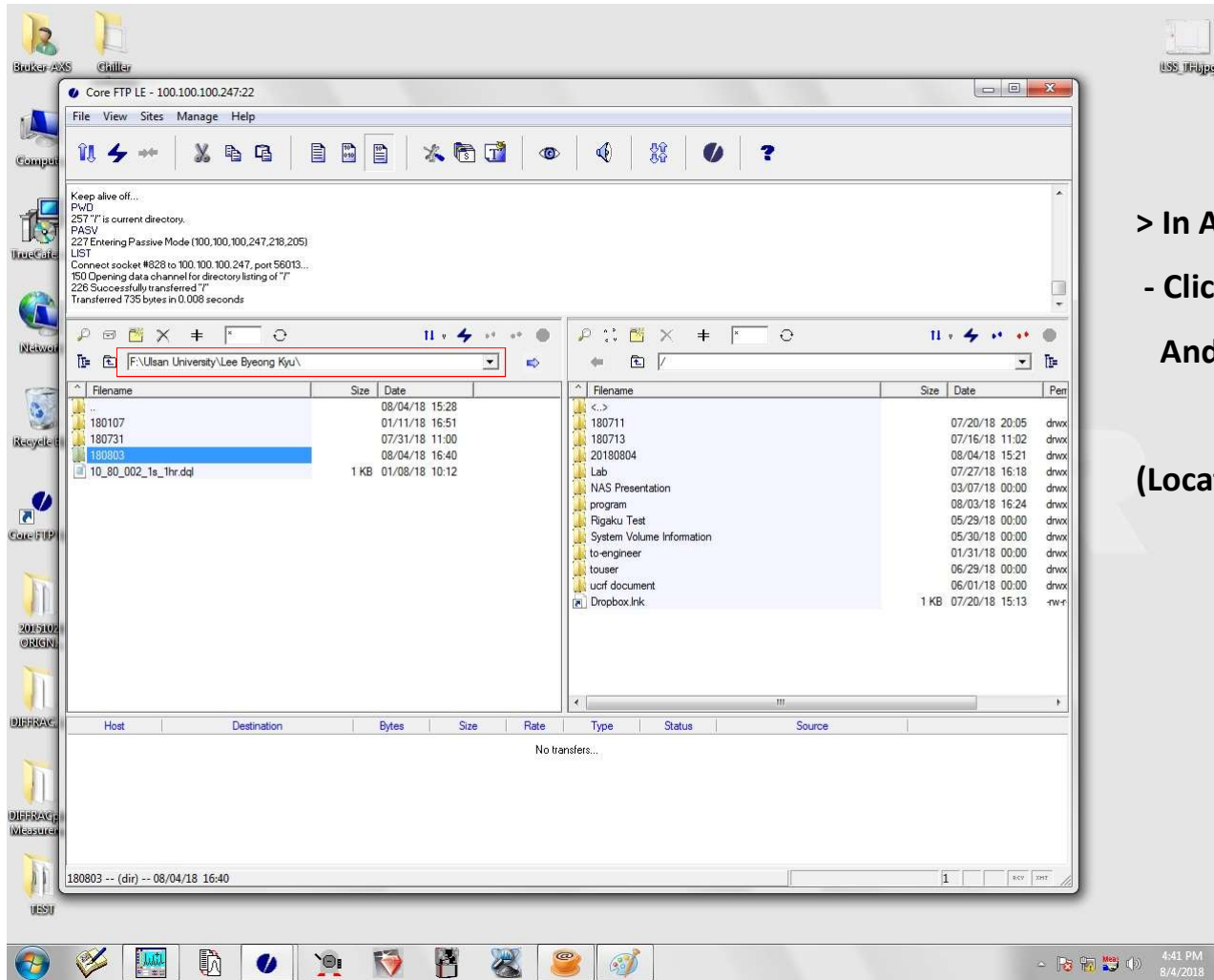
Double click one of both.

Data Upload (FileZilla)

IP: 100.100.100.30,

Username: professor name,

Password:

Port: 22

And then, Click 'Connect'
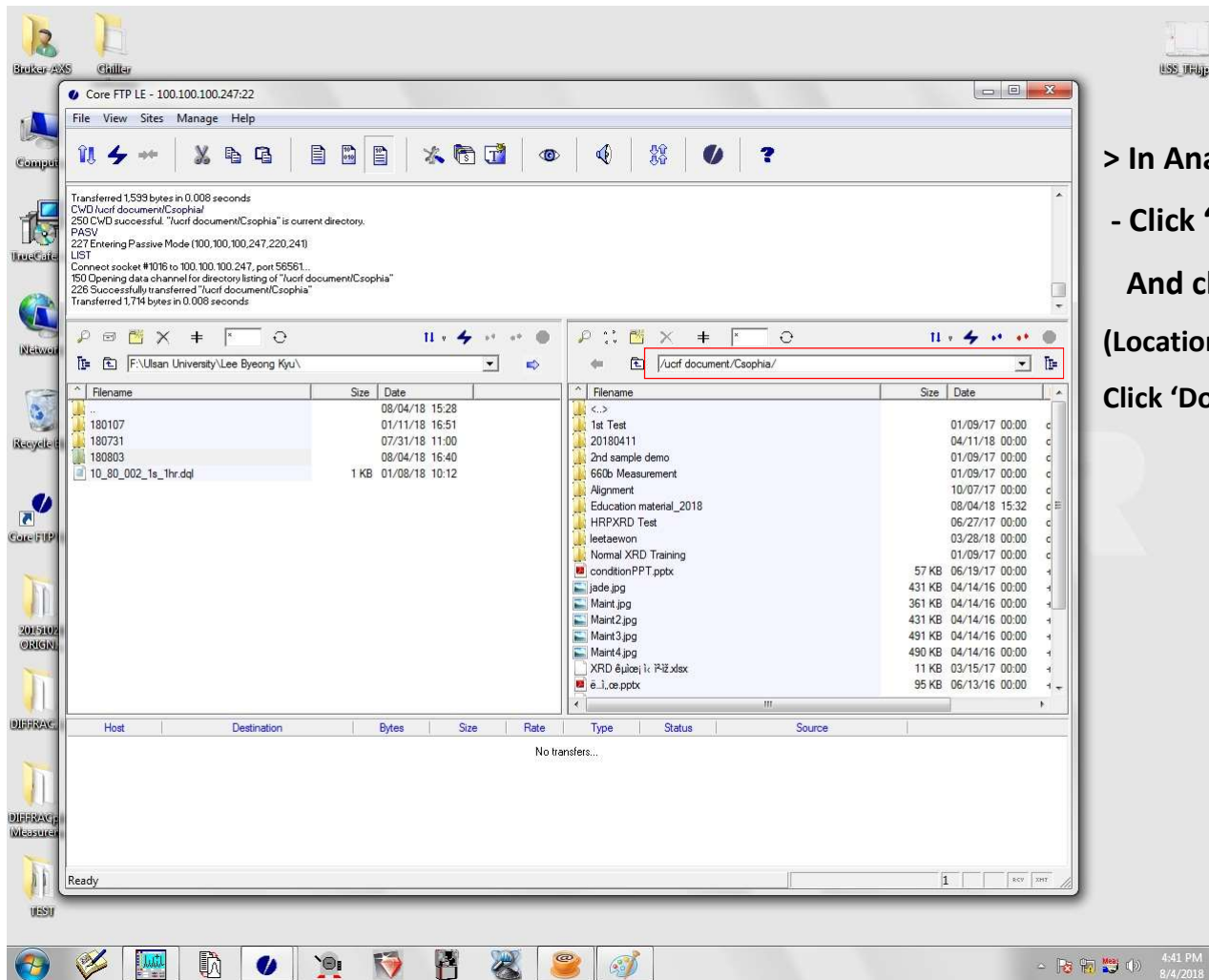
Data Upload (FileZilla)

> In Analysis PC

 - Click 'Browser' (Left page)

   And check file location that you saved.

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

Data Upload (FileZilla)
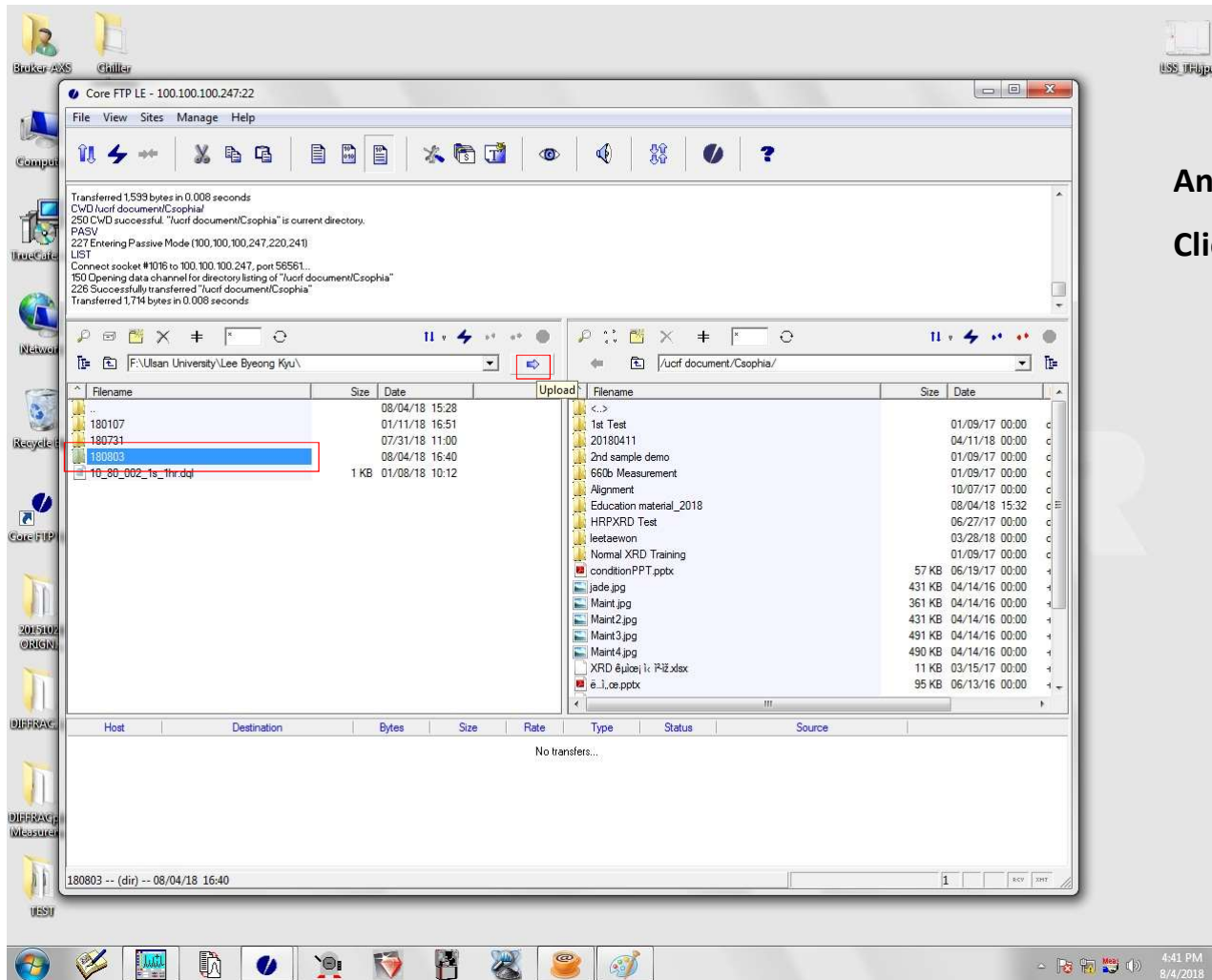

> In Analysis PC

 - Click 'Browser' (Right page)

   And check file server location that you want to save in 'FileZilla' .

(Location: D drive /Major/Lab name/Requester name/Analysis date)

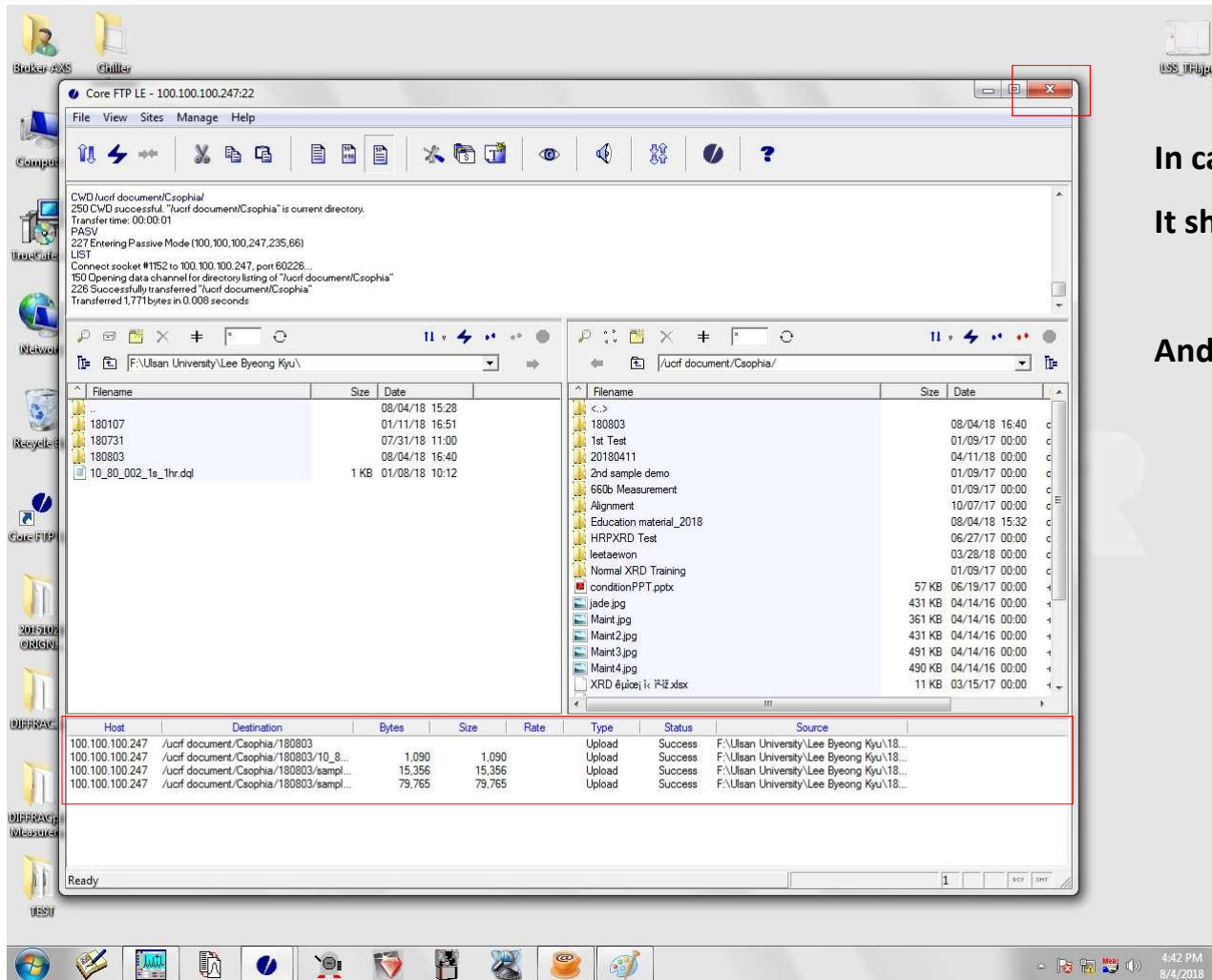Click 'Download' button after clicking folder.

Data Upload (FileZilla)

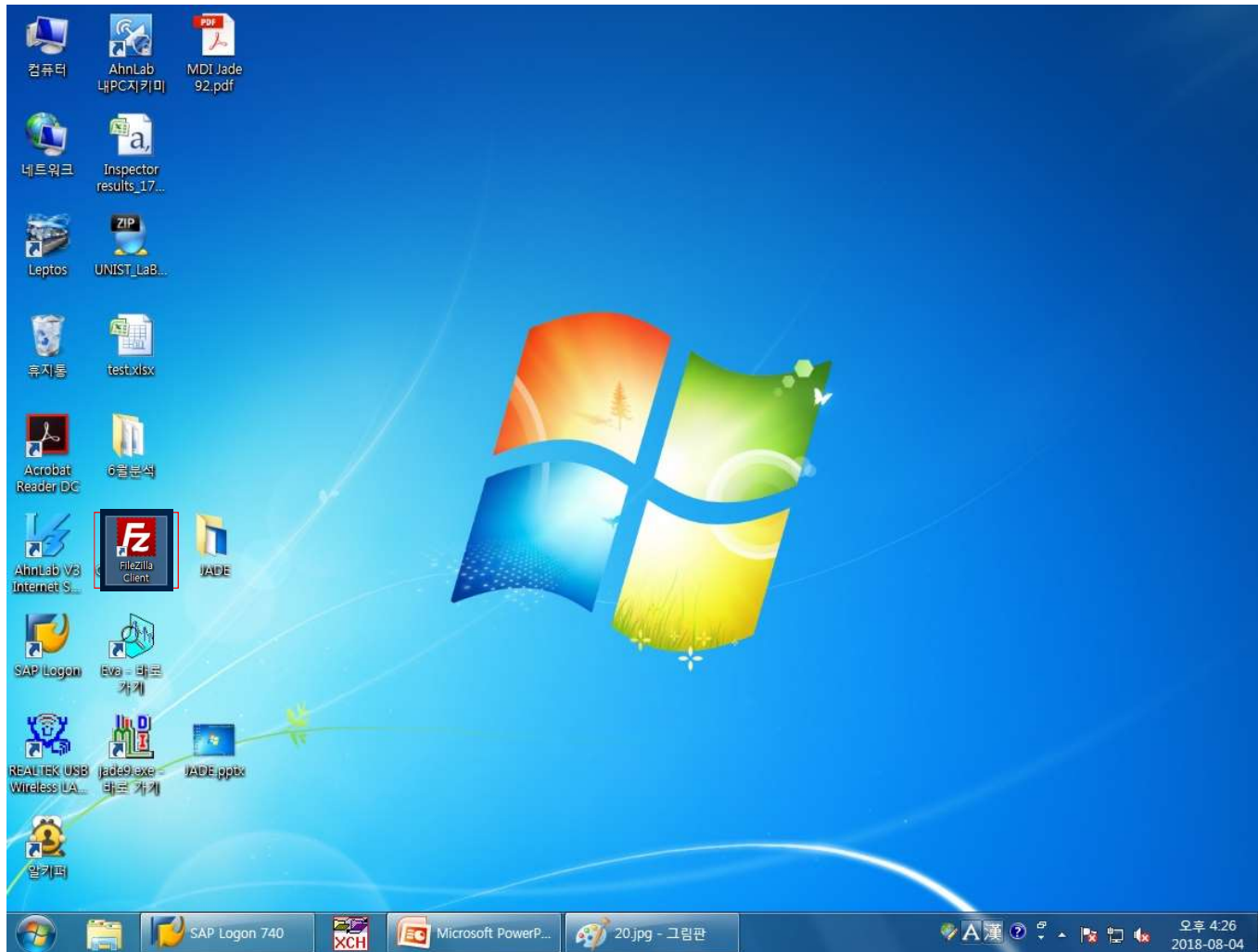**And then, Click file you want to upload in Analysis PC,**

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 'FileZilla' program.
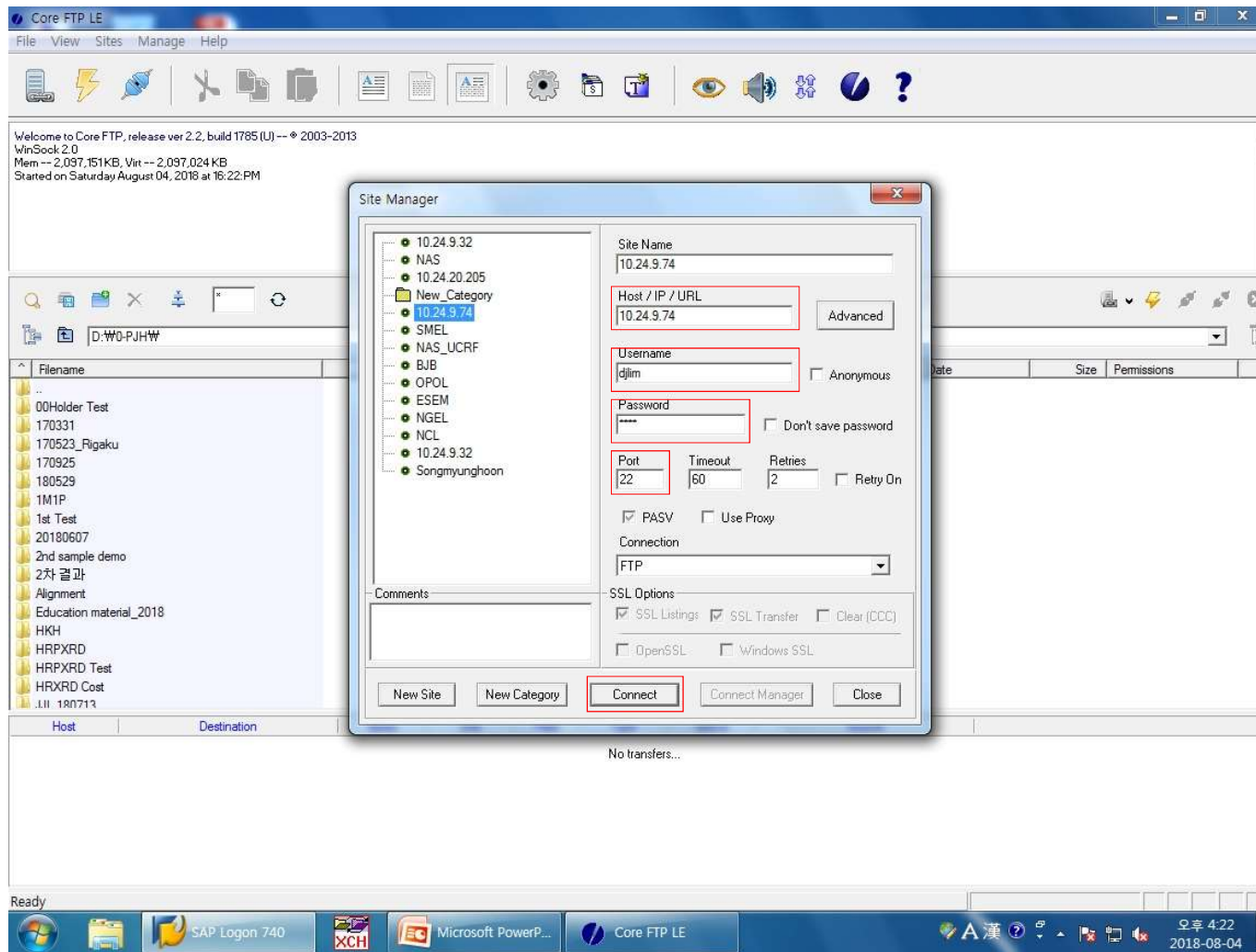
Data Download (FileZilla)

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

Double click 'FileZilla' in desktop
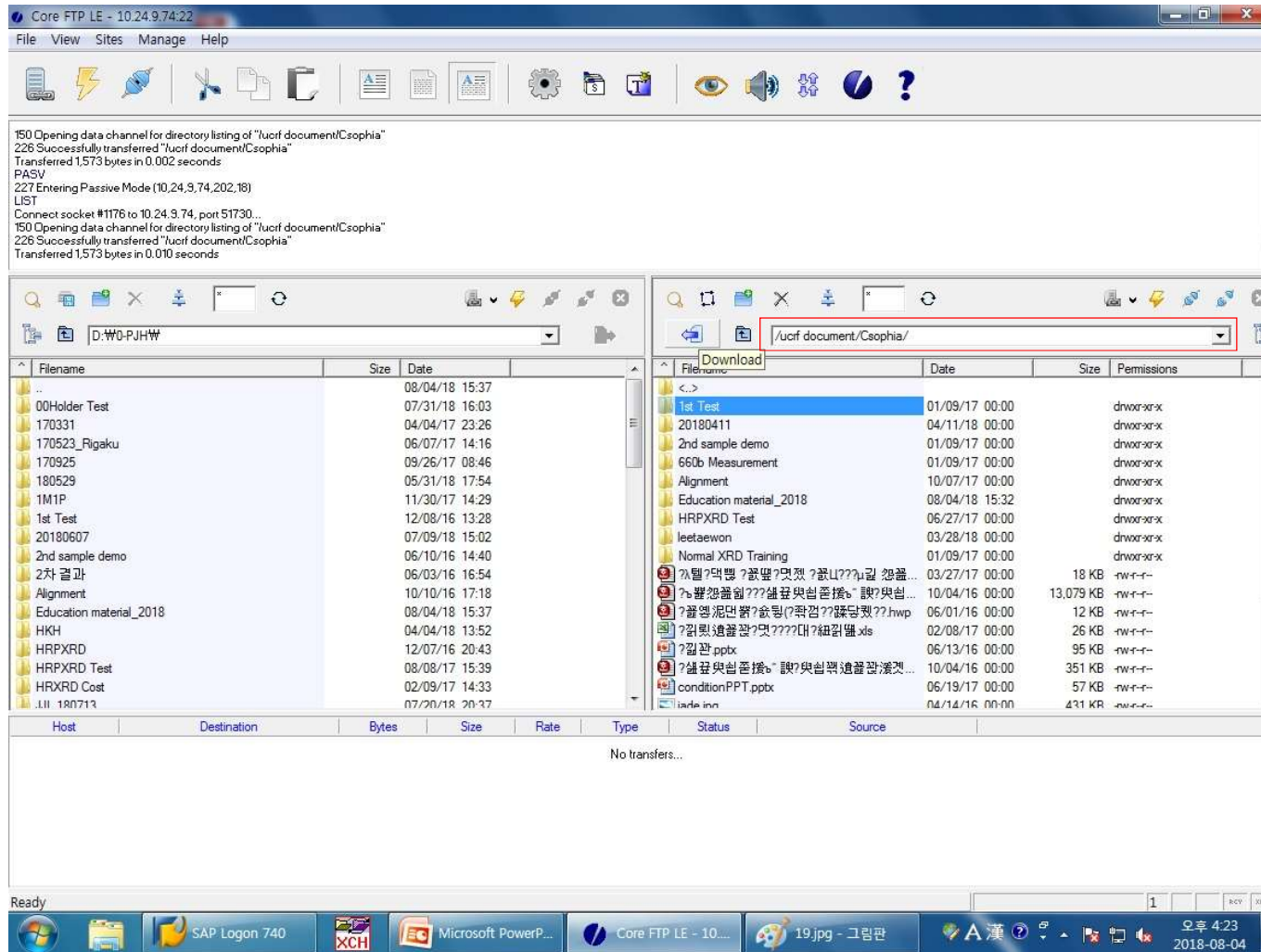
For Data Download.

Data Download (FileZilla)

IP: 10.24.9.32,

Username: professor name,

Password:,

Port: 22

Click 'Connect'.
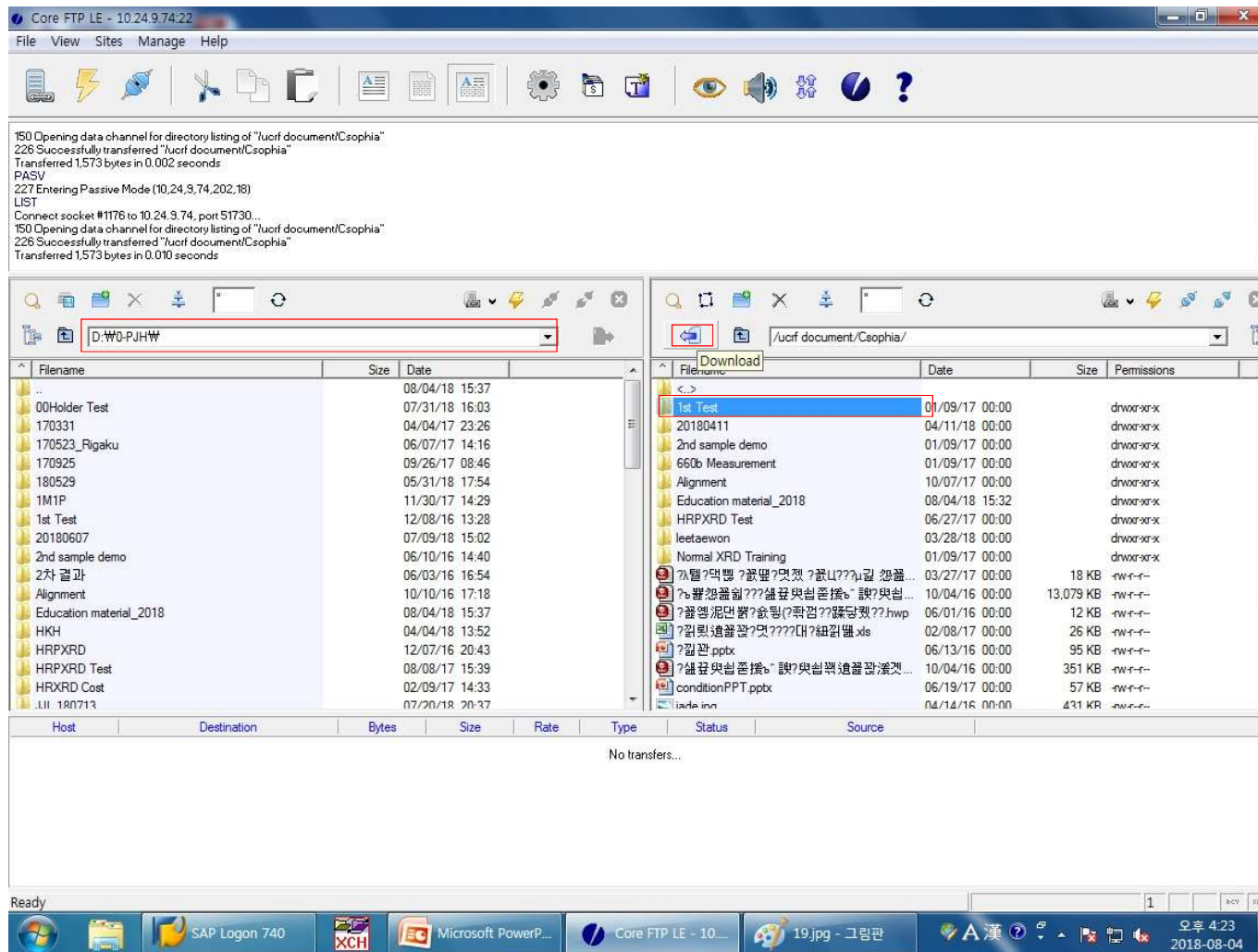
Data Download (FileZilla)

> **Move Left to Right folder**

   (opposite to Analysis PC move path)

Click 'Browse' button in Right page.

Check file location that download files exist.

FIRST IN CHANGE

Data Download (FileZilla)

Click 'Browse' button in Left page.

Check file location that you want to save.

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

Click 'Download' button after clicking folder.