

Standard Operating Procedures

Microscopy and Spectroscopy Lab

Department of Physics

COMSATS Institute of Information Technology Lahore

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1. Introduction of the Laboratory:

The Microscopy & Spectroscopy lab was established in 2015 in Department of Physics by utilizing the budget from approved Mega PC-1 project granted to CIIT Lahore through Islamic Development Bank (IDB) funding .The lab holds three major High Tech. equipments meant for characterization and testing of the samples prepared for scientific research and commercial purposes which are listed below:

1. ATOMIC FORCE MICRSCOPY (AFM), Model-XE7, PARK SYESTEMS, Korea
(Functional since September 2015)
2. IN-Via Raman Microscope (Raman &PL Setup, RENISHAW,UK)
(Functional since January 2017)
3. UV-Vis NIR Spectrometer (Lambda -750,PERKIN ELMER USA)
(Functional since April 2017)

The lab has been purposefully built to avoid a dust and humidity free environment. All the three equipments are placed in individual sections separated partially with high quality glass walls to avoid distortion and acoustic vibrations. A temperature of 22⁰ Celsius is maintained in the laboratory as a recommended condition for the functioning of equipments. All the equipments are supplied with UPS as per recommended specifications of the individual equipment. All the three equipments are necessarily provided with a software interface through an up to date PC and related hardware accessories.

2. Laboratory Code of Practice:

2.1 Personal Protection

- Appropriate footwear (i.e. enclosed shoes - no thongs or open sandals) must be worn at all times in the center.
- Appropriate personal protective equipment (lab coat, masks, and gloves) must be worn during handling.
- Protective glasses, lab coats and gloves are available in the labs.
- Use appropriate gloves for chemicals.
- Disposable masks are available.
- Discard gloves on slight contamination.

2.2 Housekeeping Rules

- No food or drink is to be consumed near any instruments.
- All areas are to be kept neat and tidy after use.
- A weekly lab cleaning Rota is to be maintained to ensure “effective” cleanliness and safety.
- All laboratory doors should remain closed, except when being used for entrance and egress.
- Please notify staff if supplies (gloves, face masks) have run low or waste disposal bins are full.
- Excessive use of mobile phones is not allowed in this area.
- Make sure you take your samples with you when you leave.
- All areas are to be kept neat and tidy after use.

2.3 Chemical Protection

- Make sure that you have done the risk assessments of all the chemicals/samples prior to working in the lab.
- Be aware of handling, spill and disposal procedures for all chemicals you use in the center.
- In case of any spill, clean immediately.
- Do not leave your samples unattended or unlabeled.
- Any unidentified samples or chemicals will be disposed of.

3. General Laboratory Procedures:

- As a user of this lab, you are required to comply by the standard operating procedures of the lab for its proper functioning. This includes the standard methods of putting the requests for sample scanning (Annex I) and general health and safety maintenance (Annex II).
- Each equipment in the lab has to be booked before your actual desired time of its operation. A booking log will be maintained as per que of the submitted samples.
- To get your samples scanned/characterized you are required to fill in the COSHH Form (Available in Annex I,II, III) and submit at least a week before your required booking slot.
- You are required to submit your samples along with the COSHH form and the suitable slot for scanning will be ssined according to the que of the samples which are in scanning process in the lab.
- The sample scanning for the atomic force microscope would be carried out by the certified/designated lab officials. This is IMPORTANT for the smooth running and functioning of this privileged facility.
- The scanned data can be collected only in DVD/CD which must be virus free/formatted which you must bring at the time of collection.
- The operation of the microscope requires certain consumables which include cantilever tips for sample scanning. Hence, a small amount of charge is being suggested in lieu of consumables fund for external users (Details in Annex III) whereas the lab does not charge the internal users for

4. Equipment wise SOPs /Risk Assessments

4.1 Atomic Force Microscopy (XE7 PARK SYSTEM, KOREA):

XE7 Atomic force Microscope (AFM), PARK SYSTEMS Korea is so far the only equipment available in Pakistan and owned by Department of Physics CIIT Lahore since 2015. It is a convenient to use equipment provided with high quality nanoscale resolution scanning of the top surface of the samples prepared in the form of a film or pellet of area around 5mm². PARK –XE7 AFM is currently operational for:

Standard imaging

Topography

Phase imaging

Force-distance spectroscopy

Using the following modes:

1. Contact AFM
2. Lateral Force Microscopy (LFM)
3. Non-contact AFM
4. Dynamic Force Microscopy (DFM/tapping mode)

Sample Types applicable for scanning:

1. Polymers
2. Semiconductors
3. Metallic
4. Nanocomposites
5. Biomedical materials
6. Ceramics
7. nanoparticles

All materials should be provided in the form of films or pellets.

4.1.1 RISK Assessment & Controls (AFM-PARK XE7)

Tasks	Hazards	Associated risks	Risk Controls
Use of Atomic Force microscope	Electrical equipment (microscopes, scanners, cameras and computers)	Possible electric shock	Regular checks of power cords for fault, fraying or wear and regular electrical safety checks. Maintenance only performed by authorized microscopy staff and microscope vendors.
	Ergonomics	Postural damage from extended periods of time working at microscope and computer	Maintain good posture at all times whilst working at the microscopes and computers. Do not work at the microscope /computer for more than two hours without taking a break.
Tasks	Hazards	Associated risks	Risk Controls
	Glass slides and coverslips	Cuts from broken glass	Carry slides/coverslips in suitable container. Dispose of any broken slides/coverslips immediately in Sharps containers provided. Dispose of any used slides/coverslips correctly (Sharps containers).
	Experiment specific chemicals and biological materials	Possible exposure to toxic or hazardous chemicals or biological materials	Specimens brought into the facility must be clean, dry, safe and enclosed/contained (eg mounted slide). Samples must be transported correctly. No hazardous chemicals will be allowed in the microscopy area.
Use of microscopes with lasers	Lasers	Eye damage from lasers (UV and visible lasers)	Do not look directly at light source. Maintenance only to be performed by authorized personnel (Lab Incharge and Park Instruments staff)

Use of Microscopy area	Room temperature and humidity	Fluctuations in room temperature and humidity	Problem currently being monitored and dehumidifiers.
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4.2 In Via Raman Microscope (Renishaw)

InVia Raman (Reflex) confocal Microscope is used for both Raman and photoluminescence measurements to obtain information on the electronic and vibrational structure of the materials. It is operational since January 2017, It performs Raman and PL measurements with following excitation wavelengths and specifications :

- Excitation Wavelengths- 457 nm, 488 nm, 514 nm
- Gratings: 1800, 2400
- Lens – 5x, 20x, 50x, 100x

4.2.1 Raman Spectroscopy Standard operating procedures for smooth and optimum System performance.

1. The room housing the Raman Spectroscopy unit should be maintained clean & dust free.
2. Shoes are not allowed inside Raman room.
3. Eating is not allowed inside the Raman room.
4. Only trained authorized person is recommended to operate the machine.
5. No one should stare at the laser beam without laser safety goggles as it is hazardous for eyes.
6. One should not lean or rest on the detector, podule or microscope binocular eye pieces.
7. Memory sticks are not allowed on Raman pc.
8. Stable electric power supply 230V, 50Hz with UPS should be made available (proper earthing need to be provided).
9. Room temperature must be maintained at 22°C +/- 1°C round the clock 24/7- 365 days and humidity must be maintained within 50-60% RH.
10. Instrument and laser warm up time is half an hour.

11. The Raman Spectroscopy instrument should be switched on every day and spectra should be collected on standard internal silicon sample for each laser/grating & maintain the record.
12. The Renishaw support should be immediately informed/contacted in case any fault is observed with the system
13. Regular log should be maintained for proper maintenance of the machine and for emergency trouble shootings.

4.2.2 Samples for Raman Spectroscopy Study.

- Thin films
- Pallets
- Powders
- Gemstones

4.2.3 Risk and Assessments(InVia Raman Microscope)

<i>Risk</i>	<i>Effect</i>	<i>Assessments</i>
<i>Hazardous chemical agents</i>	<ul style="list-style-type: none"> • <i>Serious illness</i> • <i>Immediate or long-term and range from mild eye irritation to chronic lung disease.</i> 	<ul style="list-style-type: none"> • <i>safety footwear</i> • <i>Lab Coat</i> • <i>Face Mask</i> • <i>Gloves</i> • <i>Goggles</i>
<i>High power laser</i>	<ul style="list-style-type: none"> • <i>Skin damage</i> • <i>Burns</i> • <i>Eye damage</i> 	<ul style="list-style-type: none"> • <i>Appropriate laser safety goggles must be worn while measurement.</i>
<i>Sample damage</i>	<ul style="list-style-type: none"> • <i>Burn</i> 	<ul style="list-style-type: none"> • <i>Laser power should be reduce significantly to avoid sample burning.</i>
<i>Electric shock</i>	<ul style="list-style-type: none"> • <i>Shock to user</i> 	<ul style="list-style-type: none"> • <i>Don't open the laser enclosure when it is switched on.</i>
<i>Short Circuit</i>	<ul style="list-style-type: none"> • <i>Fire</i> 	<ul style="list-style-type: none"> • <i>Fire alarm should be activated</i> • <i>Fire extinguisher should be installed and well run.</i> • <i>Fire service called.</i> • <i>Evacuate the building.</i>

4.3 UV-Vis NIR Spectrophotometer (PERKEN ELMER-Lambda 750)

UV Vis NIR spectrophotometer is operational since January 2017 and uses light to qualitatively and quantitatively analyze samples based on how much light travels through the sample and how much is reflected off of it.

Typical Application Areas

- Inorganic and organic chemistry
- Quality Control of raw materials and final products
- Biological Sciences – DNA, Protein, Blood
- Academia – teaching experiments
- Materials Science

<i>Data collection Modes</i>	<i>Ideal for</i>
<i>Wavelength Scan</i>	<i>Wavelength Scan Full or partial range spectral characterization.</i>
<i>Quant and Scanning Quant</i>	<i>Quantitative Analysis on discrete peaks or full spectra.</i>
<i>Time-Drive</i>	<i>Kinetics, including general and enzyme kinetics.</i>
<i>Wavelength program</i>	<i>Rapid measurement at single or multiple discrete wavelengths.</i>
<i>Beer's Lambert law</i>	<i>To find the unknown concentration of a solution.</i>

4.3.1 UV-Vis NIR Spectrometer Standard operating procedures for smooth and optimum System performance.

1. The room lodging UV-Vis NiR unit should be maintained clean & dust free.
2. Shoes are not allowed inside room.
3. Eating is not allowed inside the room.
4. Only trained authorized person is recommended to operate the machine.
5. If the instrument has been stored in a cold environment then it should be allowed to come to thermal equilibrium for 2 to 3 hours before operation so that start up calibration is not compromised by condensation.
6. The instrument must be placed on a stable, level bench or table capable of taking its weight with sufficient space around the instrument for ventilation to circulate freely

- 7 Memory sticks are not allowed on Spectrometer pc.
- 8 Stable electric power supply 90VAC-240VAC, 50/60Hz with UPS should be made available (proper earthing need to be provided).
- 9 Room temperature must be maintained at 10°C -35°C round the clock 24/7- 365 days and humidity must be maintained within 10-70%, non - condensing.
- 10 Wait for 20-30 minutes after switching on the lamp.
- 11 The PerkinElmer support should be immediately informed/contacted in case any fault is observed with the system
- 12 Regular log should be maintained for proper maintenance of the machine and for emergency trouble shootings.

4.3.2 Risk and Assessments (LAMBDA -750)

<i>Risk</i>	<i>Effect</i>	<i>Assessments</i>
<i>Hazardous chemical agents</i>	<ul style="list-style-type: none"> • <i>Serious illness</i> 	<ul style="list-style-type: none"> • <i>safety footwear</i> • <i>Lab Coat</i> • <i>Face Mask</i> • <i>Gloves</i> • <i>Goggles</i>
<i>Toxic Fumes</i>	<ul style="list-style-type: none"> • <i>eye irritation</i> • <i>Chronic lung disease.</i> 	<ul style="list-style-type: none"> • <i>Efficient laboratory ventilation must be provided when working with volatile solvents or toxic substances.</i>
<i>Spill of Waste disposal</i>	<ul style="list-style-type: none"> • <i>Burn</i> • <i>Skin damage</i> 	<ul style="list-style-type: none"> • <i>Disposal of some solvents and chemicals may be classed as hazardous waste and must be dealt with in accordance with local regulatory practice</i>
<i>Short Circuit</i>	<ul style="list-style-type: none"> • <i>Fire</i> 	<ul style="list-style-type: none"> • <i>Fire alarm should be activated</i> • <i>Fire extinguisher should be installed and well run.</i> • <i>Fire service called.</i> • <i>Evacuate the building.</i>



COMSATS Institute of Information Technology

M.A. Jinnah Campus Defense Road off Raiwind Road, Lahore

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COSHH Form Microscopy and Spectroscopy Lab

(For External and Internal Users)

Atomic Force Microscope (AFM): PARK-XE7

User Details:

<i>Name</i>		<i>Affiliation</i>	
<i>Department</i>		<i>Email</i>	
<i>Contact no.</i>		<i>Date:</i>	

Sample Details:

<i>Number of Samples</i>		<i>Nature of Samples</i>	
<i>Type of Investigation</i>		<i>Project ID</i>	
<i>Chemical Specifications</i>		<i>Sample IDs</i>	

Have you done the risk assessments for the samples being investigated?

Identify the hazards?

What are the control measures?

I declare that I have the assessed the risk of the samples listed above and consider that they are safe to be accessed provided that good laboratory practices are followed.

Name:

Signature:

Authorization Data:

Name of supervisor:

Signature:

For Official Use Only

For Internal Samples

Authorization provided by Lab *In charge*:

Date:

Signature:

For External Samples

Authorization provided by Head of Department:

Date:

Signature:



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COSHH Form

Microscopy and Spectroscopy Lab

(For External and Internal Users)

In Via Raman Microscope (Renishaw, Raman & PL)

User & Sample Details:

<i>Name</i>		<i>Affiliation</i>	
<i>Department</i>		<i>Email</i>	
<i>Contact no.</i>		<i>Date:</i>	

Sample Type:	Film <input type="checkbox"/>	Pallet <input type="checkbox"/>	Powder <input type="checkbox"/>
Sample IDs:	1. 2. 3. 4. 5.		
Excitation Laser Source:	457nm <input type="checkbox"/>	488nm <input type="checkbox"/>	514nm <input type="checkbox"/>
Laser Exposure Time:			
Characterization required:	RAMAN Spectra <input type="checkbox"/>		PL-Spectra <input type="checkbox"/>
	Note: Specify wavelength Range for PL spectra <u> nm </u> to <u> nm </u>		

Objective of Measurement	
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Name (Supervisor)		Signature	
Date			

Date of submission:		Received By:	
Measurement date:		User:	

“For Official Use”

Authorization Provided by: (For Internal Samples)

Name (Equipment In charge)			
Signature		Date	

Authorization Provided by: (For External Samples)

Name (Lab. In charge)			
Signature		Date	



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Microscopy and Spectroscopy Lab

(For External and Internal Users)

UV-VIS-NIR Spectrophotometer (Lambda -750)

Sample Details

Number of Samples		Date
Sample Name(s)		
Project ID (For Internal Samples)		
Formula/Composition	Liquid <input type="checkbox"/> Film <input type="checkbox"/> Other: <input type="checkbox"/> (Please specify): _____	
Reference in case of film	Air <input type="checkbox"/> Other <input type="checkbox"/> (Please specify) : _____	

User Details

Name	E-mail:
Dept.	Ext No.

Scan Parameters

Ordinate Mode (nm)	
Slit Width (nm)	
Scan Speed (nm/min)	
No of Scans	

Please Specify Scan mode/measurement parameters:

1	Absorbance	Wavelength range :	Single wavelength :	Time:
2	Transmittance	Wavelength range :	Single wavelength :	Time:
3	Reflectance	Wavelength range :	Single wavelength :	Time:
4	Beer's law	Wavelength Range: Time :	Known concentration of the standard Sample :	

Sample IDs:

Samples		Other details (If any)
1		
2		
3		
4		
5		

Hazards for Each Sample:

Hazard	Very Toxic	Toxic	Flammable	Corrosive	Harmful	Irritant	Highly Reactive
1	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
2	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
3	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
4	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Specific Precautions for Each Sample:

	Precautions			
	Gloves	Mask	Safety shield	Other (Specify)
1	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
2	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
3	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
4	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
5	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

I declare that I have assessed the risk of using the samples listed above and consider that they are safe to use provided that good laboratory practice is followed together with the safety requirements as detailed above.

Name		Signature	
Name (Supervisor)		Signature	
Date			

“For Official Use”

Authorization Provided by: (For Internal Samples)

Name (Equipment In charge)			
Signature		Date	

Authorization Provided by: (For External Samples)

Name (Lab. In charge)			
Signature		Date	

Characterization Charges & SOPs for External Users

<i>No</i>	<i>Characterization</i>	<i>Charges per Sample (PKR)</i>	
		<i>Academic</i>	<i>Commercial</i>
<i>1.</i>	<i>Atomic Force Microscopy</i>	<i>1500</i>	<i>3500</i>
<i>2.</i>	<i>Raman Spectroscopy</i>	<i>1000</i>	<i>3000</i>
<i>3.</i>	<i>Photoluminescence (PL) Spectroscopy</i>	<i>1000</i>	
<i>4.</i>	<i>UV-VIS Spectroscopy</i>	<i>500</i>	<i>1500</i>


Payment Procedure:

1. The external User will submit the samples along with the COHH Form at the rate of 5 samples per COSHH Form.
2. The Equipment Coordinator /In charge will issue the invoice for the payment.
3. The Invoice will be submitted by the payer to CIIT Lahore Account Office for Challan form issuance and record.
4. The Payer will pay the amount as per issued challan form.
5. A copy of the payed challan form will be submitted by the payer for the Lab record.
6. Necessary characterization procedures will be conducted for the submitted samples as per lab SoPs.
7. In case of more than 5 samples submitted for characterization the rates may be subsidized upon request as follows :

Academic Samples: 0.75(X)

Commercial Samples: 0.90(X)

X =original charges

 MSATS institute of information Technology Lahore			Sample Characterization Billing invoice
<i>Department Of Physics</i> <i>Microscopy and Spectroscopy Lab</i> <i>A- Block Room # A1</i> <i>Tel: 042-111-001-007 Ext. 832 Fax: 042-9203100</i>			<i>Invoice No. Date:</i> <i>Bank branch: HBL COMSATS</i> <i>Brach Lahore</i> <i>Account Title : Physics Testing</i> <i>IBAN : Pk71 HABB</i> <i>0023057902815303</i>
Bill To:			Sample Type: Commercial <input type="checkbox"/> Academic <input type="checkbox"/>
<i>Name:</i>			<i>Issue date:</i>
<i>Department/Company</i>			<i>Due date:</i>
<i>Email:</i>			
<i>Phone #</i>			
Characterization Technique	No. of Samples	Unite price	Amount
Issued by:			Total:

- Operational Manuals
- Log Book