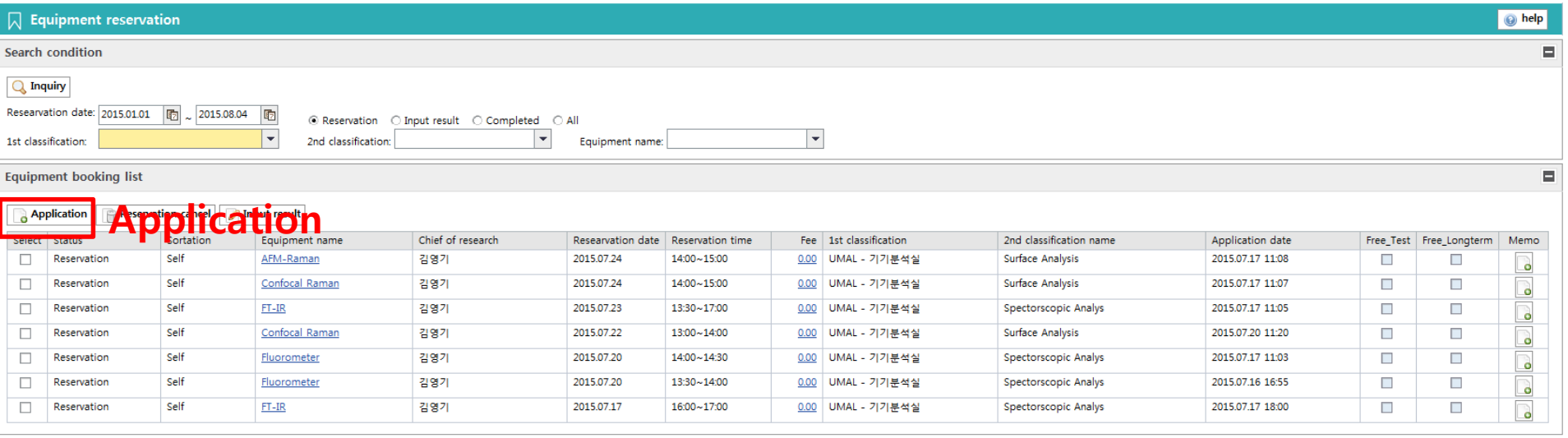
FT-IR self-user training

SEON HYE SON

UNIST Central Research Facilities (UCRF)
Ulsan National Institute of Science and Technology (UNIST)

Reservation



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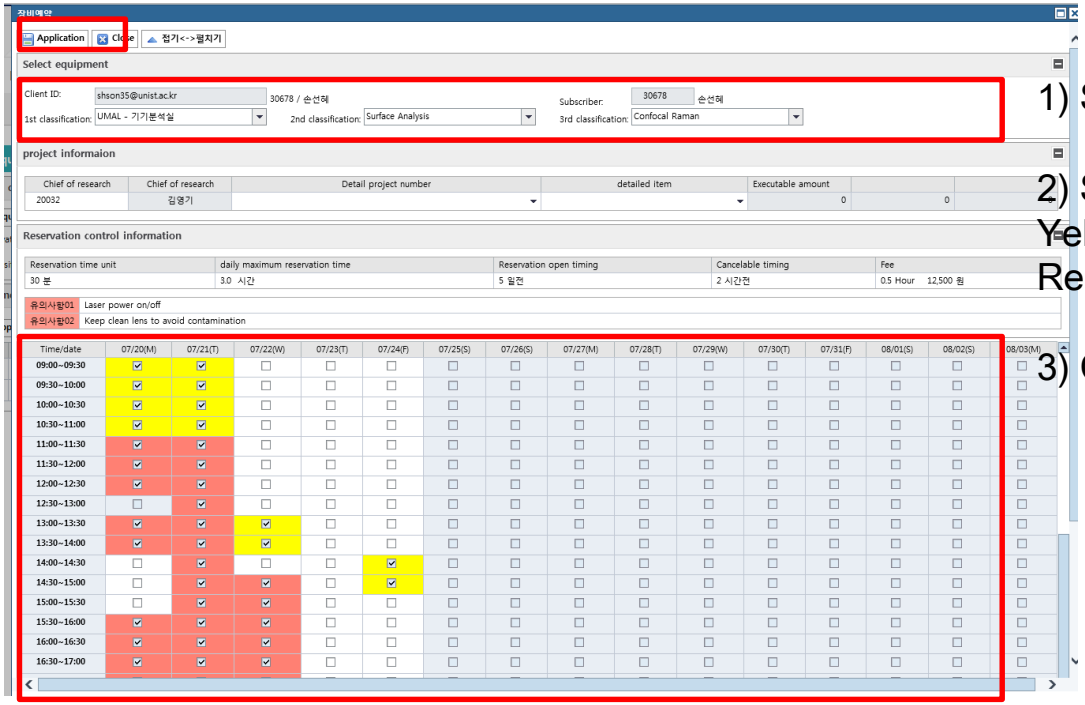


1)

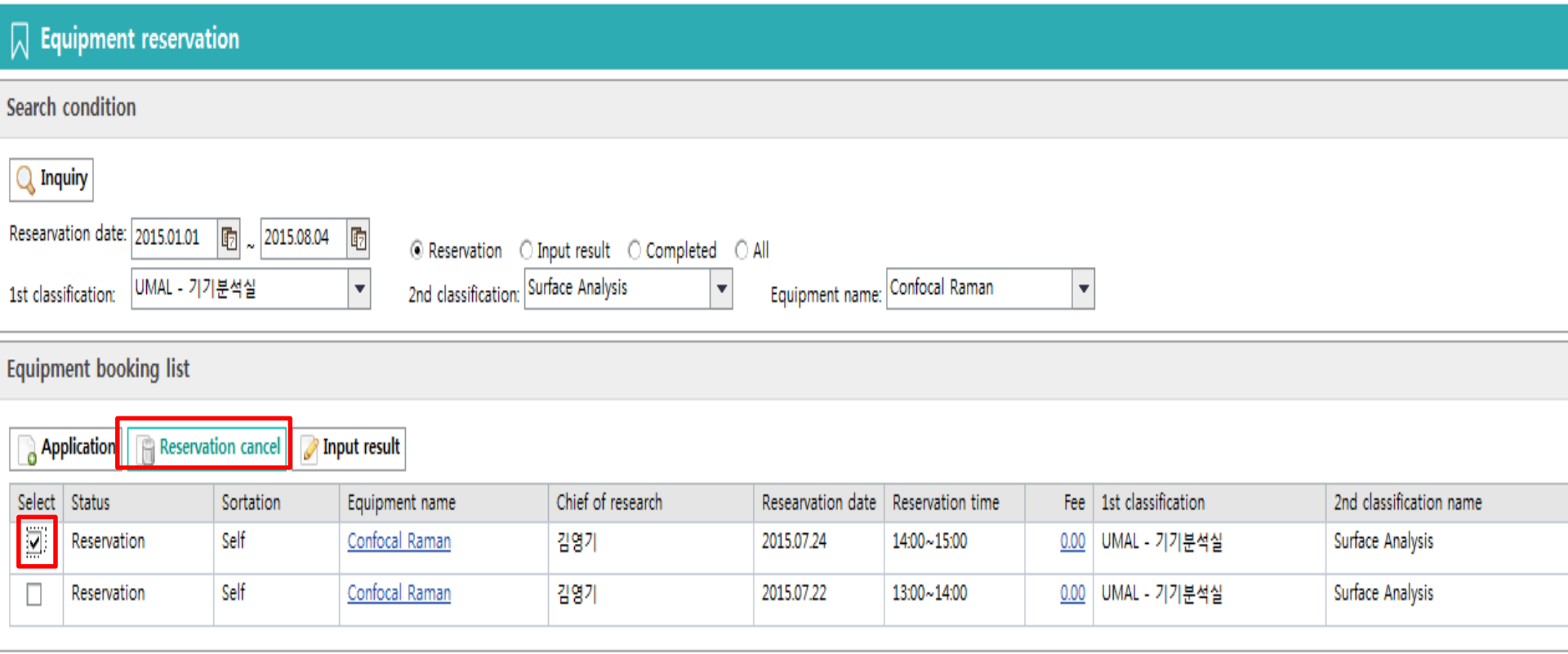
2)

1) Select the classification and equipment

2) Select the time you want on white box.
Yellow box : my reservation
Red box : others reservation

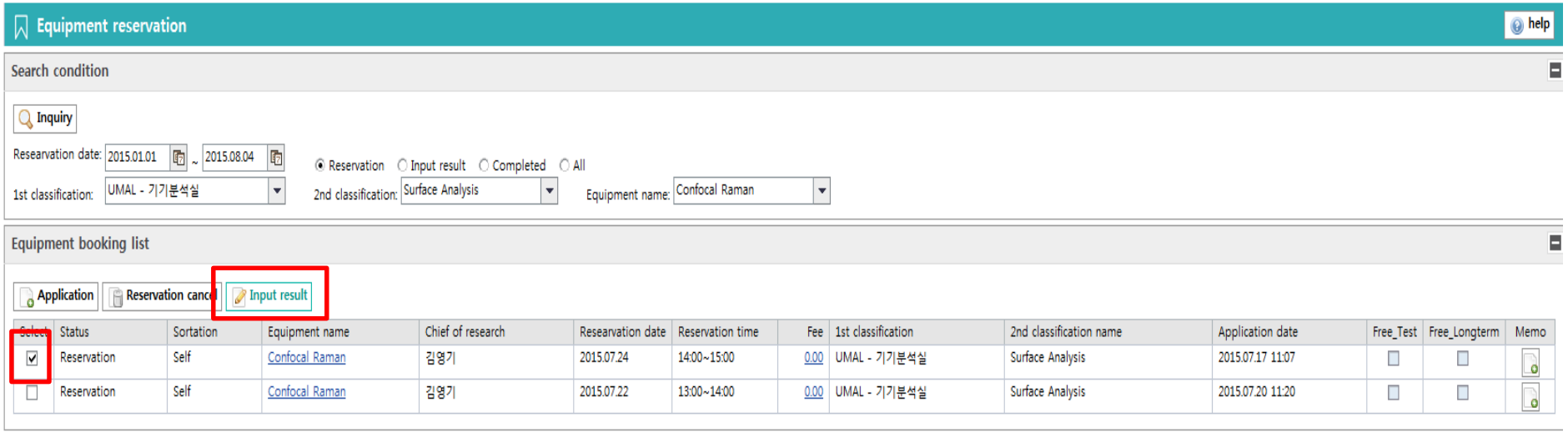
3) Click application

Reservation cancel

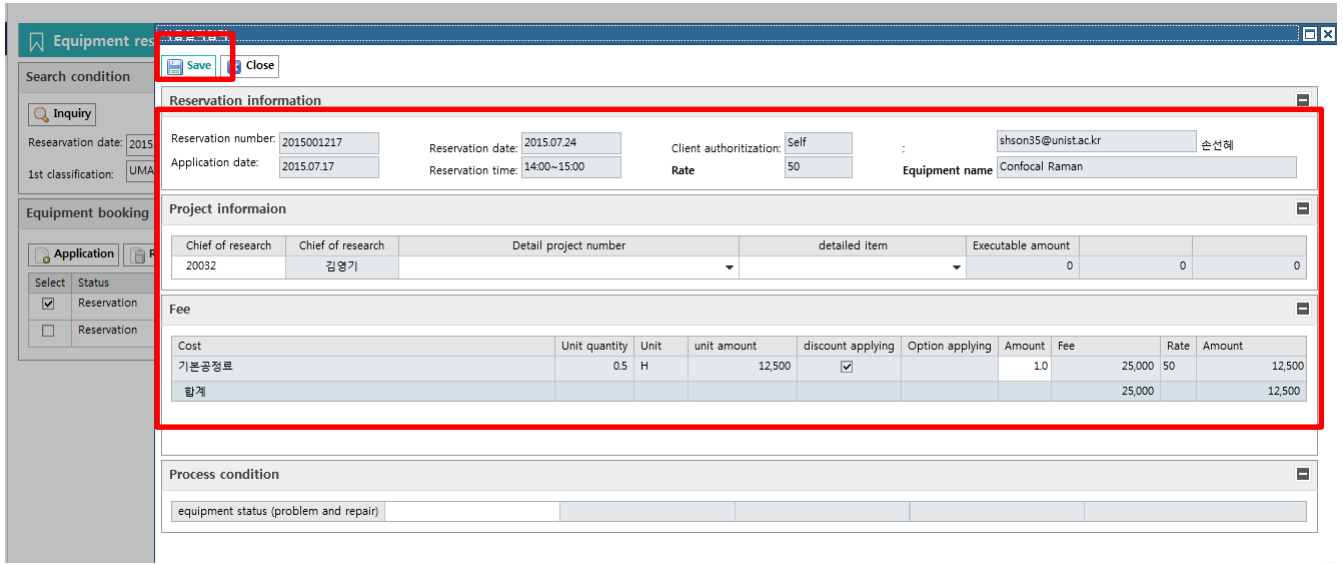


1) Select the reservation.
2) Click the ‘reservation cancel’.

Input result

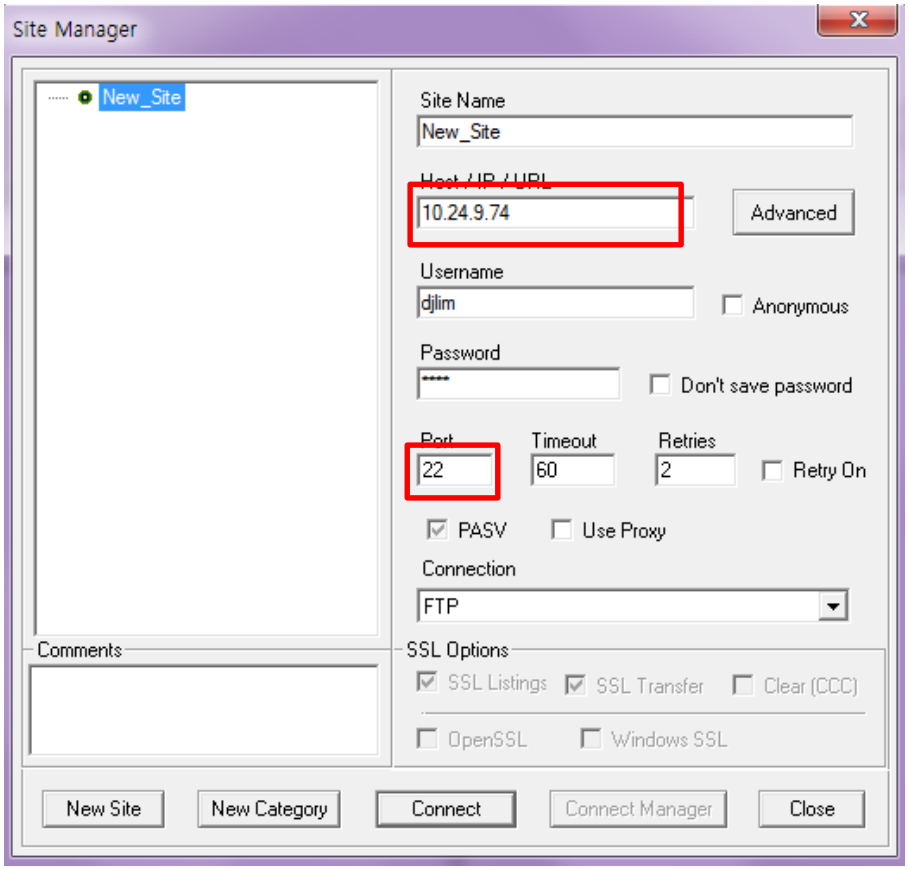


1) Select the reservation.
2) Click the ‘Input result’.
3) Check the information and click ‘save’

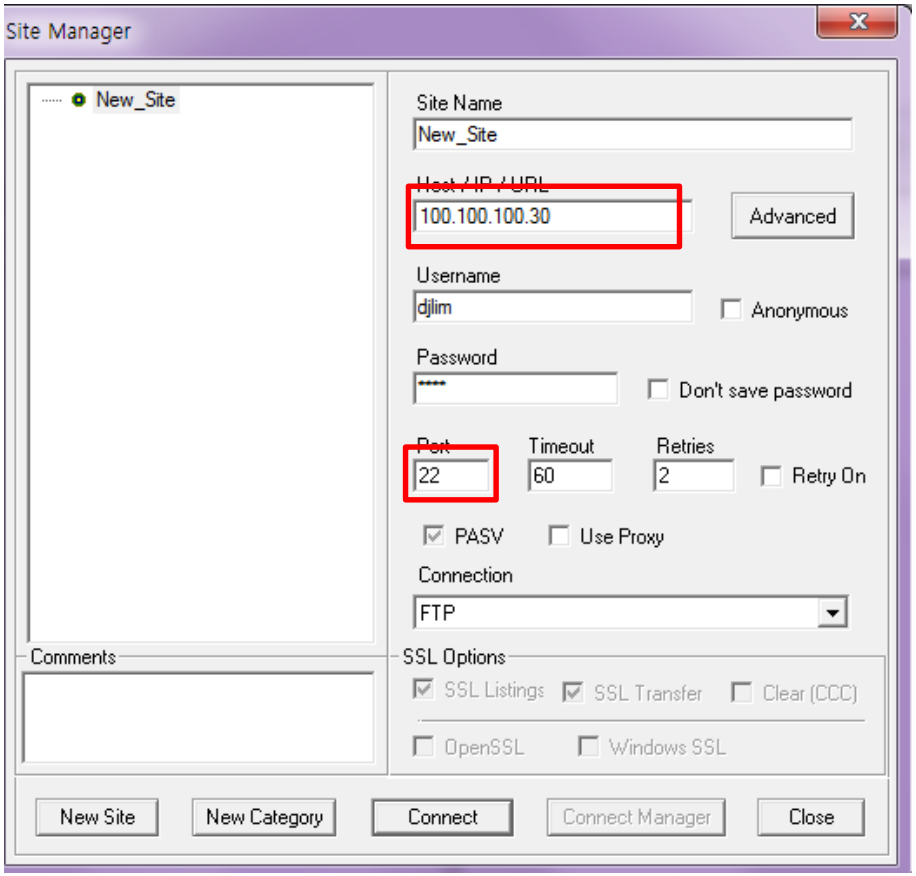


Data upload and download

My PC

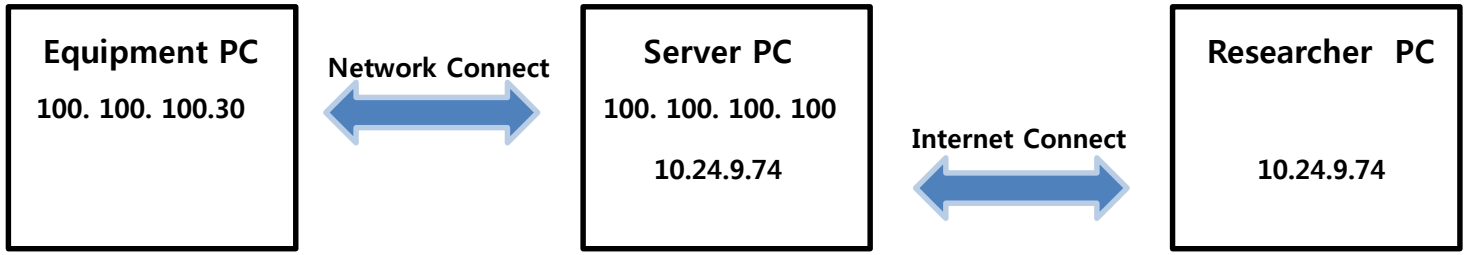


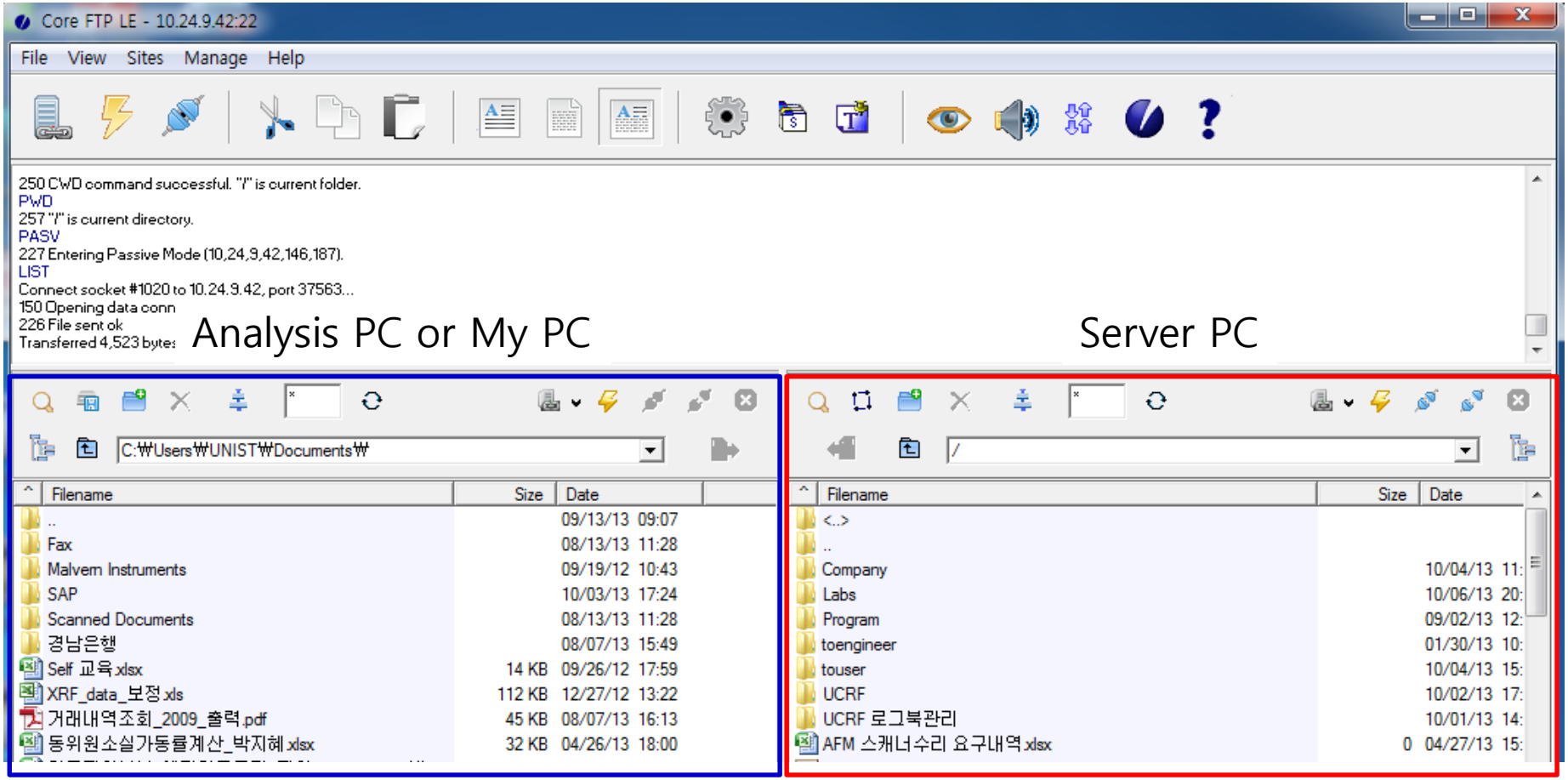
UCRF PC



Common ID : djlim
Common PW : 0254
Port : 22

Data upload and download





Guideline for the Operation of the UCRF

Article 1 (Purpose)
This guideline is intended to provide detailed requirements for operating the Central Research Facilities at Ulsan National Institute of Science and Technology (UNIST) (hereinafter referred to as “UCRF”) in accordance with Article 10, Operational Regulations of Central Research Facilities at UNIST.

Article 2 (Scope)
This guideline shall apply to faculty, graduate students, undergraduate students and researchers at UNIST, as well as external clients, who request services from UCRF, and equipment managers.

Article 3 (Definitions)
Terms used in this guideline shall be defined as follows:
“Autonomous use” means that UNIST faculty members or students use UCRF's equipment without any help from the equipment manager.
“Autonomous user” refers to users who have qualifications for the “autonomous use” of the equipment in paragraph 1 above, according to procedures set by UCRF.
“Request for analysis and processing” is a request to the equipment manager to perform a series of analyses and processes, so autonomous users can use UCRF's common equipment to obtain the results of a test analysis or process.

Article 4 (Access Management)
① If any personnel want authorized access to laboratories with restricted access, they must fill out an application form and receive approval from the supervising professor and Center manager to register their ID.
② If any personnel needs to access laboratories for equipment maintenance and repair, they must be accompanied by a competent manager or have the manager's approval to gain access to the labs.
③ For laboratories that require safety training for personnel with access, approval for access will be withheld until they complete prior training, as specified for each laboratory.

Article 5 (Requests for Analysis and Processing)
① If a client requests for analysis and processing that can be supported by UCRF, the client should discuss with the equipment manager beforehand.
② A client who requests analysis and processing shall cooperate with the equipment manager in identifying the necessary information needed to maintain the normal operations and safety of equipment or facilities.
③ Analysis and processing services will be available to clients on a first-come-first-serve basis. In any special circumstances such as equipment inspection and repair is needed, requests for such services may be reserved or cancelled at the equipment manager's discretion.

Guideline for the Operation of the UCRF

④ If there are no special requests from the client, each manager may discard any specimens that are seven days or older after the results-notice date, and may also discard the outcome or results data produced by the analysis and processing service three months from the day of said notice or later.

Article 6 (Qualifications for Autonomous Use)

① Authorized persons who qualify for autonomous use shall be limited to graduate students, researchers, professors and authorized undergraduate students (with the supervising professor's approval) at UNIST.
② Qualifications for autonomous use shall be granted to any persons who satisfy the requirements specified by each laboratory (e.g. safety training, equipment user training, evaluation, etc.).
③ A list of autonomous users shall be updated every 6 months and shall be published on the UCRF homepage.
④ An autonomous user's qualifications may be cancelled if the equipment manager deems it necessary, or if the user does not frequently use the equipment (less than the minimum limit of 10 times in the last 6 months). In such cases, users may discuss with the manager and go through a re-orientation process to be qualified for autonomous use again.

Article 7 (Responsibility of Autonomous Users)

① Autonomous users should follow the instructions for using the equipment as they learned during the orientation. If there is something significant to report, they must discuss with a competent manager and help operate and maintain the safety of the research equipment facilities.
② Autonomous users will be liable for any accidents, equipment damage, failure and loss incurred as a result of their negligence when using the equipment.
③ Equipment reservations should be made a day (24 hours) prior to when they need to use the equipment, and may be cancelled no later than 12 hours before the booked start time. If a user wants to cancel their reservation, they must inform the equipment manager via phone or e-mail during regular work hours (weekdays: 09:00 - 18:00) or via e-mail during off-hours.
④ Any reservations that are made less than 24 hours in advance may be cancelled before the booked start time. If users want to cancel their reservation, they must inform the equipment manager via phone or e-mail during regular work hours (weekdays: 09:00 - 18:00) or via e-mail during off-hours.
⑤ After using the equipment at night or during the equipment manager's off-hours, authorized users should make sure the laboratory is put back in order, the lights are turned OFF, and the entrance door is properly locked before leaving.

Guideline for the Operation of the UCRF

Article 8 (Restrictions for Autonomous Use)

① For the convenience of other users, a comfortable research environment, and to promote proper use of the equipment, UCRF may sanction users.

② Sanction criteria from the above paragraph 1 shall follow "Table 1. Penalty Points and Sanction Criteria for Users of Common Equipment."

Article 9 (Billing for Test Analysis Fees)

① Clients or autonomous users will receive bills for test analysis fees in the following month after the analysis and processing has ended, and may only pay for these bills to UCRF's bank accounts.

② Clients or autonomous users shall follow the specified procedures to pay bills charged under the standards of test analysis fees in accordance with Article 8, "Operational Regulations of Central Research Facilities at UNIST."

③ The standards of test analysis fees, as stipulated in Article 7, Operational Regulations of Central Research Facilities at UNIST, may be provided to clients or users before request or use.

④ If this is their first request or first time using the equipment, clients and users should submit copies of their business license and their bank book to UCRF's administrative offices.

⑤ When there is any change to the business license, they shall inform the administrative manager of the change and send a copy of the new business license to the manager.

⑥ Bills for test analysis fees shall be issued by UCRF's administrative office, and clients or users shall pay the bill to UCRF no later than 1 month after the bill is sent to them. If the payment is overdue, UCRF may stop supporting services for users and laboratories in arrears.

⑦ If more time is required for analysis and process due to negligence on the part of clients, additional test analysis fees may be charged.

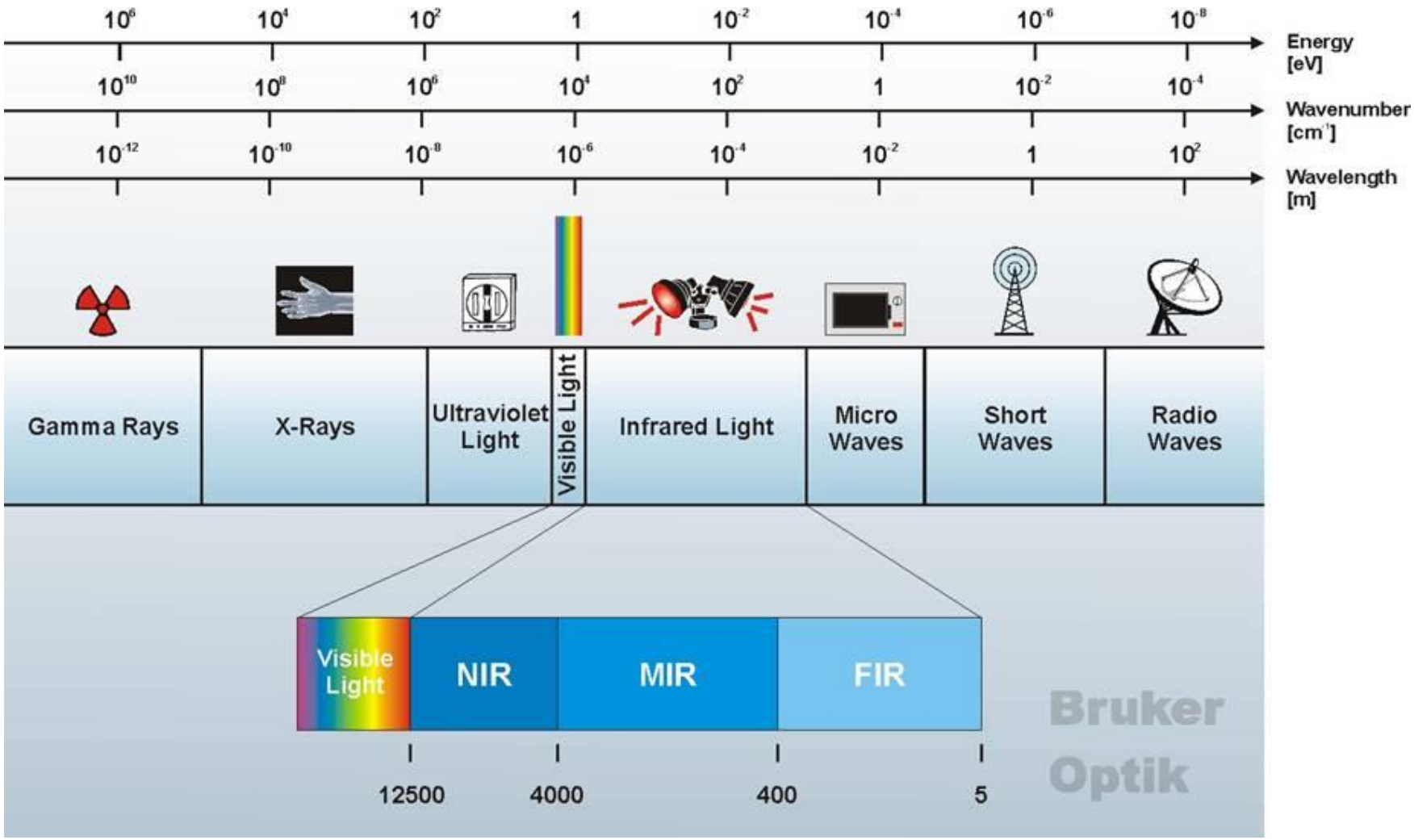
Penalty Points and Sanction Criteria

	Behaviors subject to penalty points	Penalty pts
	[Eligibility to use equipment]	
1	Unauthorized use of equipment without permission	5
2	Use of equipment without a reservation	3
3	Someone other than the equipment lessee used the equipment	3
	[Reservations for using equipment]	
4	Reserved and used equipment outside of permitted hours	1
5	Use of equipment beyond the time reserved without making another reservation beforehand for extra time	1
6	Failed to use the equipment durng the reserved time and did not cancel reservation in advance	3
7	Cancelling reservations for equipment after the cancellation deadline, under Article 7, Guideline for the Operation of the UNIST Central Research Facilities (UCRF)	1
8	Use of any equipment without giving a prior notice to the equipment manager, after making a reservation	1
	[Careless behaviors]	
9	Using functions on the equipment that are not permitted	3
10	Failure to promptly notify the manager of any errors or failures detected during use	3
11	Negligence that resulted in damages or failure to the equipment	5
12	Negligence that resulted in loss or damage to an equipment component or part	5
13	Failure to record in the equipment usage log after using any equipment, or misrepresentation or partial representation of the facts	1
14	Failure to provide specimen information required by the equipment manager to ensure normal operations and safety of equipment or facilities, thus resulting in damage or failure to the equipment	3
15	[Careless behaviors]	3
16	Using functions on the equipment that are not permitted	5
17	Failure to promptly notify the manager of any errors or failures detected during use	5

Penalty Points and Sanction Criteria

Classification	Penalty pts.	Follow-up actions
(Individual users of equipment)		
Sum up penalty points imposed to individuals	≥ 5 points	Equipment manager will notify user(s) and their supervising professor by email of their penalty points total, and shall post the details of their penalty points on the bulletin board of the equipment room. Users with penalty points 8 points or higher may not use the relevant equipment for 3 months.
	≥ 8 points	Equipment manager will notify user(s) and their supervising professor by email that the user(s) may not use the relevant equipment for 3 months until they complete the re-orientation course; will also forward an official notice to their supervising professor; and will post details of their penalty points on the bulletin board of the equipment room.
(User's laboratory)		
Sum up penalty points imposed on the students in the laboratory for the same equipment in the same laboratory	≥ 12 points	Equipment manager will notify the user(s) and their supervising professor by email that user(s) with penalty points 15 points or higher may not use the relevant equipment in the laboratory for 3 months.
	≥ 15 points	Equipment manager will email the supervising professor to inform that the user(s) may not use the relevant equipment in the laboratory for 3 months; will also forward an official notice to their supervising professor; and will post the details of their penalty points on the bulletin board of the equipment room.
Sum up penalty points imposed on the students in the laboratory for all UCRF equipment in the same laboratory	≥ 20 points	UCRF will notify students and their supervising professor by email that the user(s) with 25 penalty points or higher may not use any UCRF equipment in the laboratory for 1 month.
	≥ 25 points	UCRF will notify students and their supervising professor by email that user(s) may not use any UCRF equipment in the laboratory for 1 month; will also forward official notice to their supervising professor; and will post details of their penalty points on the bulletin board of UCRF.

Electromagnetic Radiation



Spectral ranges

NIR : 15000 – 4000 cm^{-1}

- Overtones and combination vibrations
- low absorption coefficient ⇔ high sample concentrations
- Advantage : Qualtz is transparent → fiber optics, in glass vials
- Source : tungsten lamp
- Optical material : Qualtz
- Detector : Ge, InGaAs

MIR : 4000 – 400 cm^{-1}

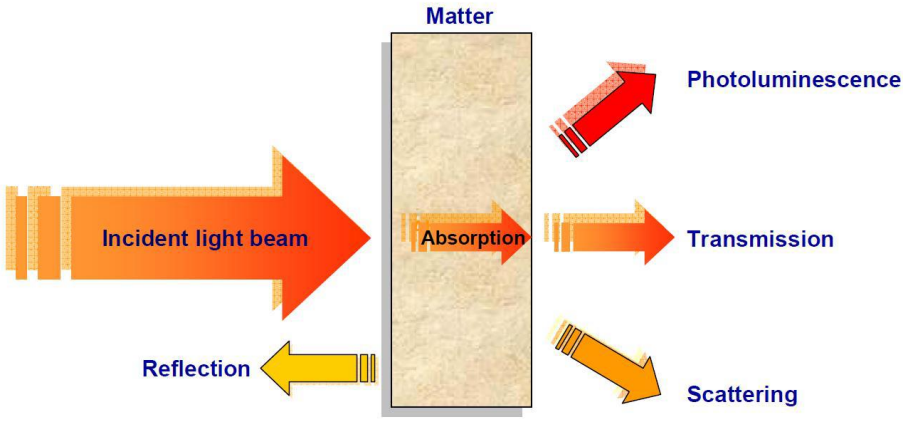
- Fundamental molecular vibrations : stretch and deformation vibrations
- high absorption coefficient ⇔ low sample concentrations
- Source : Globar
- Optical material : KBr, ZnSe
- Detector : DTGS, MCT

FIR : 400 – 5 cm^{-1}

- backbone vibration of large molecules, molecules with heavy atoms
- low absorption coefficient, strong water vapor absorption → vacuum spectrometer
- Source : Globar, Hg lamp
- Optical material : PE, CsI
- Detector : DTGS, Bolometer

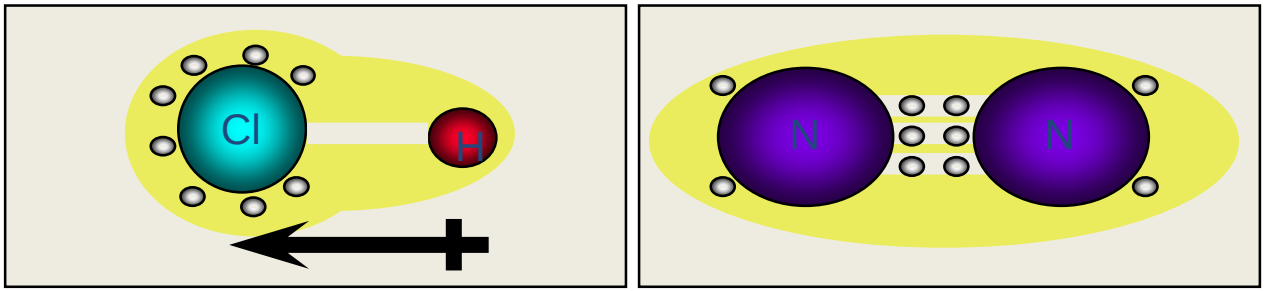
Vibration Theory

The absorption of Infra-red Light



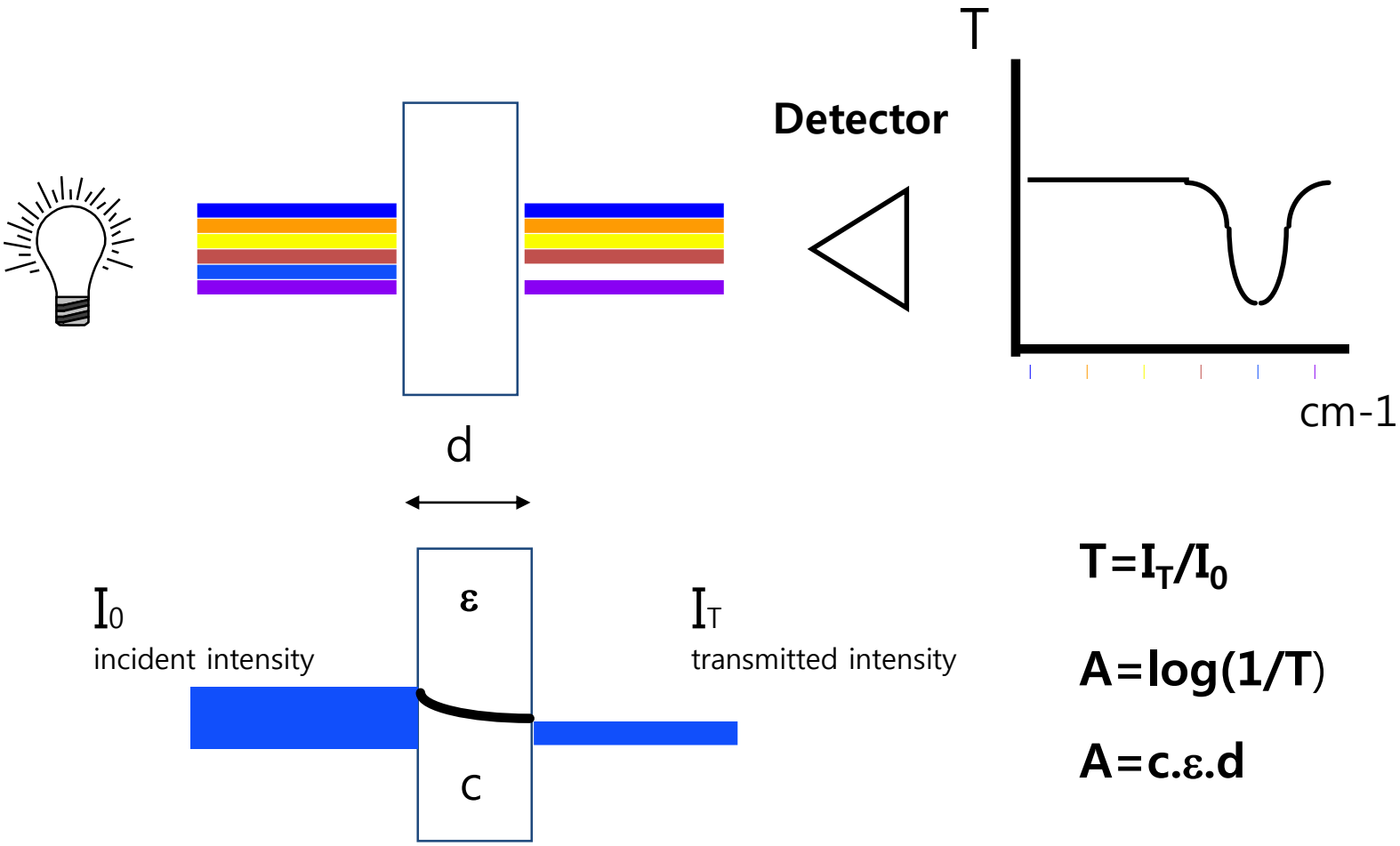
· Infrared light can only be absorbed by a molecule if the dipole moment of the specific group of atoms changes during the vibration.

·The greater the change in dipole moment, the stronger the corresponding IR absorption band will be.



Vibration Theory

Principles of Spectroscopy

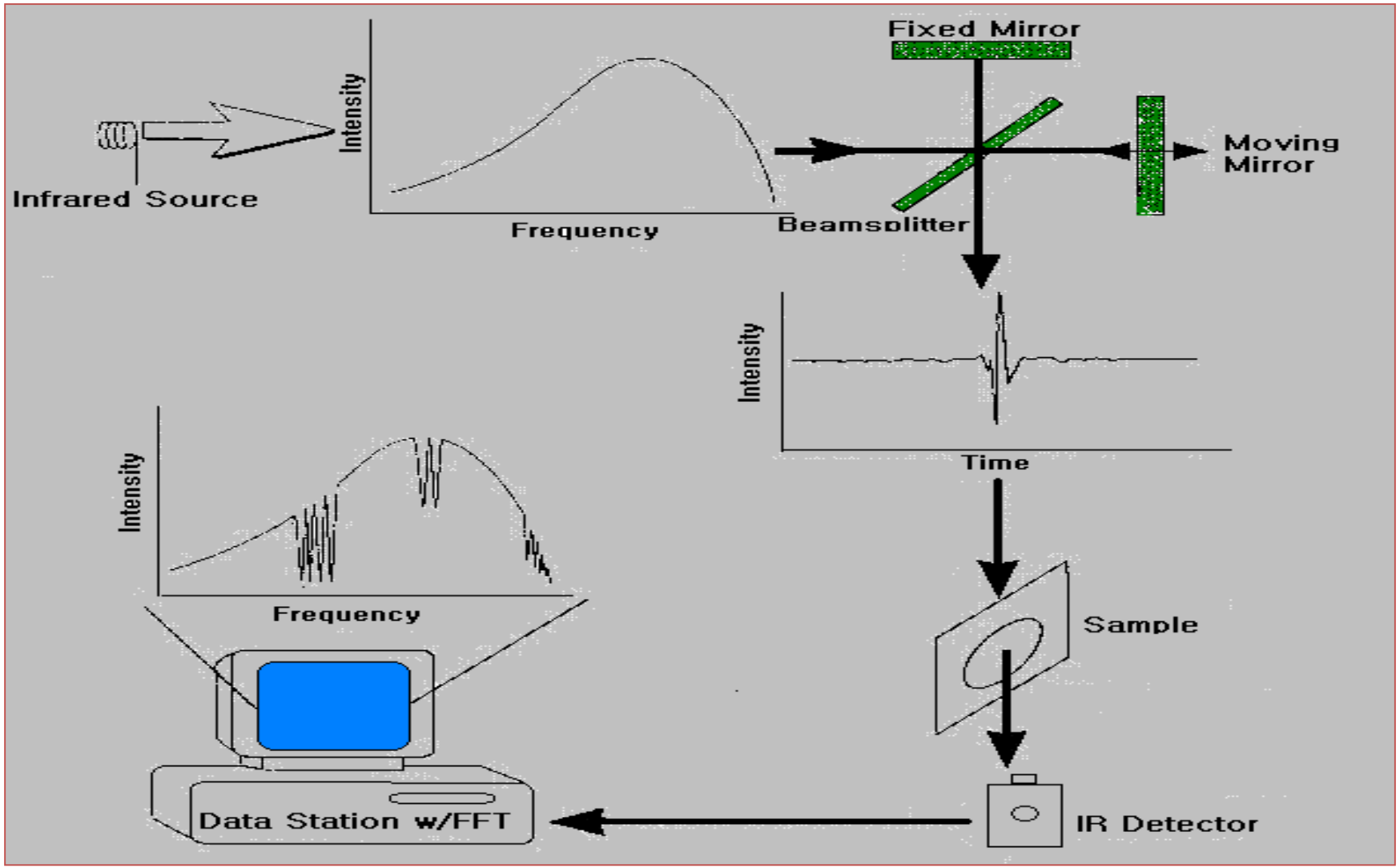


$$T = I_T / I_0$$

$$A = \log(1/T)$$

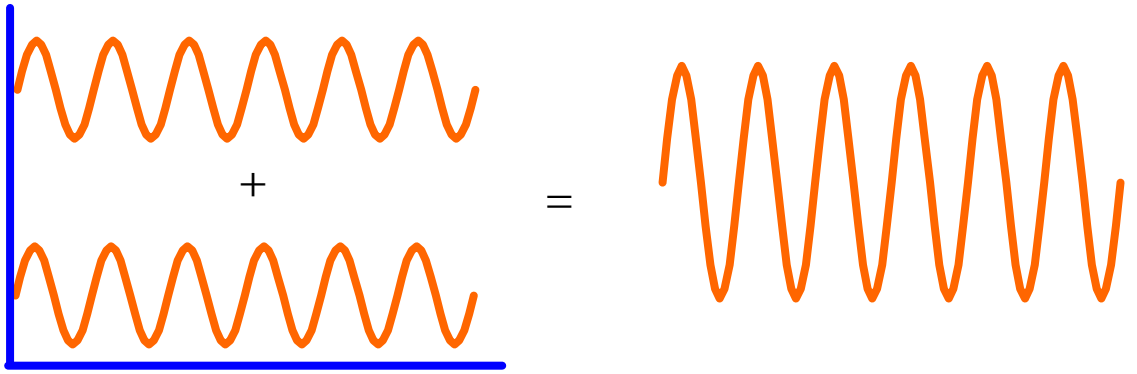
$$A = c.\varepsilon.d$$

Schematic of FT-IR

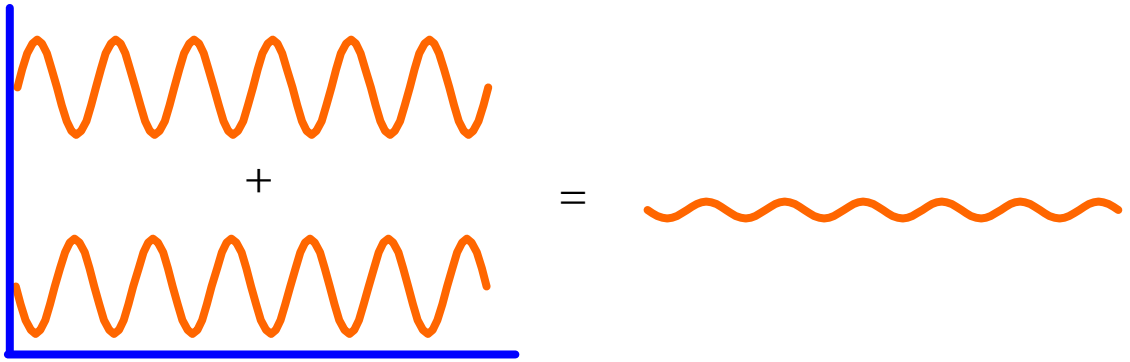


Wave Interaction (Interference)

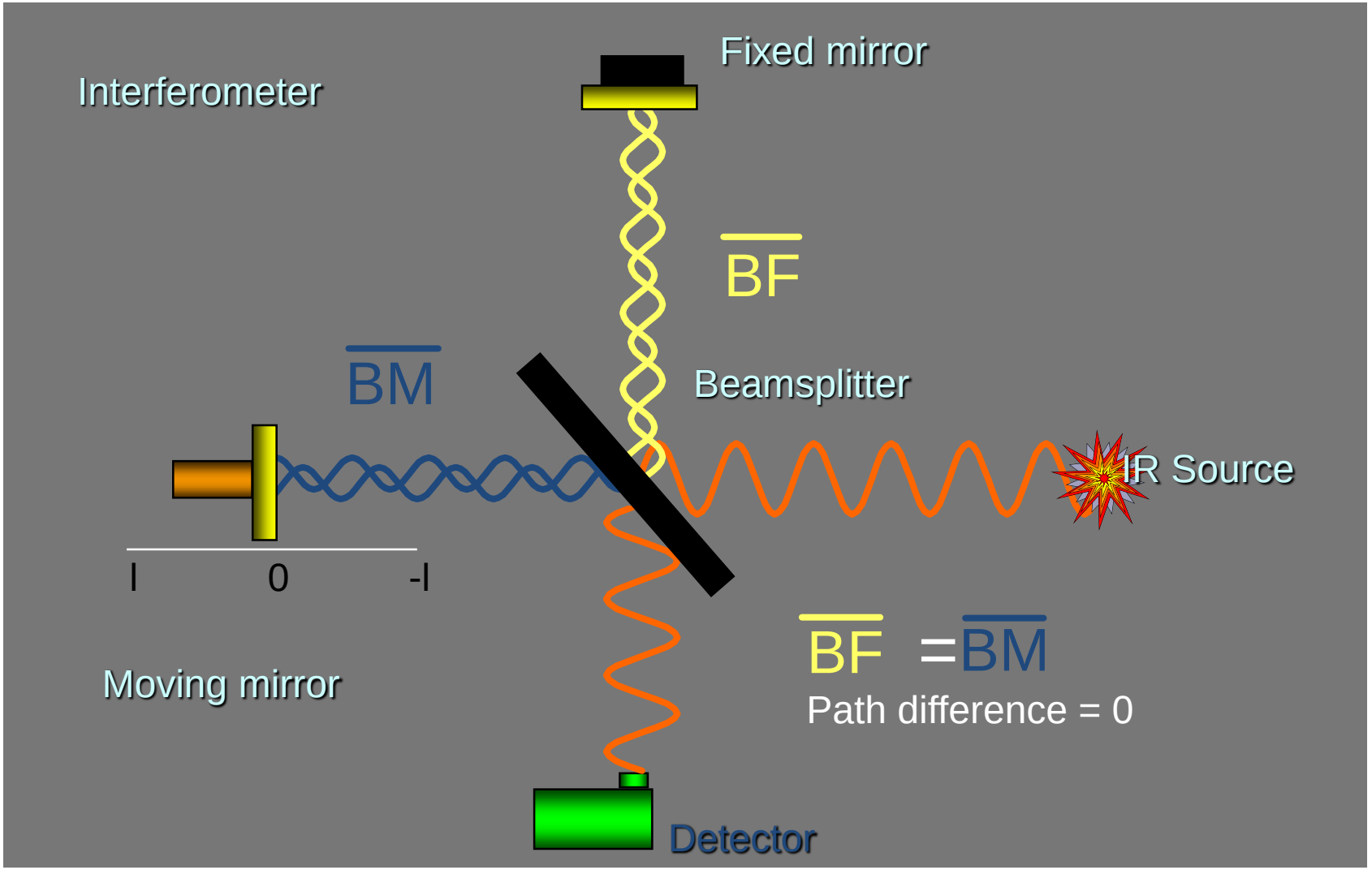
- In-phase
  - *Constructive interference*



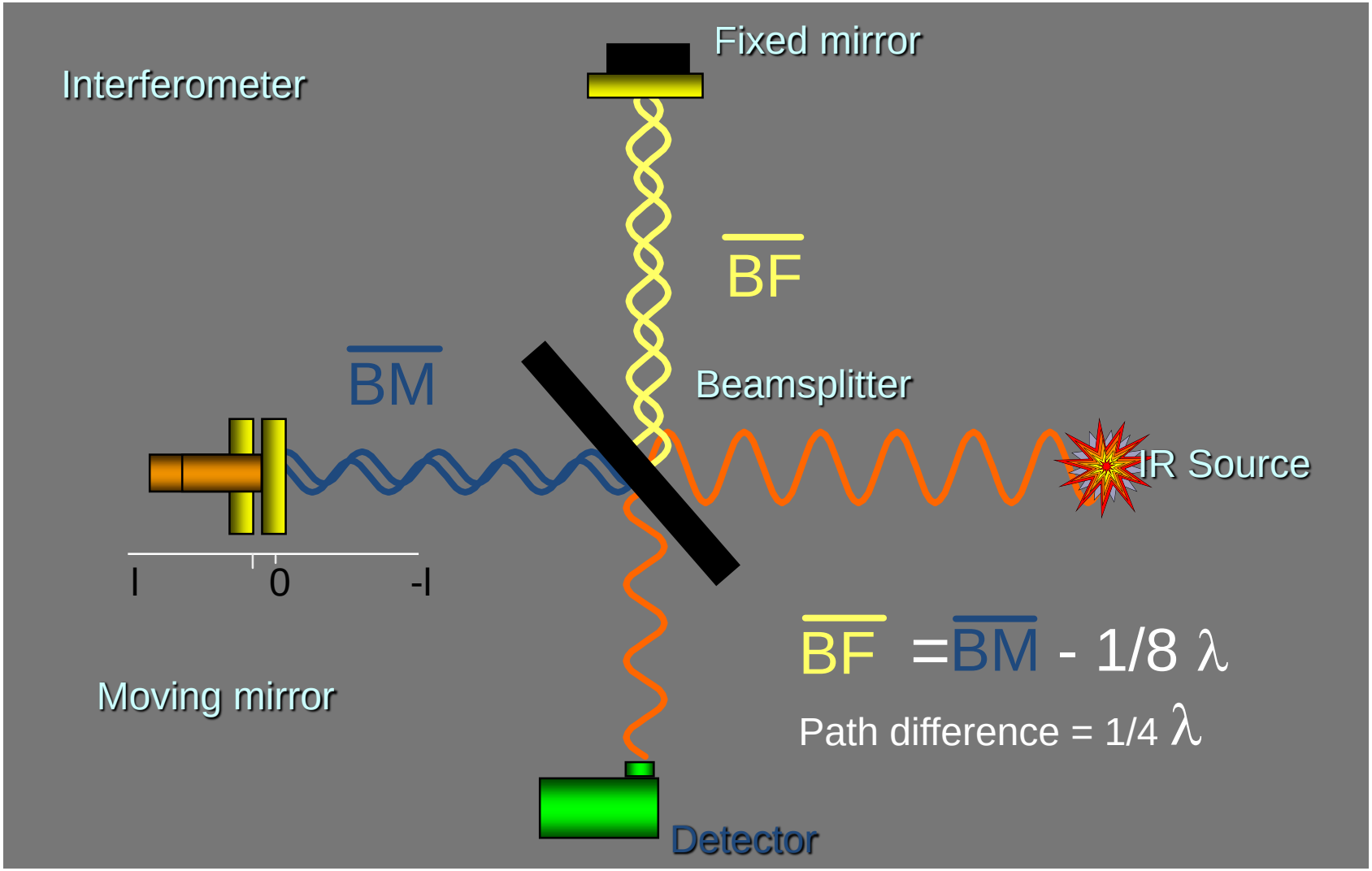
- Out-of-phase
  - *Destructive interference*



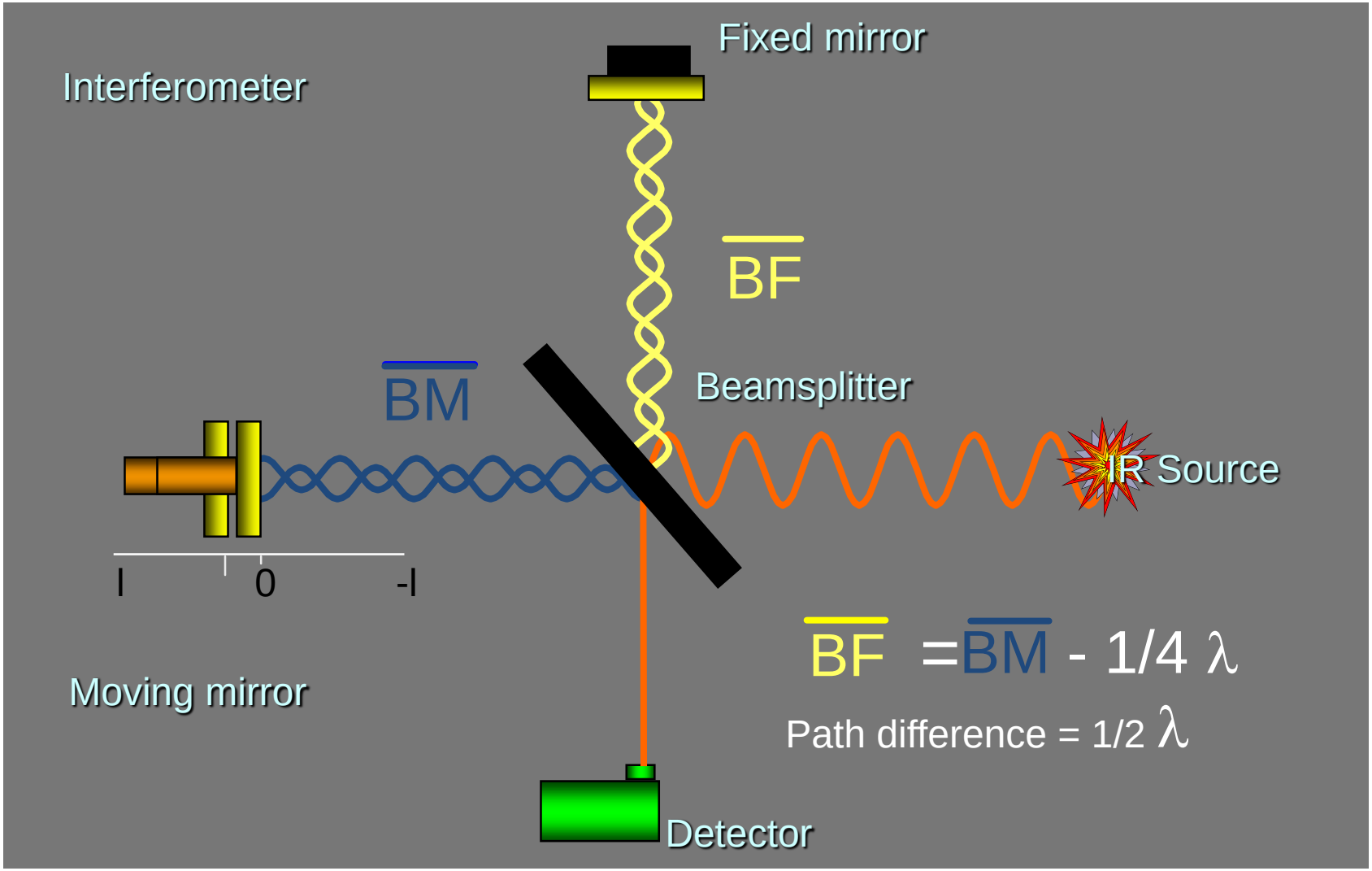
Michelson Interferometer



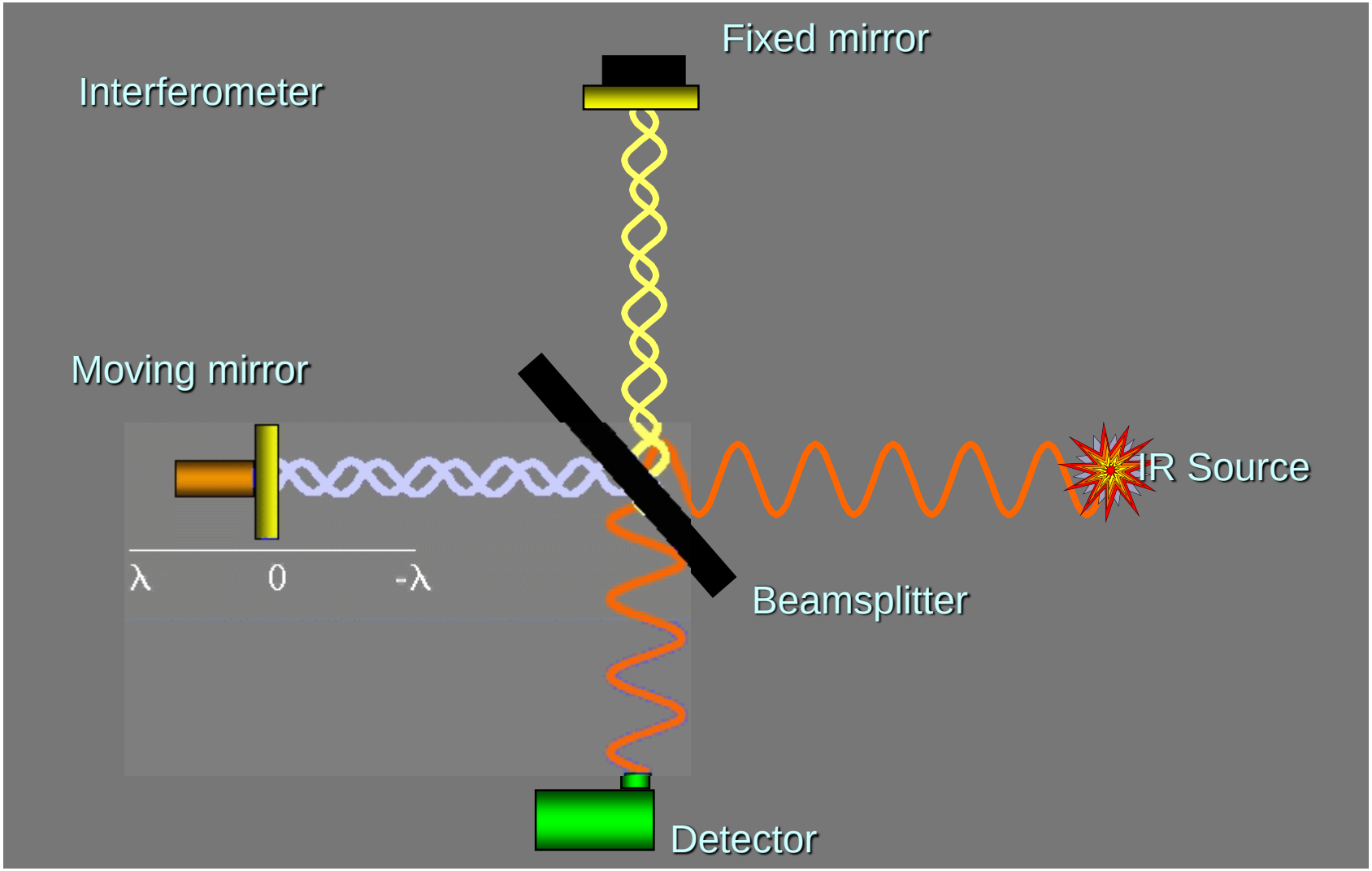
Michelson Interferometer



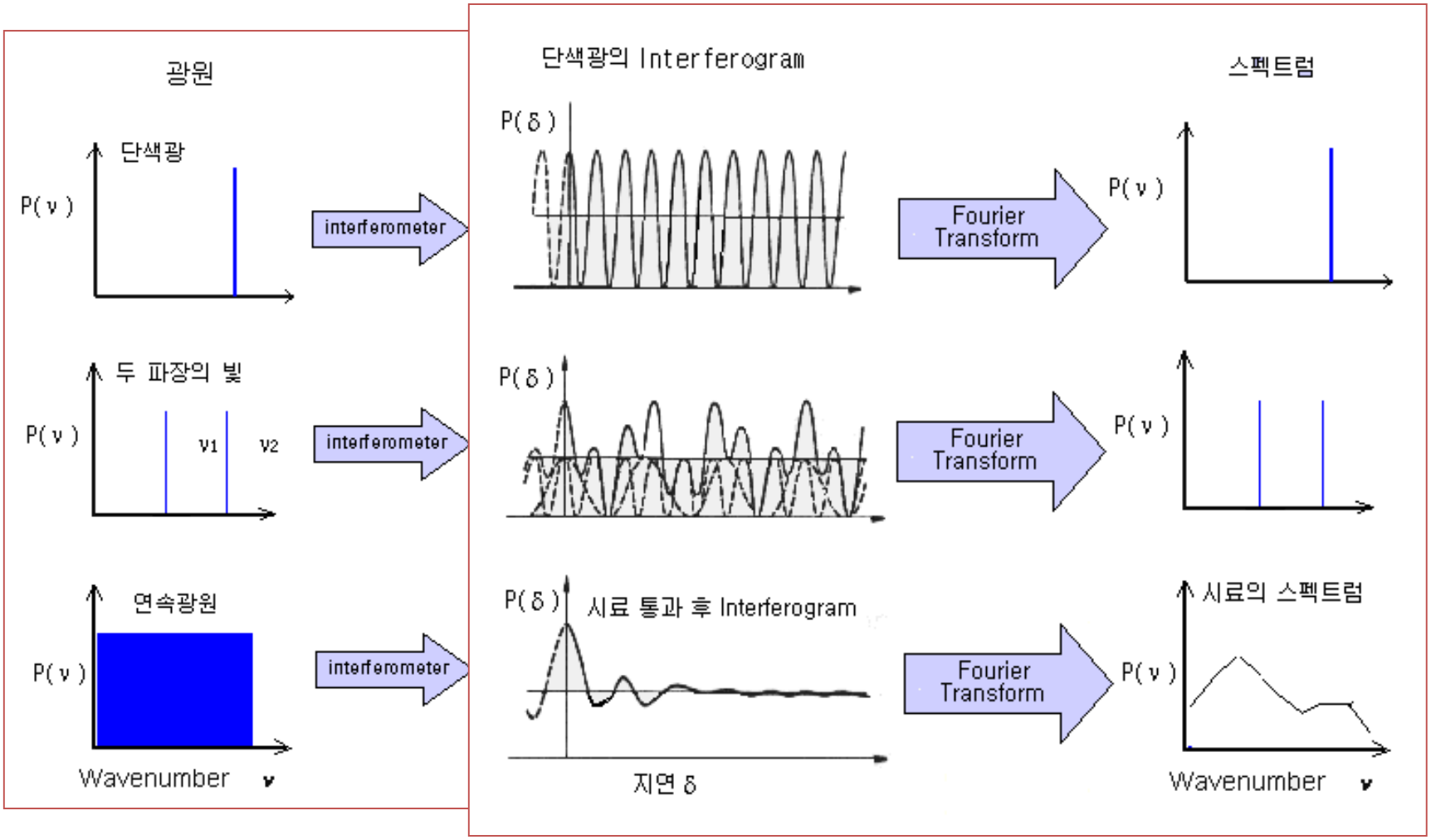
Michelson Interferometer



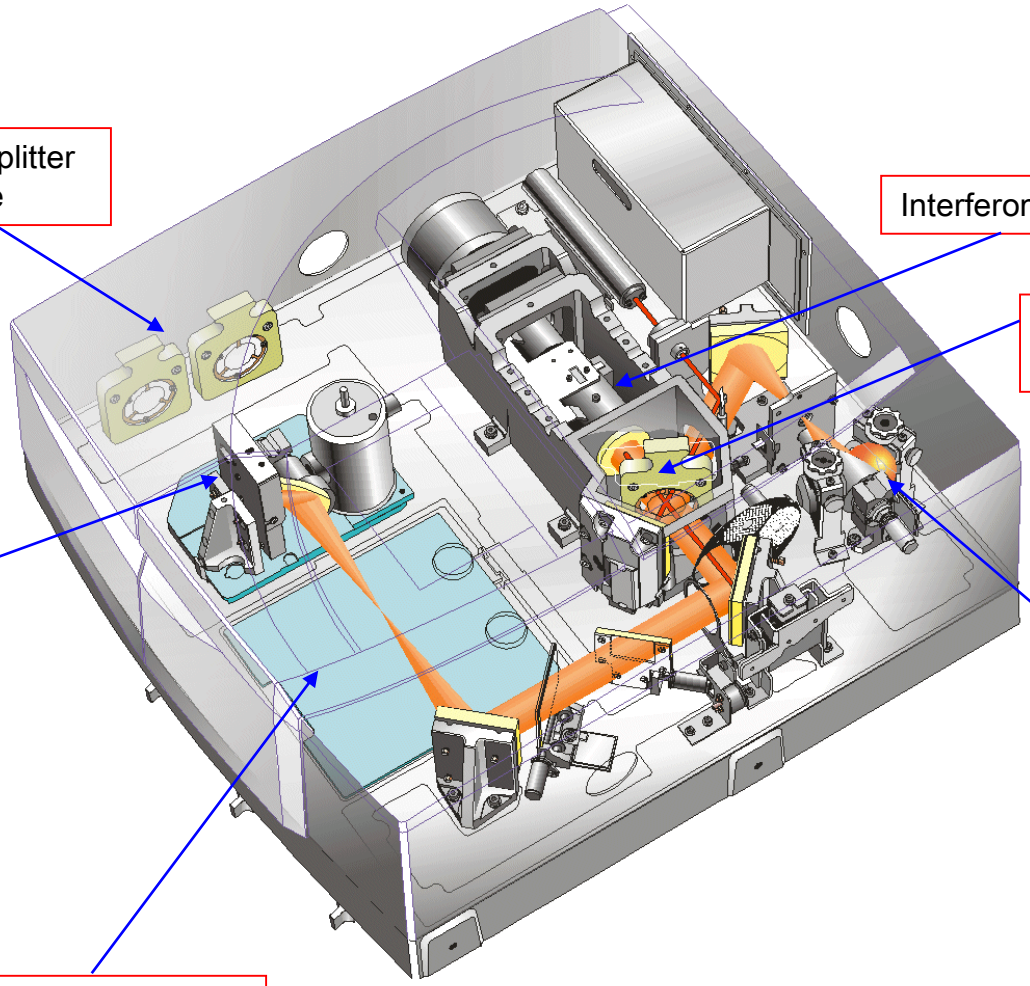
Michelson Interferometer



Conversion to Spectrum



670 FT-IR Systems



The Type of Samples

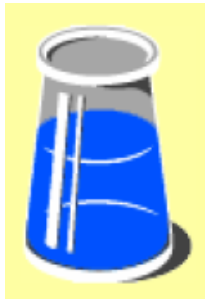
Powder

Coatings

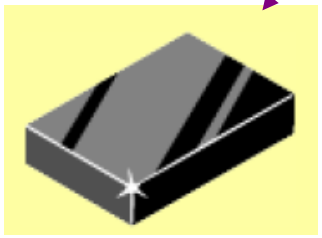
Fiber and Beads

Paints

The Type of Samples



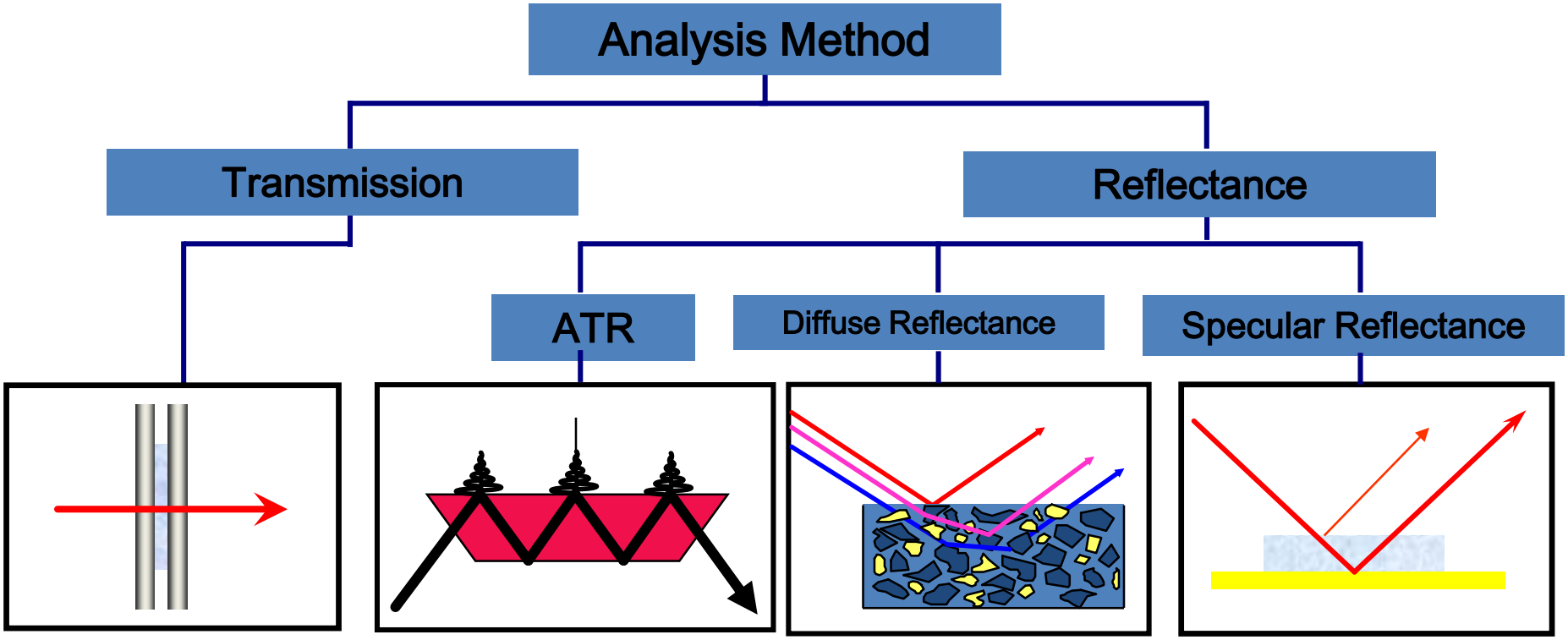
Liquid



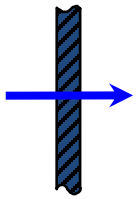
Films

Gas

Sampling Techniques



Sampling Techniques

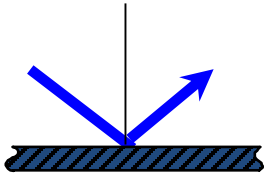


$I^o = 0^o$

Transmission

(Powder, Drugs-Kbr pellt/Films, Coatings, Paints-film holder/Liquid-window cell)

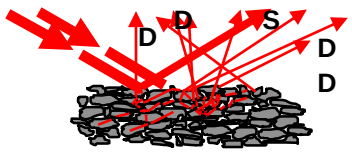
- Absolute reference measurement
- Sample preparation can be difficult and time consuming



$I^o = R^o$

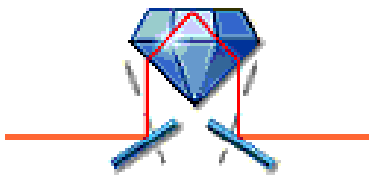
Specular Reflectance

- Sample must have a back reflective layer or must be on a mirror
- (layer thickness = single molecule)



Diffuse Reflectance

- Solids and powders, diluted in a matrix of KBr or KCl
- Analysis of non-reflective materials



ATR (Attenuated Total Reflectance)

(Powder, Drugs, Films, Coatings, Paints, Liquid, Rubber)

- The infrared beam is focused into a crystal
- Creating an evanescent wave about 1 – 2 microns deep
- No sample preparation

Sampling Accessories – Transmission (Powder/Film)

Transmission by KBr Pellet : Powder, Drugs, Film

1. Grind sample together with the KBr for several minutes until fully mixed using an agate mortar and pestle.

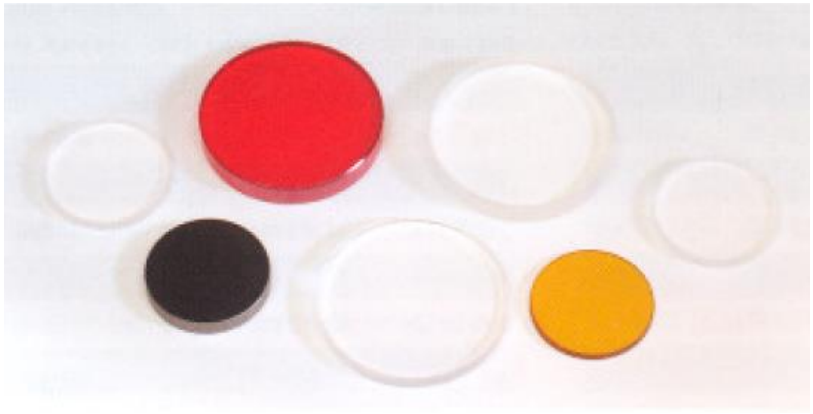
2. Assemble the Die set and add the mixture into the die. Put the die into the press.
(8 ton, Hold time 1min. Release time 1min.)

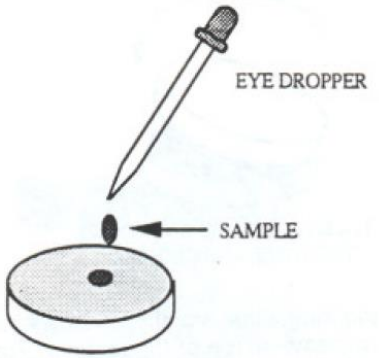
- Mounting a Pellet Sample

Sampling Accessories – Transmission (Liquid)

Transmission using window materials (Liquid sample)

Step 1. Choice the window material





Step 2. Place a small amount of sample onto the window using the eye dropper or spatula.

Step 3. Once enough sample is deposited on the window, place the other infrared window on top of the sample.

Step 4. Twist the windows together in opposite directions to get rid of air bubbles and to decrease the pathlength.

Sampling Accessories – Transmission (Liquid)

Transmission using window materials (Liquid sample)

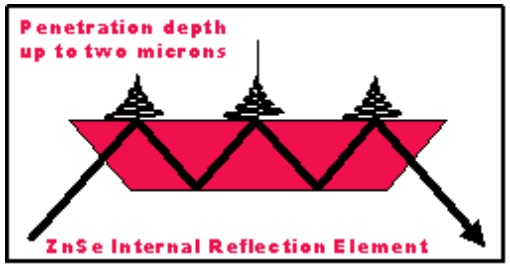
Material	Water Sol. (g/100g H_2O) @25 °C	Max. Useful Temp. In Air (°C)	Density g/cm³	pH Range	Cleaning Agents	Solvents Which Attack Material	Hardness (Knoop#)
AgCl	0.00015	200	6.47	N/A	acetone, CH_2Cl_2	complexing agents*	9.5
AL_2O_3 (Sapphire)	insol.	1700	4.00	1 – 14	alcohol, acetone, H_2O	acids, alkalies	1370
AMTIR	insol.	300	4.40	1 – 9	alcohol, acetone, H_2O	alkalies	170
BaF_2	0.17	500	4.83	5 – 8	acetone, alcohol	NH_4 + salts, acids	82
CaF_2	0.0013	900	3.18	1 – 9	acetone, alcohol	NH_4 + salts, acids	158
CdTe	insol.	300	6.2	1 – 9	alcohol, acetone	acids, HNO_3	56
CsI	44.4	200	4.50	N/A	anhydrous solvents	lower alcohols "wet" solvents	20
Diamond	insol.	750	3.51	1 – 14	alcohol, acetone	$K_2Cr_2O_7$, conc. H_2SO_4	7000
Ge	insol.	270	5.32	1 – 14	alcohol, acetone, H_2O	H_2SO_4, aqua regia	550
KBr	53.5	300	2.75	N/A	anhydrous solvents	lower alcohols "wet" solvents	7
KRS-5	0.05	200	7.37	5 – 8	MEK	complexing agents*	40
NaCl	35.7	400	2.17	N/A	anhydrous solvents	lower alcohols "wet" solvents	15
Si	insol.	300	2.33	1 – 12	alcohol, acetone, H_2O	HF, HNO_3	1150
SiO_2 (Quartz)	insol.	1200	2.65	1 – 14	alcohol, acetone, H_2O	HF, some hot acids and bases	820
ZnS	0.00069	300	4.08	5 – 12	acetone, alcohol	acids	178
ZnSe	insol.	300	5.27	5 – 9	alcohol, acetone, H_2O	acids, strong alkalies	137

*typical complexing agents include ammonium salts & materials such as EDTA.

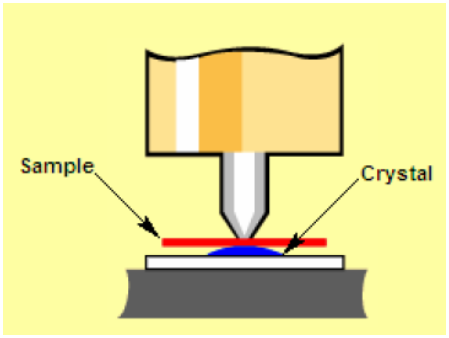
Sampling Accessories - ATR

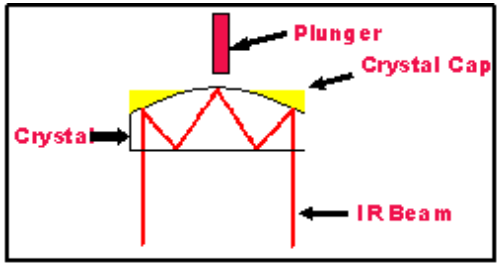
ATR Accessories

- Main Frame
- Crystal
  (ZnSe, Diamond, Ge, Si)
- High Pressure Clamp



Multi-bounce ATR





Single-bounce ATR

Sampling Accessories - ATR

Material	ATR Spectral Range (cm^{-1})	Refractive Index	Depth of Penetration (μ) (at 45º & 1000 cm^{-1})	Uses
Germanium	5,500 - 675	4	0.66	Good for most samples, especially strong absorbing samples, such as dark polymers
Silicon	8,900 - 1,500 & 360-120	3.4	0.85	Resistant to basic solutions
AMTIR	11,000 - 725	2.5	1.77	Very resistant to acidic solutions
ZnSe	15,000 - 650	2.4	2.01	General use
Diamond	25,000 - 100	2.4	2.01	Good for most samples. Extremely caustic or hard samples

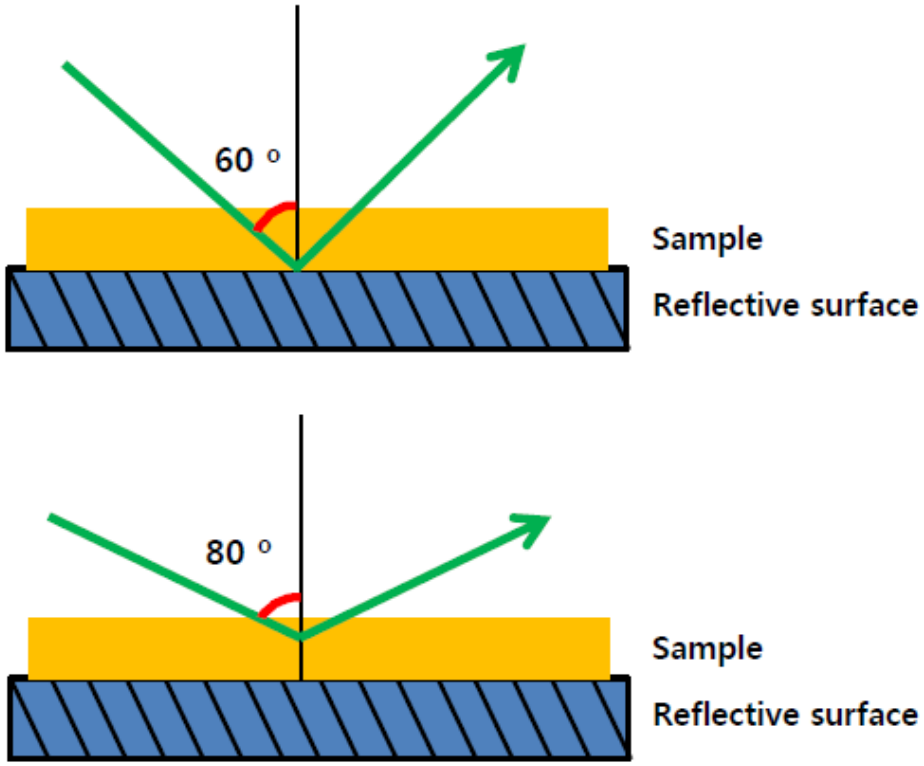
Sampling Accessories – Specular Reflectance

Specular Reflectance

Thin films on reflective surfaces : Au, Ag, Al great

Polarization dependent : Polarized light improves s/n of results

Grazing angle improves interaction with sample

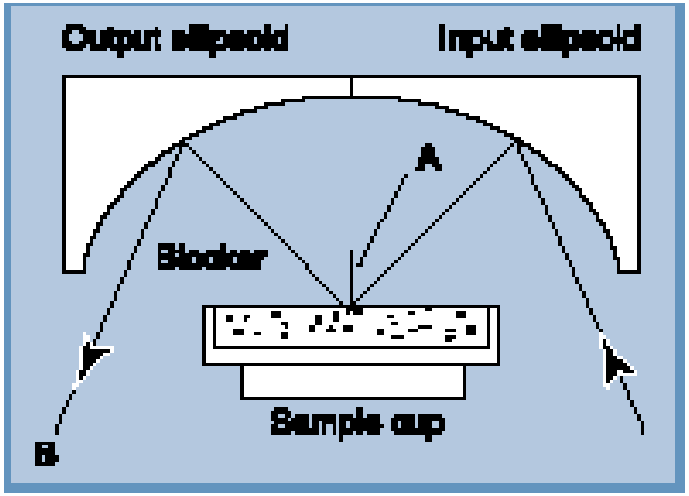


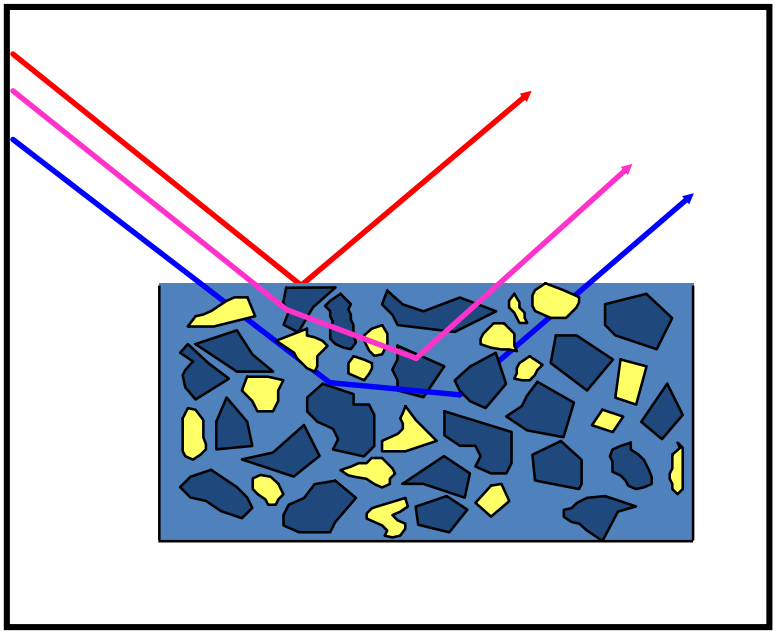
Seagull

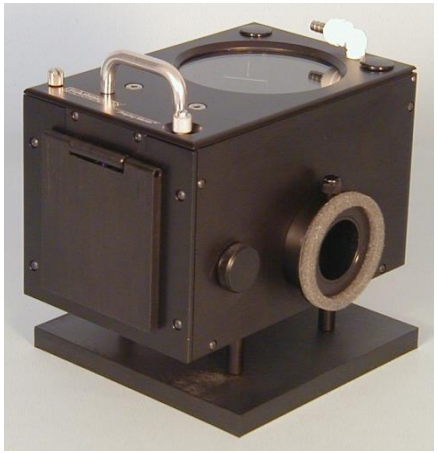
Sampling Accessories – DRIFTS

Diffuse Reflectance (DRIFTS)

Preferred choice for dilute powders
Analysis of non-reflective materials
Minimal sample preparation





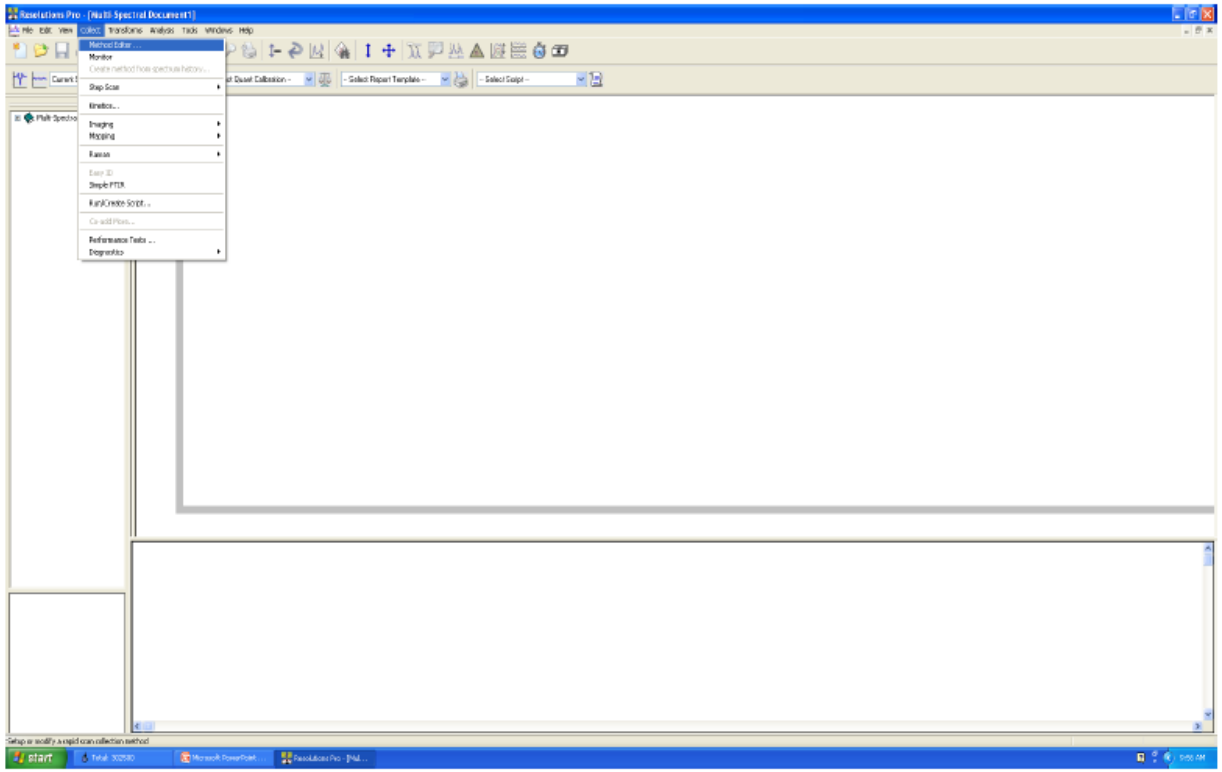


Praying Mantis

Manual – ATR mode

1. Agilent Resolution Pro. STATR

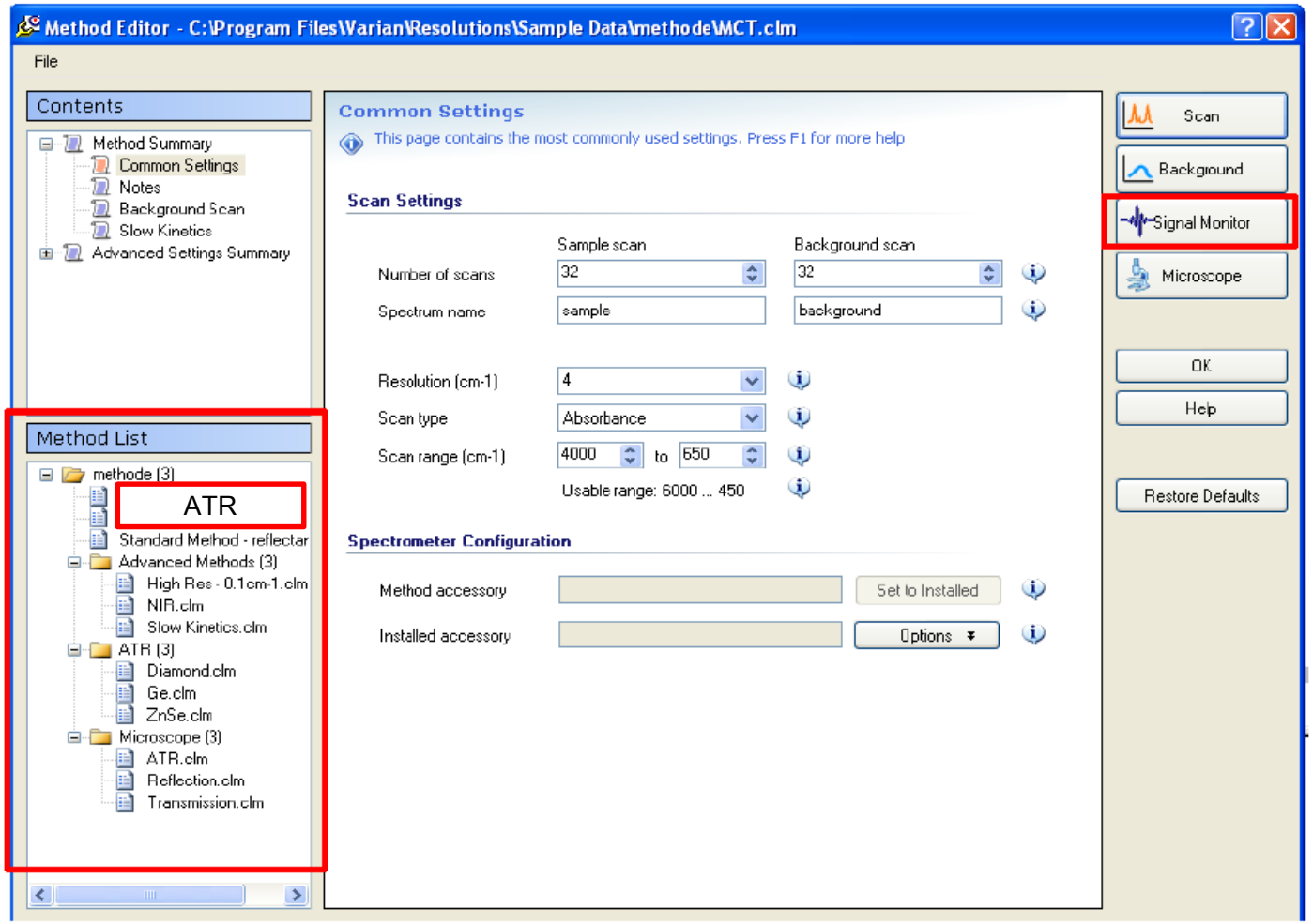
2. Collect ⇒ Method Editor



Manual – ATR mode

3. Method List ⇒ select method_ATR

4. **Signal Monitor** click

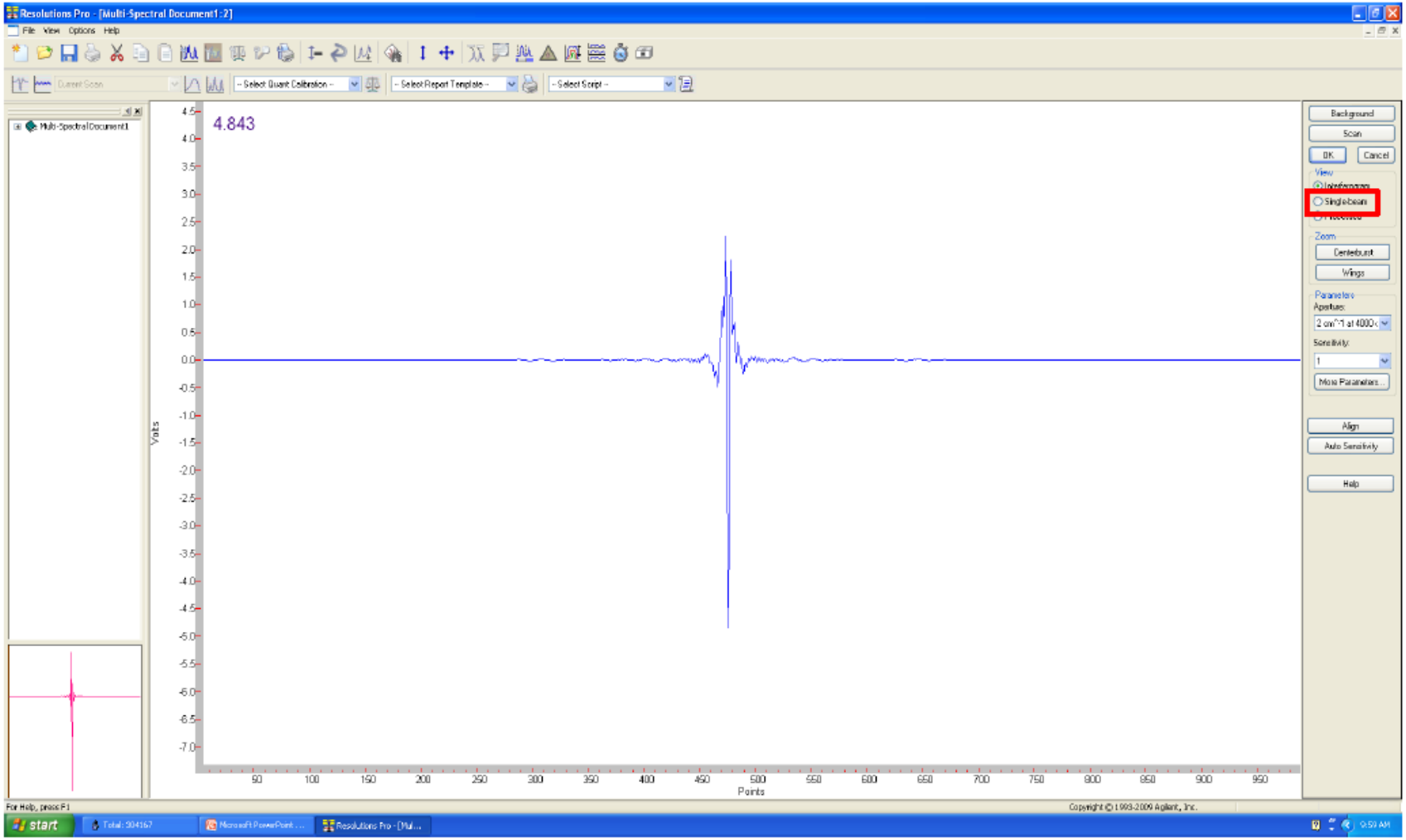


4

Manual – ATR mode

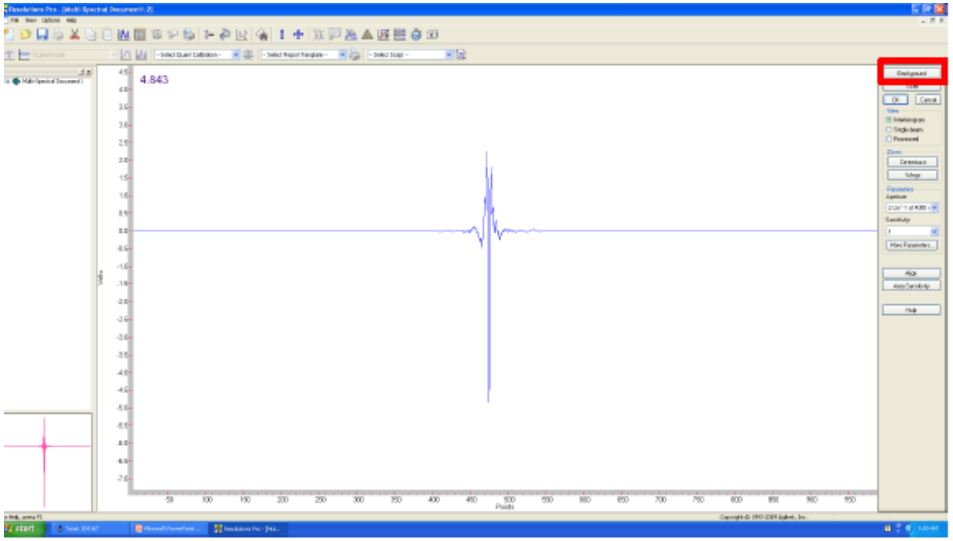
5. Check the **Inteferogram**

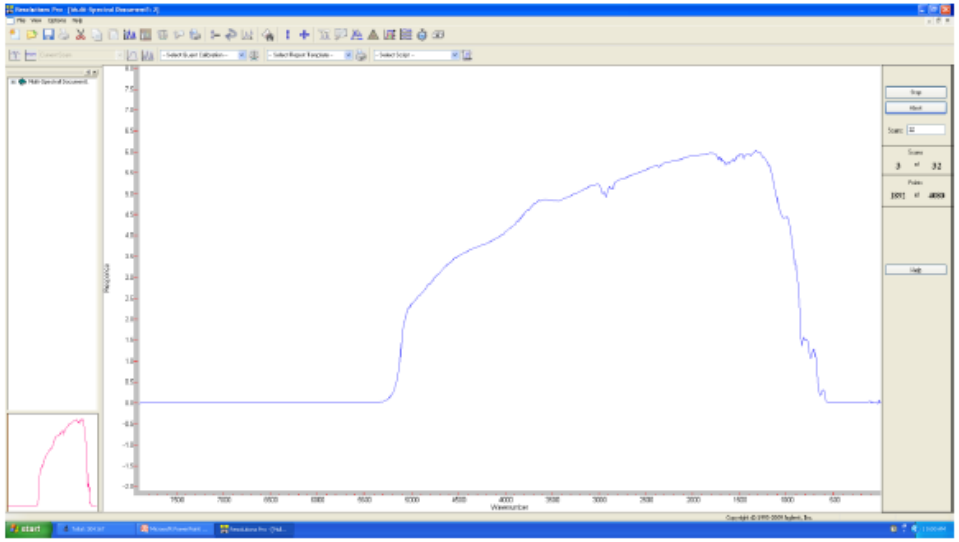
6. Click **Singlebeam** – Check background condition



Manual – ATR mode

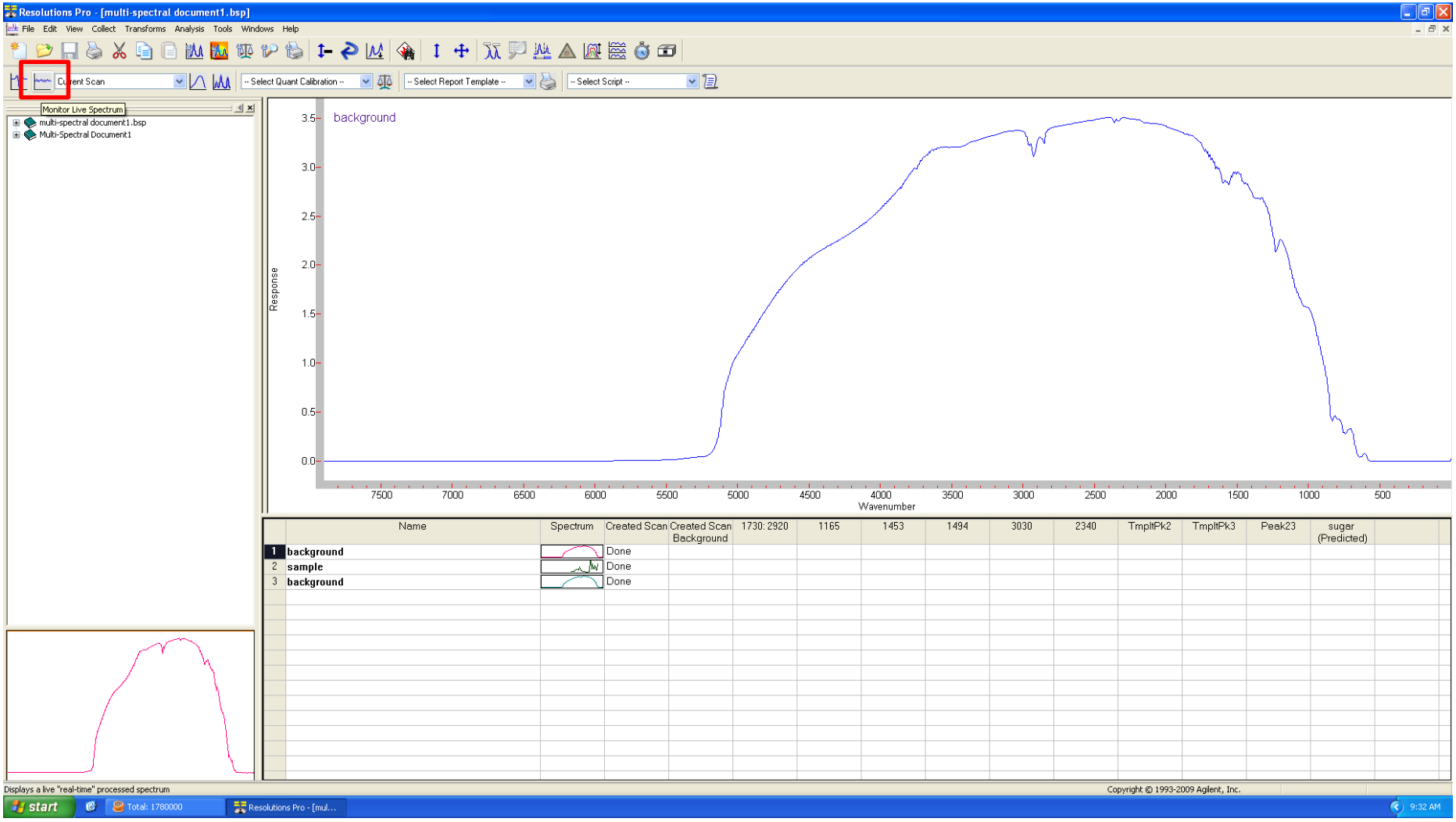
7. Click **Background** ⇒ Save .bsp file





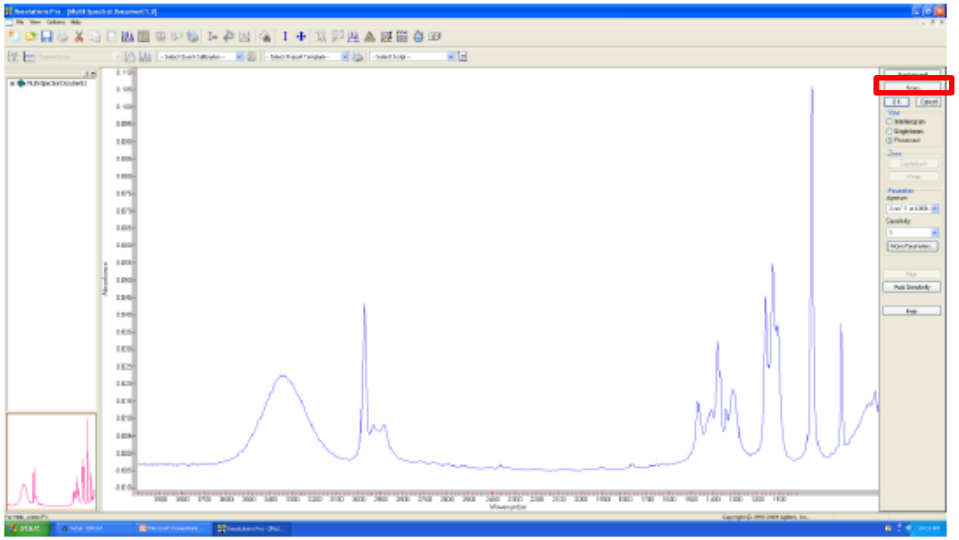
Manual – ATR mode

8. Sample loading ⇒ Monitor live spectrum click

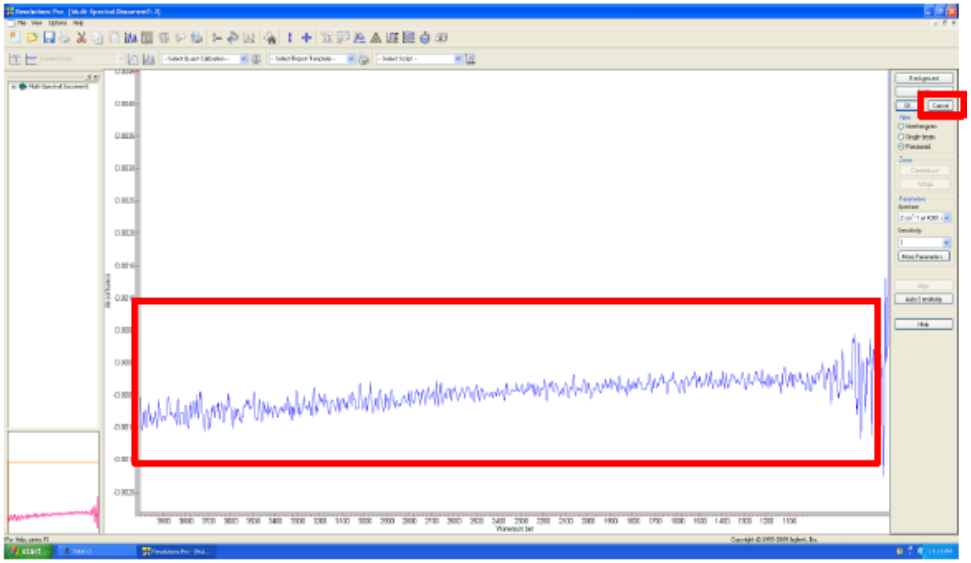


Manual – ATR mode

9. Scan click

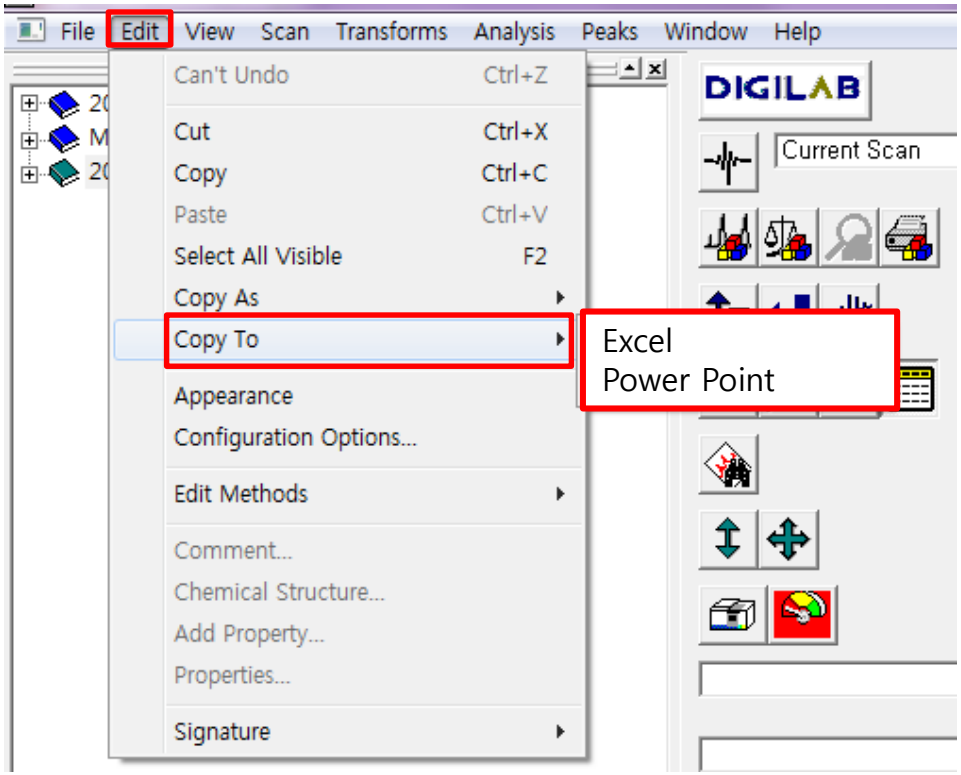


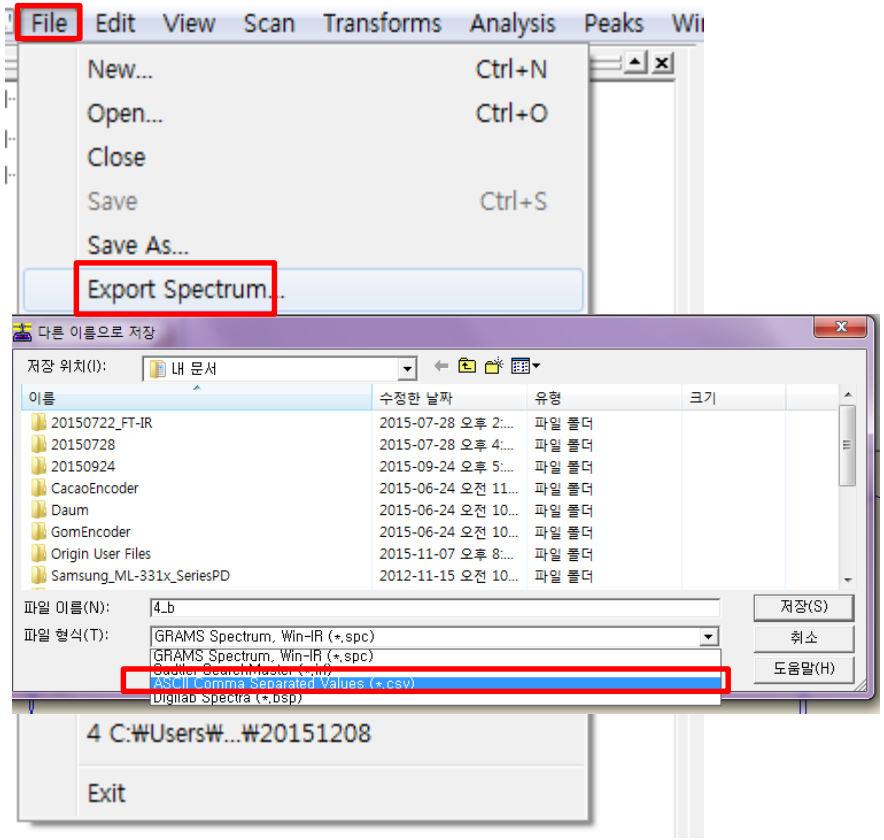
10. ATR cleaning ⇒ Monitor live spectrum ⇒ If there is no peak ⇒ Cancel click



Manual – ATR mode

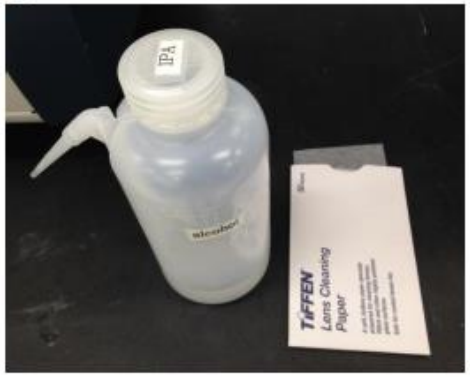
11. Export spectrum
    1) Select data – Edit – Copy to
    2) Select data - File – Export Spectrum – .csv file



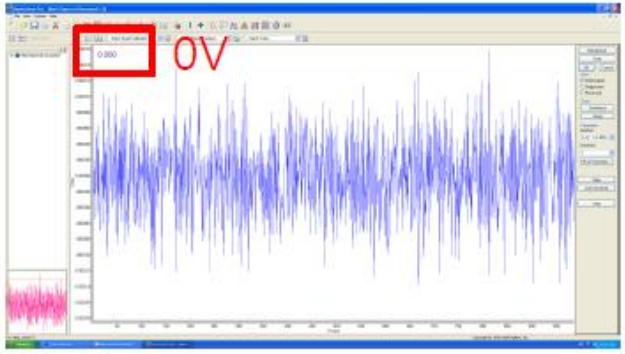


Cautions

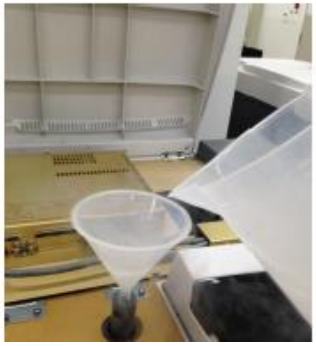
1. ATR crystal cleansing



2. Fill up the MTC detector with liquid nitrogen



After close program, fill up!



Charge

		Main body		Microscope
		ATR	ACC	
UNIST students	Client(70%)	21,000/hr	31,500/hr	42,000/hr
	Self-user(50%)	15,000/hr	22,500/hr	30,000/hr

True Café using time	Charging time
0.1~0.5 hr	0.5 hr
0.6~1.0 hr	1.0 hr
1.1~1.5 hr	1.5 hr
1.6~2.0 hr	2.0 hr
…	…