Pharmaceutical Chemistry Department, Umm Al-Qura University Instrumentation SOPs 2022

Device Name	Rotary evaporator
Manufacturing Company	BUCHI
S. O. P	The purpose of this document is to ensure that Rotary evaporator is used in proper way.

1. Ensure that both the aspirator pump and the recirculating water bath (5 gallon bucket) are filled with ice.
2. Check that the power strip is turned on and plugged in.
3. Verify that the bump trap is clean and dry.
4. Add your sample to a round-bottomed flask. (Note: The RB flask should not be more than half full with liquid.) Correct (left) Incorrect (right)
5. Attach the round-bottom flask to the ground glass joint of the bump trap at the end of the distillation tube. (Note: Some assemblies will have an adapter between the bump trap and round-bottom flask.)
6. Ensure that all glassware is held securely in place with a plastic Keck clip and/or ring cap.
7. Turn on the rotary evaporator motor (green switch).
8. Adjust the dial to rotate the flask at medium speed.
9. Turn the aspirator pump.
10. Seal the vacuum by closing the valve at the top of the diagonal rotary evaporator condenser. (i.e. turn the knob until the arrow on it points straight down towards the attached tubing)
11. If necessary, carefully lower the round-bottom flask into the water heating bath. (Note: The hot water should cover the liquid level in the flask. If the RB flask is more than half-way

	full, the water should touch the bottom of the RB flask and as the liquid evaporates, the RB flask can be further immersed in the water.) Stop the rotovap when there is no more liquid dripping from the condenser coils for 30 seconds. (Note: For small volumes, dripping may not occur, wait 1-2 minutes and observe if there is any change.)

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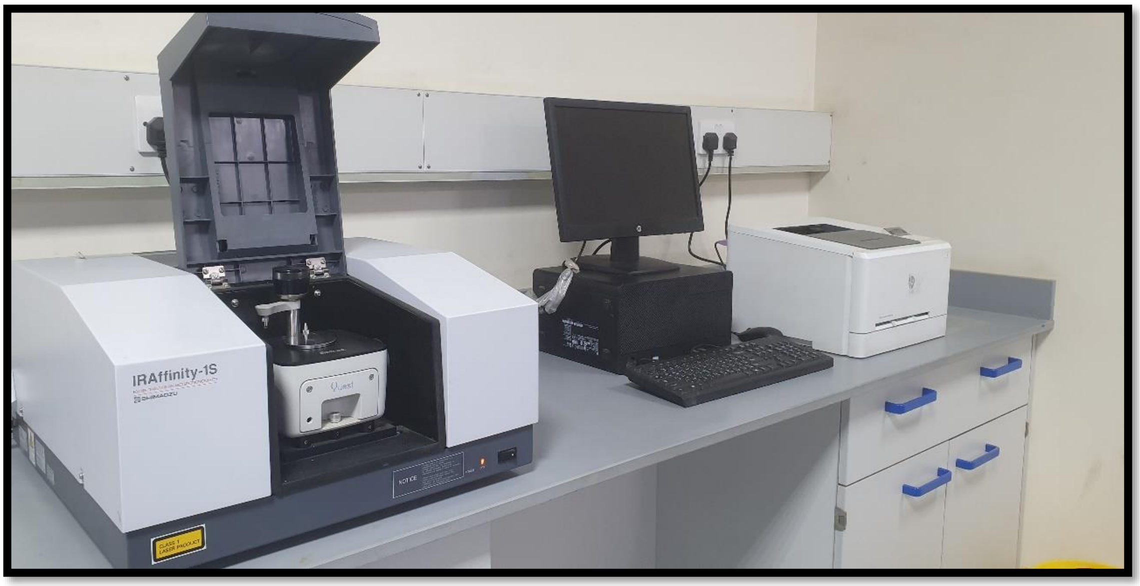

IRAffinity-1S

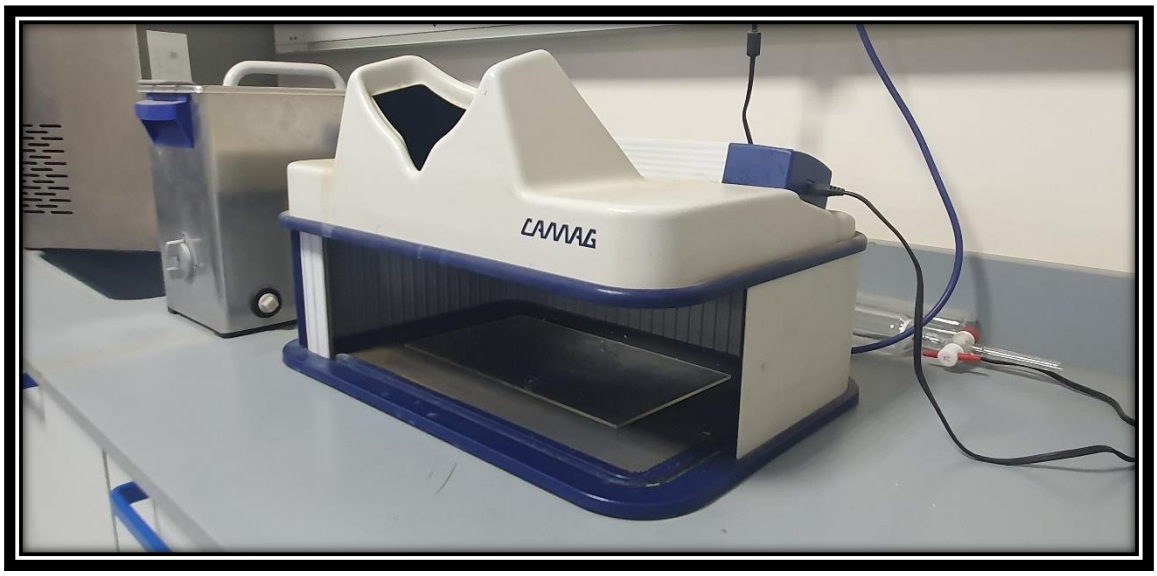
Device Name	**IRAffinity-1S**
Manufacturing Company	**Shimadzu**
S. O. P	1. Loosen black knob in middle of Pike ATR sample holder to raise the arm and swing to either side out of the way. Clean the ATR crystal and ATR crystal plate with an appropriate cleaner on a kimwipe (e.g. isopropyl alcohol, do NOT use acetone). Leave the arm out of the way. 2. Before operation, ensure that the orange light next to the power switch is illuminated (this indicates that the instrument is dry and eligible for operation). 3. Turn on power switch on front of the instrument (Green light will illuminate). 4. Open IRsolution program on computer desktop. Once the program opens, it will automatically begin its initialization procedure, let this run to completion (a popup may appear asking to perform an autoadjustment, if so hit “Cancel”). 5. Another popup will appear with the title, “QuickStart Accessories”, select “OK”. At this point, there should be four green lights along the right side of the program window. If so, the instrument is ready to use! (NOTE: if some or all of the lights are not green, please notify Tom Allston immediately!) 6. Select the option for “Measure”, then under the Data tab towards the bottom right side of program window, the parameters (i.e. scan range and number of scans) can be optimized by the user depending on the sample (a standard measurement is between 4000-400 1/cm and 10 to 20 scans). 7. Under the “Measure” button, you will see a line for Data file. Click the “…” button to select the folder needed and save your data by typing in your file name and click “Save.”

	8. Under “Measure” press BKG (background). The “Please prepare sample compartment for background scan” window will pop up. Press “OK” to begin the scans. The measurement then takes place (as indicated by the bottom left hand corner “scanned x of # scans” or if the scans have been completed “FTIR measurement ready.” 9. For a solid sample place enough of the solid on the crystal surface to fully cover it. Move the arm back to the center, and twist the knob clockwise until it stops. If using a liquid sample put the liquid crystal plate on the instrument by screwing it on the crystal holder. Put a few drops of sample in the area over the crystal and leave the handle out of the way. 10. Under “Measure” press Sample. The scanning will automatically begin. 11. Once complete, the program automatically goes to “View.” The background is automatically subtracted out from the sample scan. 12. Under the Manipulation 1 drop down menu, select peak table (at bottom of selection). The parameters to the right can be changed to optimize peaks. 13. The peaks are displayed by selecting “Calc”. If you get too many peaks, reduce the parameters starting with changing just the minimum area to a larger number. The peak table is displayed by selecting OK.” 14. Selecting “OK” after performing any data manipulation will display the changes to the original data file. To save changes, go to file and either choose the option to save (which saves changes over the original spectra) or save as (which will save the changes as a new spectra). 15. Clean crystal plate and apparatus with cleaner and kimwipe and go on to next sample. If finished running samples, close out of the IRsolution software (if there is any unsaved data, a prompt will appear asking you to do so. Either select yes to save changes or no

	to get rid of them) and turn off the instrument using the power switch on front.


ITV
IQ 85C

Device Name	Ice maker
Manufacturing Company	**ITV**
S. O. P	The purpose of this document is to ensure that ice maker is used in proper way. 1. Open faucet, make sure there are no leaks and check that water level is adequate. 2. Turn switch ON. Press the green button. Ice maker is ready


CAMAG

Device Name	UV Cabinet
Manufacturing Company	**CAMAG**
S. O. P	The purpose of this document is to have a closer look on the prepared sample that requires a quick inspection under UV light e.g. TLC plates during and after the reaction. 1. Turn switch On. 2. Put the wanted sample under the UV cabinet to analyze 3. Turn the switch off and keep the cabinet clean.

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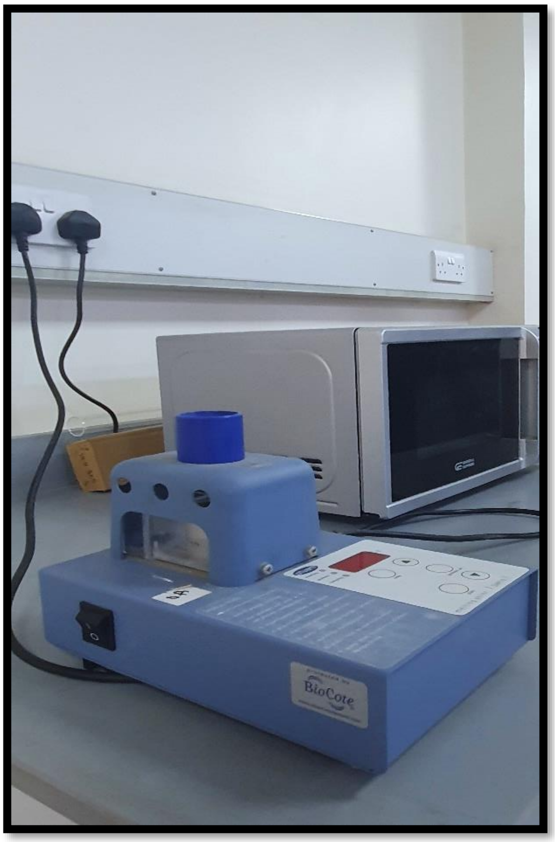
Device Name	Seven compact conductivities
Manufacturing Company	**Mettler Toledo**
S. O. P	For a detailed Standard operative procedure, check section 7 In the company manual in the provided code



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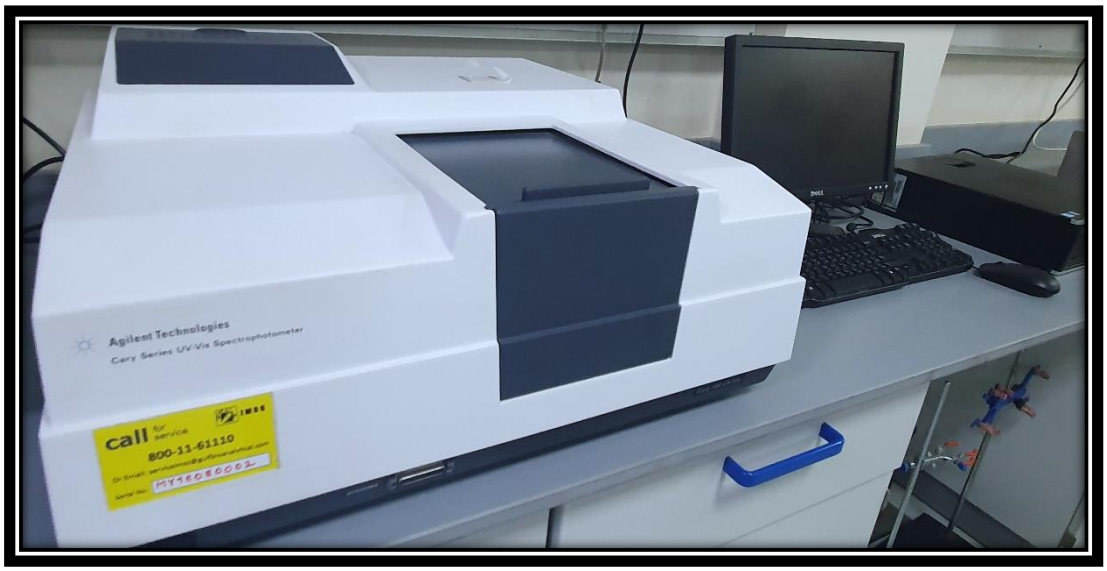
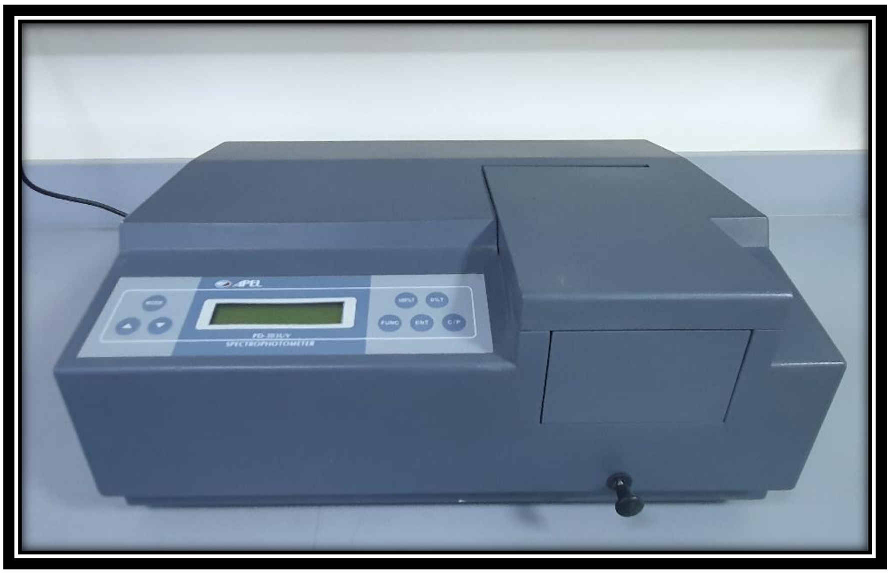
Device Name	**Melting point SMP10**
Manufacturing Company	**Stuart**
S. O. P	1 Prepare sample by placing a small amount in the end of a glass capillary tube. 2 Decide on a suitable plateau temperature. This should be approximately 10°C below the expected melting point of the sample. 3 Check that all 3 function lights are extinguished. If not, press the stop button. 4 Press and hold the set button (the plateau light will flash). The display will now show the current plateau temperature. When first switched on this will be 50°C but note that if the unit has recently been used the value may have been re-set by the previous user. 5 The desired plateau temperature may now be set using the arrow keys to scroll the display up or down as required. 6 Release the set button. The new plateau temperature is now set, and all function lights will go out (The plateau setting can be checked at any time during operation by pressing and holding the set button). This action will not interfere with the operation of the unit). 7 Insert the tube into the side of the heating block via the holes provided. For convenience this can be done from either side of the block. Look down the magnifier and position the tube so that the sample can be observed clearly 8 Press the start key. The unit will quickly heat up to the plateau temperature 9 Observe the sample until the melt occurs and record the temperature from the digital display 10 After the melt has occurred press the stop button. All function lights will go out and the unit will cool to ambient temperature.

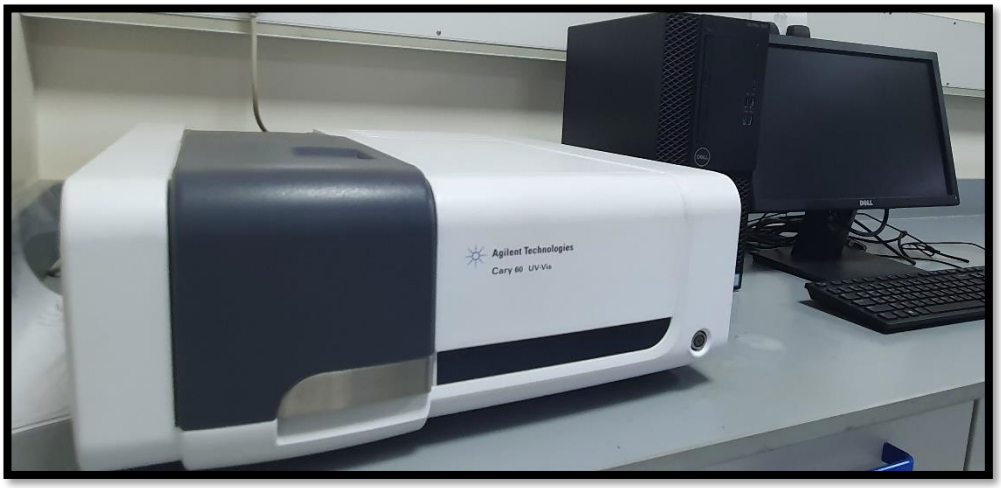

Agilent Technologies
Cary Series UV-Vis Spectrophotometer
call for service
800-11-61110

Device Name	CARY UV-Vis spectrophotometer
Manufacturing Company	**Agilent Technologies**
S. O. P	The purpose of this document is to ensure that spectrophotometer is used in proper way. 1. Turn the PC ON. 2. Turn the instrument ON. 3. Press Cary WinUV icon on the desktop. 4. Operation. 4.1 Scan. 4.1.1 Double click on scan icon 4.1.2 Wait till the reading on the upper corners become red. 4.1.3 From toolbar, press setup, new window will show up. According to experiment change the parameters; X mode (wavelength), Y mode (response hight), beam mode (by default dual beam), scan mode (advance), Baseline (baseline correction). Press OK, and in main window press START. 4.2 Simple read; 4.2.1 Double click in simple read icon 4.2.2 Press setup and change the parameters; Wavelength (the desired wavelength to measure the response), Y mode is the response (A, %T, or %R). press OK

	4.2.3 In main window press START. 4.3 Concentration; 4.3.1 Press concentration icon 4.3.2 Setup the parameters this includes wavelength, Y mode (response and height), according to experiment entre number of replicates. 4.3.3 From sub-toolbar, entre number of standers, STD concentration and STD units. 4.3.4 From sub-toolbar, entre samples name and numbers 4.3.5 From main window press START. Clean the cuvette and switch of the instrument.


SPECTROPHOTOMETER

Device Name	PD-303 UV Spectrophotometer
Manufacturing Company	APEL
S. O. P	The purpose of this document is to ensure that PD-303 UV is used in proper way. 1. Turn on the power. 2. The default diagnosis performs self-initializing and self-calibration. 3. Instrument function parameters can be edited by press [FUNC]. 4. To change any function press [ENT] the parameter will change automatically. The parameters should be changed according to the experiment conditions. 5. Measuring mode (Abs., Transmittance, or temperature) can be changed by pressing [MODE]. 6. Change wavelength value by press arrows. 7. Measure A or %T; A. Eliminate solvent absorbance by filling the blank solution on the cuvette and press 100%T. Measure individually all standards and samples solution, and record the values.


Agilent Technologies
Cary 60 UV-Vis
DELL

Device Name	CARY UV-Vis spectrophotometer
Manufacturing Company	**Agilent Technologies**
S. O. P	The purpose of this document is to ensure that spectrophotometer is used in proper way. 1. Turn the PC ON. 2. Turn the instrument ON. 3. Press Cary WinUV icon on the desktop. 4. Operation. 4.1 Scan. 4.1.1 Double click on scan icon 4.1.2 Wait till the reading on the upper corners become red. 4.1.3 From toolbar, press setup, new window will show up. According to experiment change the parameters; X mode (wavelength), Y mode (response hight), beam mode (by default dual beam), scan mode (advance), Baseline (baseline correction). Press OK, and in main window press START. 4.2 Simple read; 4.2.1 Double click in simple read icon 4.2.2 Press setup and change the parameters; Wavelength (the desired wavelength to

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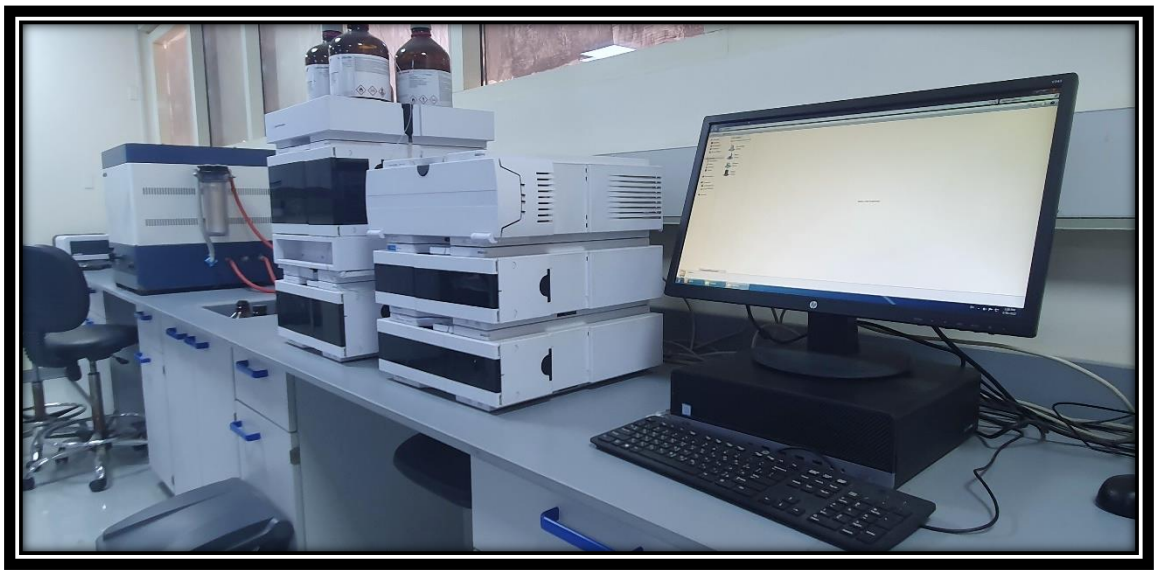


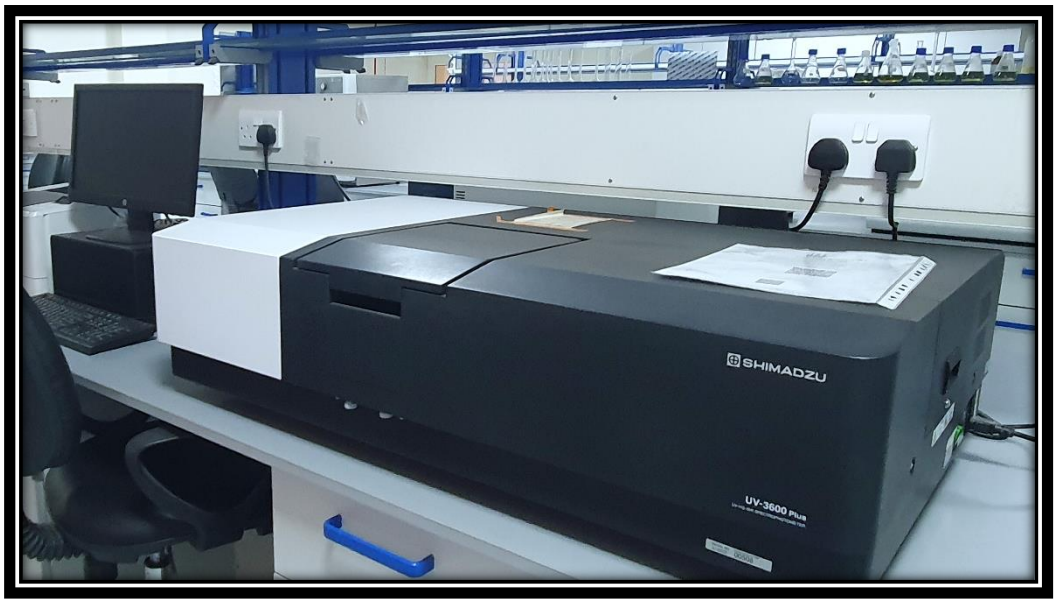
Device Name	HPLC 1260 Infinity II
Manufacturing Company	**Agilent Technologies**
S. O. P	1 Switch on the detector. 2 Fill the solvent bottles with adequate solvents for your application. 3 Place solvent tubing with bottle head assemblies into the solvent bottles. 4 Place solvent bottles into the solvent cabinet. 5 Solvent bottle filling dialog (in the software). 6 Purge the pump (in normal usage scenario). OR Prime the pump (after installation of the system). 7 Change solvent (if necessary). 8 Choose the tray format of the sampler. 9 Add a new column 10 Enter the column information 11 Select the column position 12 Set the detector according to the needs of your method For more information, check the official website of Agilent Technologies for the checkup, running methods and the software setup

Device Name	2010-PI GCMS
Manufacturing Company	Shimadzu
S. O. P	The purpose of this document is to ensure that GCMS is used in proper way. Before starting this procedure, the gas pressure in gas cylinder. 1. Starting GCMS 1.1. Turn ON the power to the MS, GC, and auto sampler 1.2. Turn ON the power to the PC. 1.3. At the desk top double click the (GCMS Real time analysis), and press OK 2. Startup the system 2.1. Make sure the carrier gas cylinder valve to supply carrier gas is open 2.2. Click the [Vacuum control] icon in assistant bar 2.3. Click [Auto startup] icon 2.4. When completed is displayed, click [close] 3. Checking for vacuum leaks 3.1. After 15 minutes from startup, click [Tuning] icon on the assistant bar. 3.2. Click [Peak Monitor View] icon. 3.3. Check for vacuum leaks A. Click the filament to turn on. B. Wait for 1 minute and compare the peak heights for m/z 18 (water) and m/z 28 (nitrogen). Water peak should be higher than nitrogen peak C. Close the tuning 4. Creating a folder with data explorer 4.1. Click the [data acquisition] icon 4.2. Click [data explorer] on toolbar 4.3. Open project folder and create new folder (specify name and location), when finish press close. 5. Qualitative Analysis

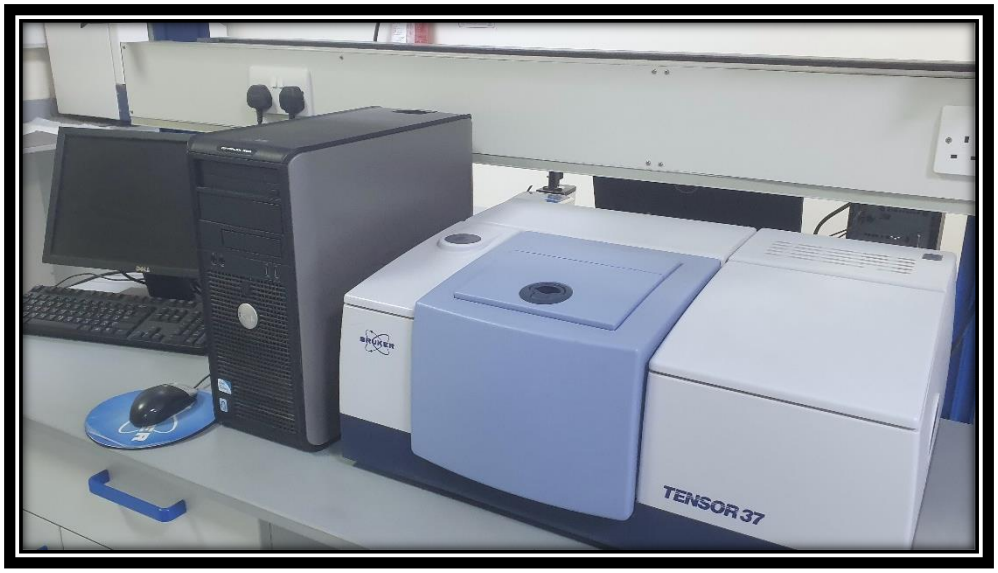
5.1. Click [Data acquisition] icon on the assistant bar.
5.2. Click [Method file] from file at toolbar.
5.3. Edit parameters in Sampler, GC, and MS according to experiment chromatographic procedure. Save method.
5.4. Running samples
   A. Check the solvent vials on auto-sampler (Methanol)
   B. Prepare samples according to the procedure and filter it and use new labeled vial. Put the vial at specific location on the auto-sampler.
   C. From method icon on toolbar, select download method parameters, wait till GC and MS be ready.
   D. For single sample use sample log to run the sample, or batch process for more than one samples.
   E. Fill all fields and information then click [Start] icon at the assistant bar.

6. Analyzing data.
6.1. Double-click the GCMS post-run analysis icon on the desk-top
6.2. Click the qualitative icon on the assistant bar.
6.3. Open file used on the qualitative analysis
6.4. Double-click on the sample to be analyzed.
6.5. Zoom in on a peak in the chromatogram (peak of interest)
6.6. On the highest point of interested peak click right-click and choose search for similarity.
6.7. According to the sample type, nature and structure choose the chromatogram.
6.8. Save the result.

7. Printing analysis reports
7.1. Click [Report] icon on the assistant bar.
7.2. Different report format can be chosen from the list. Press OK
7.3. Report can be edited
7.4. Click print.


SHIMADZU
UV-3600 Plus

Device Name	**UV-3600 Plus**
Manufacturing Company	**SHIMADZU**
S. O. P	For a detailed Standard operative procedure, check the filed information from the official website in the provided code


TENSOR 37

Device Name	Tensor 37
Manufacturing Company	Bruker
S. O. P	For a detailed Standard operative procedure, check the filed information from the official website in the provided code

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Mettler Toledo
METTLER TOLEDO

Device Name	Compact titrator G20
Manufacturing Company	**Mettler Toledo**
S. O. P	The purpose of this document is to ensure that automatic titrator is used in proper way. 1. Rinse and fill burette: 1.1. Empty the burette. Press the "burette" button. The screen will go to a menu. 1.2. Press the down arrow until the cursor is beside the burette functions at the bottom of the screen. Press the right arrow until the screen reads "Flush". 1.3. Press the √. The titrator will empty. Be sure an empty beaker is under the electrode to collect liquid. 2. Calibrate. Press the "pH/mV" button. Place the electrode manually in the calibration solutions. The titrator will read the value of calibration solution if the values within the range, the electrode is calibrated. Press the √. 3. Titrate samples. Check the method (according to the experiment) Begin titrations by pressing the "Run" button.

	4. To add titrant during the day, be sure that a set is not in progress. Clean area. Wipe counters, wipe equipment

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Ministry of Education
Umm Al-Qura University
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وزارة التعليم
جامعة أم القرى
٠٣١

الرقم :
التاريخ :
المرفقات :

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Device Name	**Lambda 365 UV vis spectrophotometer**
Manufacturing Company	**Perkin Elmer**
S. O. P	1 Turn on the power switch and allow the instrument to warm-up for at least 20 minutes. * Check the LED light, which is the lower left hand in front of the system. - Power: Blue -> Power on - Ready: White-> the communication of between PC and system - System: White-> during the checking of system status 2 Double-click UV Express folder. 3 Execute Scan mode icon in the UV Express Software folder. The following message box will be displayed. Empty the cell holder and close the lid firmly. Click OK. 4 Start System Self-Test. * System Self-Test is the procedure that the system checks instrumental conditions by itself. * System Self-Test is performed whenever the system's power is turned on and the software is executed.

Pharmaceutical Chemistry Department, Umm Al-Qura University Facilities 2022

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NMR AVANCH III HD 500 HZ