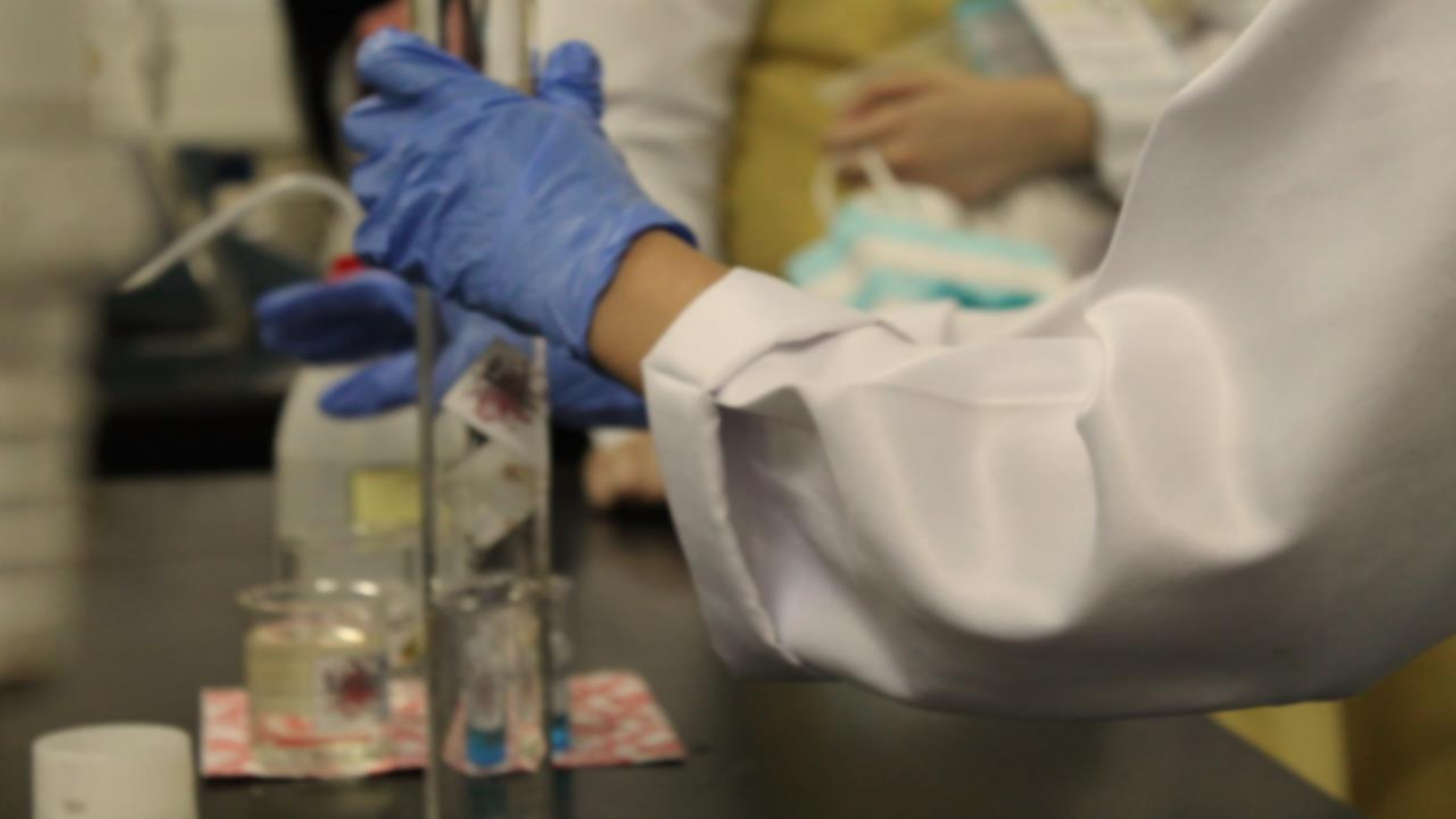



قسم
الكيمياء
Department of Chemistry

تقرير عن الأجهزة المعملية في مختبرات قسم الكيمياء

إعداد وتقديم :

أ/ رجاء عبدالوهاب نقلي

إشراف

د/ نها معتوق حلواني

أ/ زيني عرفه زيني

محتويات التقرير

محتويات التقرير

محتويات التقرير

المختبر

يُعرَّف المختبر بأنه المكان الذي تتم فيه أداء التجارب العملية وعمليات التحليل واختبار النظريات وتطبيقها، وتحويل المفاهيم المجردة ملموسة،
تحتوي مختبرات الكيمياء في العادة على أدوات وأجهزة مختصة للمساعدة على إجراء التجارب العلمية ، وفق إرشادات واستراتيجيات محددة للعمل فيه ، كما أنه البيئة التي توفر للطلبة في صفوفهم المخبرية فرصة اكتشاف الأساليب التي استُخدمت من قبل العلماء في مجالاتهم التخصصية ، وتتضمن هذه الصفوف بعض مهارات كتحضير التقارير الشفوية والمكتوبة ، وكيفية استخدام الأجهزة العلمية.
تسعى كلية العلوم التطبيقية إلى الارتقاء بأسس السلامة بمعامل الكلية إلى أفضل المعايير لتوفير بيئة أمنه لجميع طالبات وطلاب ومنسوبي الكلية بما يؤهلهم للقيام بواجباتهم.

الأمن والسلامة في المختبرات

تعدّ إجراءات السّلامة في المختبرات من ضمن القواعد والأسس العالمية، التي يجب أن يتبعها كل من يعمل في هذا المضمار، حمايةً لنفسه وللعاملين داخل المختبر، بالإضافة إلى حماية المواد والأبحاث التي يتم العمل فيها وتطويرها داخل المختبر:

- نشر الوعي والتثقيف بمدى أهمية الأمن والسلامة داخل المعامل عن طريق عقد الدورات التدريبية ووضع اللوحات الإرشادية والمنشورات والكتيبات.
- تطبيق اشتراطات ومتطلبات السلامة ووضع معايير السلامة داخل المعامل.
- تنفيذ اشتراطات الوقاية من الحريق داخل جميع معامل الكلية.
- عمل قاعدة بيانات لجميع المواد الكيميائية والأجهزة ومستلزمات المعامل.
- حصر احتياج المعامل من أجهزة وسائل الإنذار، والإطفاء والإنقاذ، والإسعافات الأولية، ومعدات مكافحة الحريق، والعمل على تأمينها بشكل دائم.
- المتابعة المستمرة والإشراف الدائم للتحقق من تطبيق تعليمات السلامة.
- متابعة أعمال الصيانة لجميع التجهيزات الخاصة بالسلامة داخل المعامل والتأكد من جاهزيتها في جميع الظروف.
- وضع الضوابط اللازمة للتأكد من التزام الطالبات ومنسوبي الكلية بارتداء تجهيزات الوقاية الشخصية داخل المعامل.
- اجراء التحقيقات في حوادث السلامة داخل المعامل ووضع توصيات

يقدم قسم الكيمياء تشكيلة واسعه من أجهزة المختبر عالية الأداء للمساعدة على إجراء التجارب العلمية
حيث يحتوي القسم في مقر الطلاب بالعابدية : ١٣ مختبراً طلابياً ، و ٦ مختبرات متخصصة لأبحاث العلمية . وفـي مقر الطالبات يوجد : ٦ مختبرات طلابية ، و ٤ مختبرات متخصصة لأبحاث العلمية ، وهذه المعامل مجهزة بالمواد الكيميائية ، والأجهزة العلمية الحديثة ، والأدوات الازمة ، يتسع المختبر الواحد إلى حوالي عشرون طالبة او طالب ، جميع هذه المختبرات مؤثثة بأثاث مختبري جيد وتحتوي على مختلف الزجاجيات وبعض الأجهزة المختبرية مثل الموازين الحساسة والحمامات المائية والسخانات الكهربائية بأنواعها والأفران الكهربائية وغيرها من الأجهزة والأدوات المختبرية الأخرى.

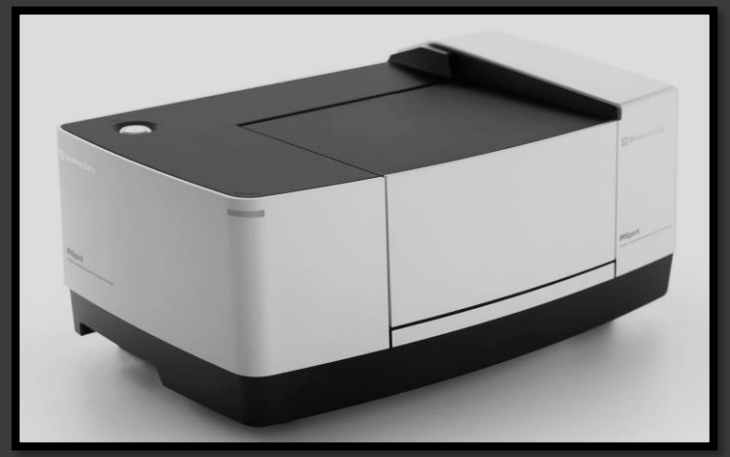
ويمكنكم الاطلاع على المزيد من المعلومات على الأجهزة لتبسيط عملك في المختبر،

Infrared spectroscopy

IR Spirit, Ready to Run

Fourier Transform Infrared Spectrophotometer

IR Pilot Identification Test Program



Space-Efficient with High Expandability

- Compact FTIR that travels where it’s needed.
- For sites with only a narrow space available, samples can be measured with the unit positioned horizontally or vertically.
- With the widest sample compartment in its class, it easily accommodates Shimadzu and third-party accessories.

IR Spirit

Dedicated IR Pilot Program Ensures Immediate and Easy System Operation

- IR Pilot includes 23 application programs as standard.
- Includes an identification test program convenient for routine inspections as standard.
- Includes a pass/fail judgment program specialized for contaminant analysis as standard.

High Reliability Ensures the System CanBe Introduced with Confidence

- Stable interferometer performance based on technology inherited from high-end models.
- Designed to endure even high-humidity environments (KRS-5 window is selectable).
- Instrument status monitoring function enables users to understand the instrument status easily
- Anti-theft and anti-drop keylock can be installed.

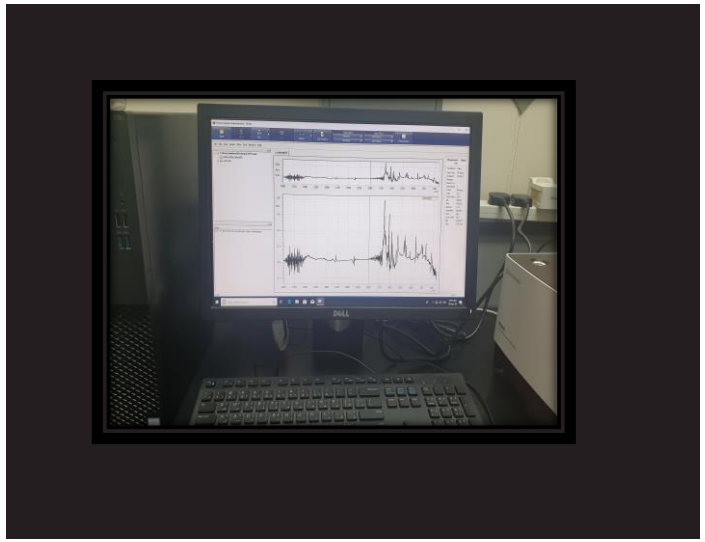
Features

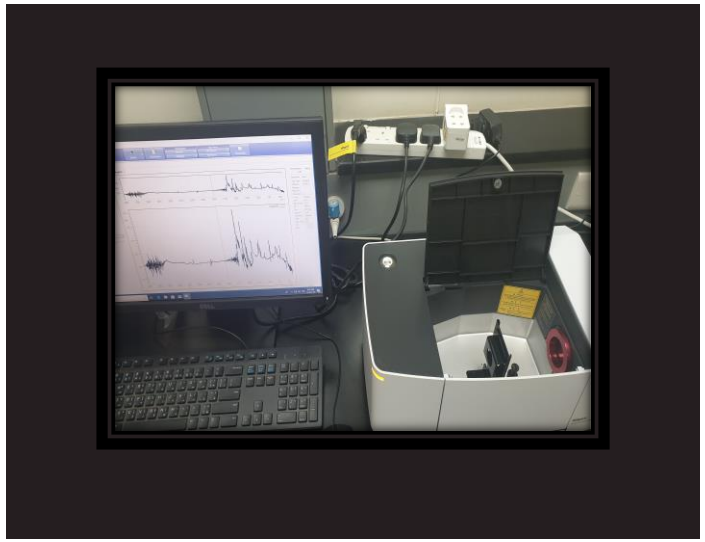
- ٣٠٠٠٠:١S/N or higher (1 minute integration)
- Resolution 0.5 cm-1
- Highly Stable Interferometer with Dynamic Alignment
- Built-in auto dryer – continuously monitors moisture inside the interferometer
- Self-diagnostics programs for excellence reliability
- Large sample compartment
- LabSolutions IR software easily executes FTIR operations such as scanning, data manipulation, quantitation, reporting, saving, user administration, and more.
- High-level administrative functions, a variety of data manipulation functions, and user-friendly macros provide for an easier, more user-friendly analysis environment.
- In addition, numerous optional programs are available to address all modern laboratory needs.

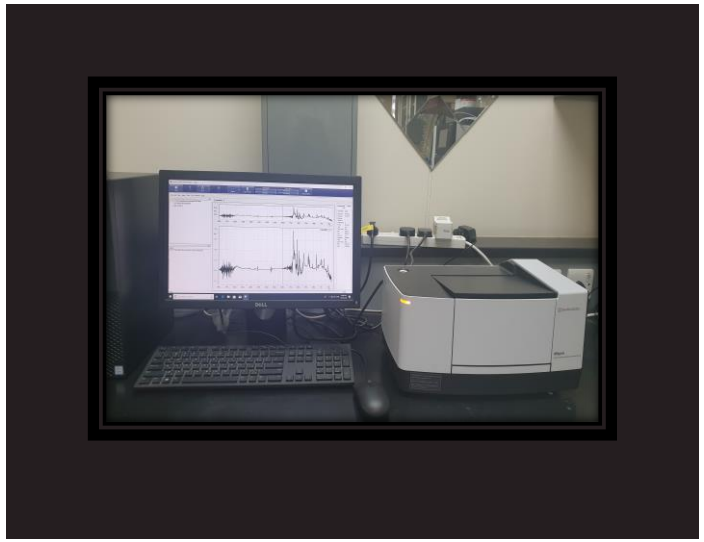
Operating an FTIR Spectrometer

- An FTIR spectrometer is a robust, modern instrument with many capabilities, but it must be used with care and respect. The most difficult step in taking the IR spectrum of a sample is often the preparation of the sample. If you are using the FTIR spectrometer, you can use the following operating procedure.
- 1. Prepare the sample.
- 2. Briefly open the sample compartment and confirm that there is nothing in the sample beam. Close the compartment.
- 3. Run a background scan. The data are collected, processed, and stored in the instrument's computer memory. The instrument indicates when this operation is completed.
- 4. Briefly open the sample compartment and place the sample in the sample beam. Close the compartment.
- 5. Run a sample scan. The data are collected and processed. The background scan is automatically subtracted from the sample scan. The result, an infrared spectrum of the sample, is displayed on the monitor.
- 6. Use the instrument's software to mark the frequency of each major peak in the region of 4000–1500 cm1. Having the exact frequencies (wavenumbers) of these peaks on the printed spectrum can be helpful in analyzing it.
- 7. Format the spectrum and print out a copy for analysis and for inclusion in your laboratory notebook.

Operating an FTIR Spectrometer







UV Spectroscopy

Instrumentation of UV Spectroscopy

Light Source

- Tungsten filament lamps and Hydrogen-Deuterium lamps are most widely used and suitable light source as they cover the whole UV region.
- Tungsten filament lamps are rich in red radiations; more specifically they emit the radiations of 375 nm, while the intensity of Hydrogen-Deuterium lamps falls below 375 nm.

Monochromator

- Monochromators generally is composed of prisms and slits.
- Most of the spectrophotometers are double beam spectrophotometers.
- The radiation emitted from the primary source is dispersed with the help of rotating prisms.
- The various wavelengths of the light source which are separated by the prism are then selected by the slits such the rotation of the prism results in a series of continuously increasing wavelength to pass through the slits for recording purpose.
- The beam selected by the slit is monochromatic and further divided into two beams with the help of another prism.

Sample and reference cells

- One of the two divided beams is passed through the sample solution and second beam is passé through the reference solution.
- Both sample and reference solution are contained in the cells.
- These cells are made of either silica or quartz. Glass can’t be used for the cells as it also absorbs light in the UV region.

Detector

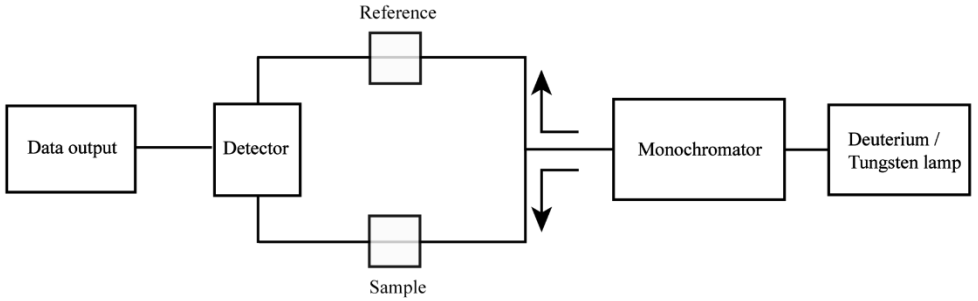
- Generally two photocells serve the purpose of detector in UV spectroscopy.
- One of the photocell receives the beam from sample cell and second detector receives the beam from the reference.
- The intensity of the radiation from the reference cell is stronger than the beam of sample cell. This results in the generation of pulsating or alternating currents in the photocells.

Amplifier

- The alternating current generated in the photocells is transferred to the amplifier.
- The amplifier is coupled to a small servometer.
- Generally current generated in the photocells is of very low intensity, the main purpose of amplifier is to amplify the signals many times so we can get clear and recordable signals.

Recording devices

- Most of the time amplifier is coupled to a pen recorder which is connected to the computer.
- Computer stores all the data generated and produces the spectrum of the desired compound.



Instrument Setup/operation

1. Turn on the PC and monitor.

2. Turn on the instrument, the switch is at the lower right corner.

3. Wait until the spectrometer has made some clicking noises.

4. Click enter then F4 on the UV-Vis program icon to start the program.

5. A program login box will appear. Simply press “cancel” or “Ok”.

6. The software should now open in the standard view with the visible lamp lit.

7. If you need to take a spectrum below about 400 nm click on the UV lamp icon to turn it on. The lamp takes a few seconds to light. You should allow the lamps to stabilize for about 15–20 minutes. If the exact absorbance is not important you can begin measurements immediately.

8. For wavelengths below 350nm both lamps are needed.

9. Place your samples in the UV/quartz cuvettes for the measurements.

Acquiring Data

1. You should first run a spectrum of you sample cuvette to make sure it is clean.

2. With nothing in the sample holder select “Blank” or “baseline” from the source window.

3. The resulting air blank should be a straight line with the noise below ±0.002 A.u. The noise will be higher in the region above 1000 nm.

4. After running an instrument blank, the previously grayed out “Sample” icon will now be accessible. Now put your solvent in your cuvette and run a spectra by selecting “Run” or “Sample”.

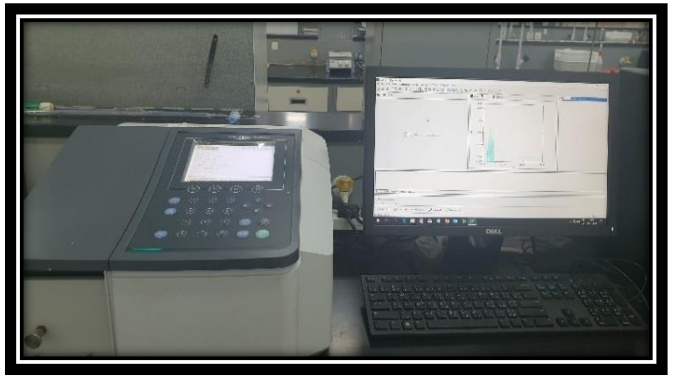
5. Again, your line should be mostly flat but not at 0. If the absorbance anywhere goes about 0.5 A.u. above the flat section, the cuvette or solvent is either not clean or is not suitable for the region of the spectra you are looking at. (Note in the UV you must use a quartz cuvette.)

6. If you are going to use two cuvettes one for your solvent blank and one for your sample:

a. You should run a blank with solvent in the blank cuvette.

b. Then a spectrum with solvent in the sample cuvette.

c. If the Absorbance is not zero and flat, the cuvettes are dirty or not matched.



7. To take a spectrum of your sample:

a. With your solvent in a cuvette in the sample holder, select “Blank” from the source pane.

b. Place your sample in the cuvette and select “Sample”. Your spectra should appear on the screen.

Processing Data

1. If you are going to save your data, you need to create a folder.

2. Data is not saved automatically.

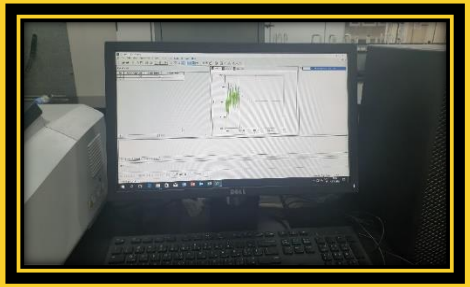
3. Each successive “Sample” measurement is overlaid in the “Overlaid Sample Spectra” view and added to the Results Table.

4. You can manipulate the current data; including zooming in, annotating more or fewer peaks, and printing results, but when you exit the program your data will not be automatically saved.

5. A spectral region may be selected by dragging a box around the area of interest.

6. Use (View -> Reset Current View) to return to the full spectral display.

7. When additional spectra are acquired, they will be overlaid in the spectra window.



8. To delete a spectrum from the view, select it by clicking on the appropriate trace in the “Sample Spectra” view. The selected points will appear on the selected trace, and a “Delete Selected Sample” button option will appear below the Sample Spectra view. The selected trace may now be deleted.

9. To save data files, click on the spectrum to disk icon located in the tool bar. Alternatively, you can select File -> Save -> Samples. Then, store your data in your folder.

10. If you want to export the data save it as a CSV. These files can be easily read by Excel. The data will appear in three columns, the wavelength in nm, the Absorbance (or Transmittance), and a noise or error estimate. Usually the last column is ignored.

11. If the Absorbance is > 1.3 or the Transmittance is < 0.05, the data is likely to be unreliable and should dilute your sample solution.

12. After finishing your measurements:

a. Exit the software.

b. Turn off the instrument power.

c. Shut down the computer and turn off the monitor.

Gas Chromatography

(GC)

Gas Chromatography (GC)

Gas chromatography (GC) is an analytical technique used to separate the chemical components of a sample mixture and then detect them to determine their presence or absence and/or how much is present. These chemical components are usually organic molecules or gases. For GC to be successful in their analysis, these components need to be volatile, usually with a molecular weight below 1250 Da, and thermally stable so they don’t degrade in the GC system.

GC is a widely used technique across most industries: for quality control in the manufacture of many products from cars to chemicals to pharmaceuticals; for research purposes from the analysis of meteorites to natural products; and for safety from environmental to food to forensics. Gas chromatographs are frequently hyphenated to mass spectrometers (GC-MS) to enable the identification of the chemical components.

Gas Chromatography Analysis

As the name implies, GC uses a carrier gas in the separation, this plays the part of the mobile phase (Figure 1 (1)). The carrier gas transports the sample molecules through the GC system, ideally without reacting with the sample or damaging the instrument components.

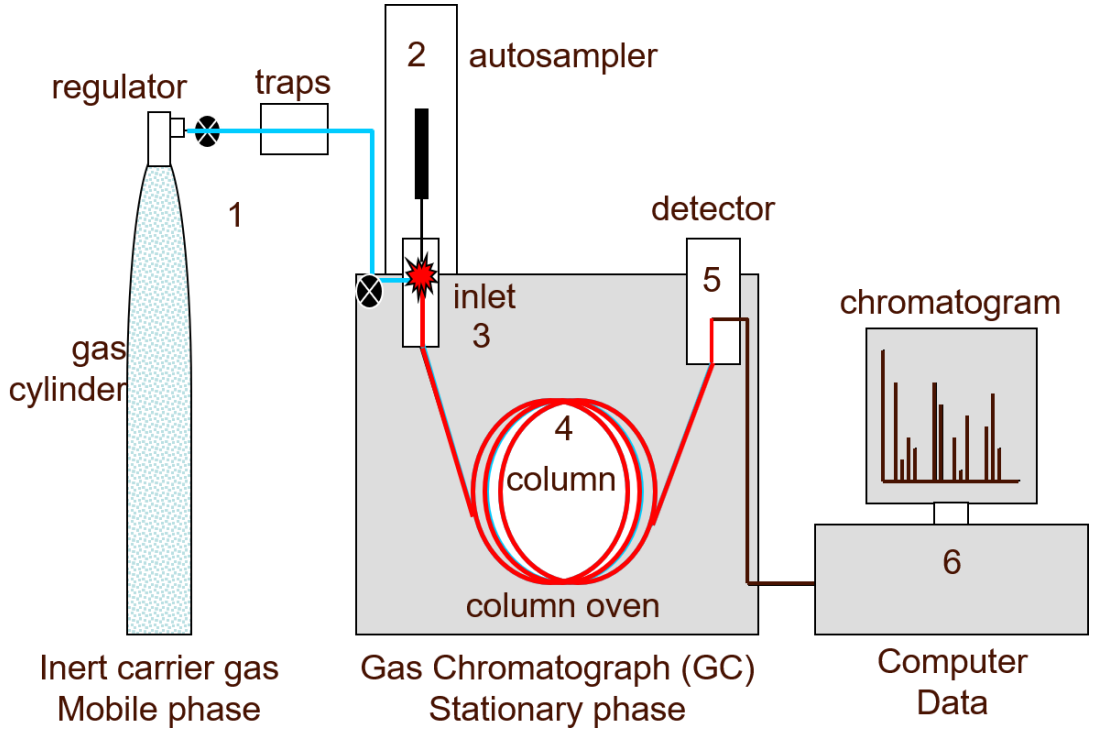


Figure 1: A simplified diagram of a gas chromatograph showing: (1) carrier gas, (2) autosampler, (3) inlet, (4) analytical column, (5) detector and (6) PC. *Credit: Anthias Consulting.*

2- The sample is first introduced into the gas chromatograph (GC), either with a syringe or transferred from an autosampler (Figure 1 (2)) that may also extract the chemical components from solid or liquid sample matrices.

3- The sample is injected into the GC inlet (Figure 1 (3)) through a septum which enables the injection of the sample mixture without losing the mobile phase.

4- Connected to the inlet is the analytical column (Figure 1 (4)), a long (10 – 150 m), narrow (0.1 – 0.53 mm internal diameter) fused silica or metal tube which contains the stationary phase coated on the inside walls.

5- The analytical column is held in the column oven which is heated during the analysis to elute the less volatile components.

6- The outlet of the column is inserted into the detector (Figure 1 (5)) which responds to the chemical components eluting from the column to produce a signal.

7- The signal is recorded by the acquisition software on a computer to produce a chromatogram (Figure 1 (6)).

8- After injection into the GC inlet, the chemical components of the sample mixture are first vaporized, if they aren't already in the gas phase. For low concentration samples the whole vapour cloud is transferred into the analytical column by the carrier gas in what is known as splitless mode. For high concentration samples only a portion of the sample is transferred to the analytical column in split mode, the remainder is flushed from the system through the split line to prevent overloading of the analytical column.

9- Once in the analytical column, the sample components are separated by their different interactions with the stationary phase. Therefore, when selecting the type of column to use, the volatility and functional groups of the analytes should be considered to match them to the stationary phase. Liquid stationary phases mainly fall into two types: polyethylene glycol (PEG) or polydimethylsiloxane (PDMS) based, the latter with varying percentages of dimethyl, diphenyl or mid-polar functional groups, for example cyanopropylphenyl. Like separates like, therefore non-polar columns with dimethyl or a low percentage of diphenyl are good for separating non-polar analytes. Those molecules capable of π-π interactions can be separated on stationary phases containing phenyl groups. Those capable of hydrogen bonding, for example acids and alcohols, are best separated with PEG columns, unless they have undergone derivatization to make them less polar.

10- The final step is the detection of the analyte molecules when they elute from the column. There are many types of GC detectors, for example: those that respond to C-H bonds like the flame ionization detector (FID); those that respond to specific elements for example sulfur, nitrogen or phosphorus; and those that respond to specific properties of the molecule, like the ability to capture an electron, as is used with the electron capture detector (ECD).

High-performance liquid chromatography (HPLC)

High perfomance Liquid Chromatography

HPLC has, without a doubt, grown to be the most popular and versatile of all analytical techniques in laboratories today. HPLC can be used for applications in such diverse industries as food and beverages, forensics, pharmaceuticals, drug discovery, environmental, and petrochemical. With a wide range of systems and components, including data systems, and outstanding reputation for long life, precision, and practically maintenance-free operation, Shimadzu has the resources to meet user requirements in nearly every market and application.

High performance liquid chromatography (HPLC) is basically a highly improved form of column liquid chromatography.

Instead of a solvent being allowed to drip through a column under gravity, it is forced through under high pressures of up to 400 atmospheres. That makes it much faster.

All chromatographic separations, including HPLC operate under the same basic principle; separation of a sample into its constituent parts because of the difference in the relative affinities of different molecules for the mobile phase and the stationary phase used in the separation.

Types of HPLC

There are following variants of HPLC, depending upon the phase system (stationary) in the process :

Normal Phase HPLC

This method separates analytes on the basis of polarity. NP-HPLC uses polar stationary phase and non-polar mobile phase. Therefore, the stationary phase is usually silica and typical mobile phases are hexane, methylene chloride, chloroform, diethyl ether, and mixtures of these.

Polar samples are thus retained on the polar surface of the column packing longer than less polar materials.

Reverse Phase HPLC

The stationary phase is nonpolar (hydrophobic) in nature, while the mobile phase is a polar liquid, such as mixtures of water and methanol or acetonitrile. It works on the principle of hydrophobic interactions hence the more nonpolar the material is, the longer it will be retained.

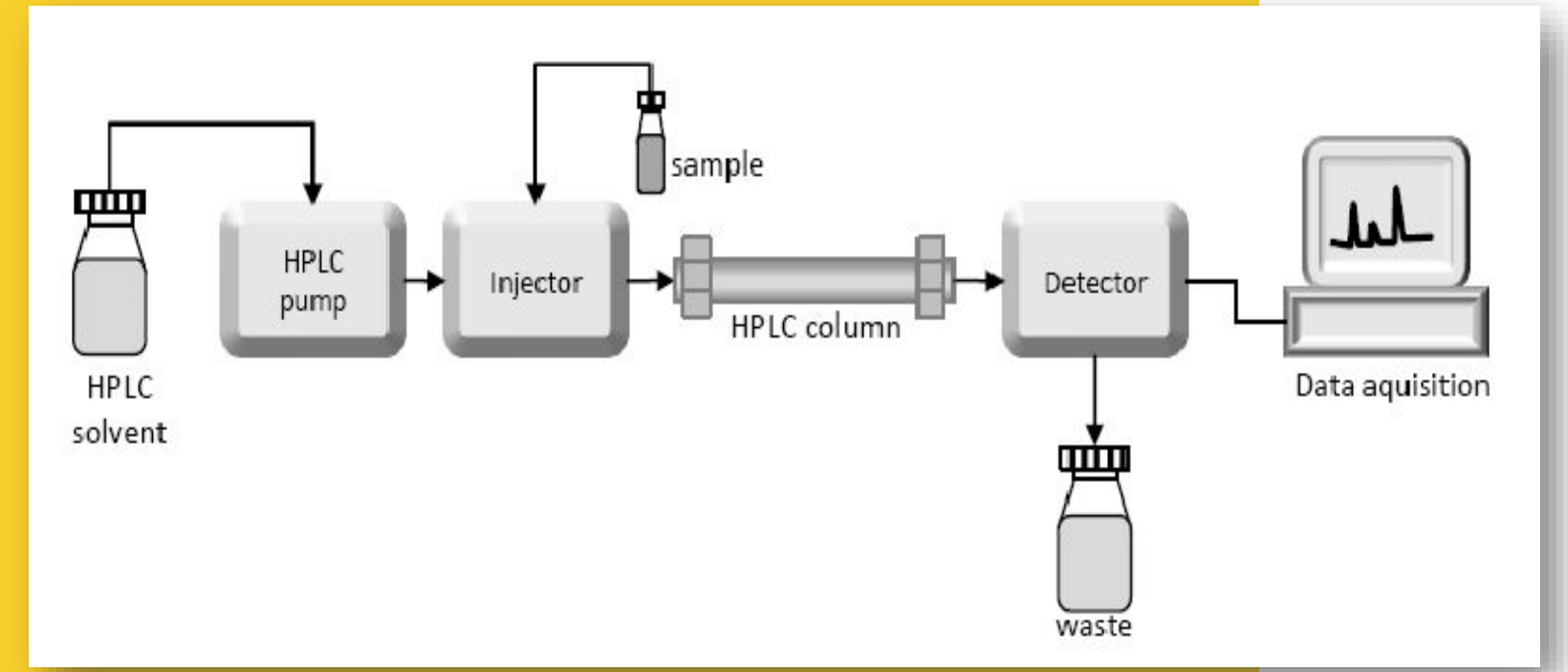
Size-exclusion HPLC

The column is filled with material having precisely controlled pore sizes, and the particles are separated according to its their molecular size. Larger molecules are rapidly washed through the column; smaller molecules penetrate inside the porous of the packing particles and elute later.

Ion-Exchange HPLC

The stationary phase has an ionically charged surface of opposite charge to the sample ions. This technique is used almost exclusively with ionic or ionizable samples.

The stronger the charge on the sample, the stronger it will be attracted to the ionic surface and thus, the longer it will take to elute. The mobile phase is an aqueous buffer, where both pH and ionic strength are used to control elution time.



Instrumentation of HPLC

As shown in the schematic diagram in Figure above, HPLC instrumentation includes a pump, injector, column, detector and integrator or acquisition and display system. The heart of the system is the column where separation occurs.

Solvent Resorvoir

Mobile phase contents are contained in a glass resorvoir. The mobile phase, or solvent, in HPLC is usually a mixture of polar and non-polar liquid components whose respective concentrations are varied depending on the composition of the sample.

Pump

A pump aspirates the mobile phase from the solvent resorvoir and forces it through the system’s column and detecter. Depending on a number of factors including column dimensions, particle size of the stationary phase, the flow rate and composition of the mobile phase, operating pressures of up to 42000 kPa (about 6000 psi) can be generated.

Sample Injector

The injector can be a single injection or an automated injection system. An injector for an HPLC system should provide injection of the liquid sample within the range of 0.1-100 mL of volume with high reproducibility and under high pressure (up to 4000 psi).

Columns

Columns are usually made of polished stainless steel, are between 50 and 300 mm long and have an internal diameter of between 2 and 5 mm. They are commonly filled with a stationary phase with a particle size of 3–10 µm.

Columns with internal diameters of less than 2 mm are often referred to as microbore columns. Ideally the temperature of the mobile phase and the column should be kept constant during an analysis.

Detector

The HPLC detector, located at the end of the column detect the analytes as they elute from the chromatographic column. Commonly used detectors are UV-spectroscopy, fluorescence, mass-spectrometric and electrochemical detectors.

Data Collection Devices

Signals from the detector may be collected on chart recorders or electronic integrators that vary in complexity and in their ability to process, store and reprocess chromatographic data. The computer integrates the response of the detector to each component and places it into a chromatograph that is easy to read and interpret.

HPLC Operation

The basics of HPLC have been outlined and the technique will be demonstrated in the laboratory. HPLC can be used to separate and quantify components for example diet soda.

HPLC Operation

1- To prepare the mobile phase, add 400 mL of acetonitrile to 1.5 L of purified deionized water. Then carefully add 2.4 mL of glacial acetic acid. Dilute the solution to a total volume of 2 L. The resulting solution should have a pH between 2.8 and 3.2.

2- Adjust the pH to 4.2 by adding 40% NaOH, drop-wise to the stirring solution, with the use of a calibrated pH meter.

3- Filter the mobile phase through a 0.47-µm membrane filter under vacuum to degas the solution and remove solids that could plug the column. It is important to degas the solution, as bubbles can cause voids in the stationary phase, or work their way to the detector cell and cause instability in measurements.

4- Prepare three component solutions of caffeine, benzoate, and aspartame, which are three typical components of diet sodas. These component solutions are then used to prepare the standard solutions that will be utilized to determine the unknowns. Prepare 500 mL of the caffeine and benzoate solutions.

5- Prepare 100 mL of the aspartame component solution. Store the solution in the refrigerator when not in use to avoid decomposition.

6- prepare 7 standard solutions, each with different concentrations of caffeine, benzoate, and aspartame. Pipet the proper amount of each component into a volumetric flask, and dilute to the 50-mL mark with mobile phase.

7- The first 3 solutions each contain one component, to enable peak identification. The other 4 solutions contain a range of concentrations of all 3 components, in order to correlate peak height to concentration.

8- Pour each standard solution into a labeled vial in a sample rack. Store the sample rack with samples and the remaining solutions in the refrigerator.

HPLC Operation

9- Set up the mobile phase and waste containers. Ensure that the waste lines are fed into a waste container, and are not recycling back into the mobile phase. Ensure that the inlet mobile phase line is fed into the mobile phase container.

10- Verify that the flow rate of the mobile phase is set to 0.5 mL/min. This flow rate will enable all components to elute within 5 min, but is slow enough to ensure resolution of individual peaks.

11- Verify the minimum and maximum pressures on the solvent delivery system. These settings shut the pump off in case of a leak or clog, respectively.

12- Press "zero" on the detectors front panel, to set the blank. Rinse a 100-µL syringe with deionized water, then with several volumes of 1 of the 7 working standards. Then fill the syringe with that solution. Begin with the 3 single-component samples in order to identify the peak of each component.

13- Manually inject the solution, by placing the injector handle in the load position. Slowly inject the 100 µL of solution through the septum port.

14- Verify that the data collection program is set to collect data for 300 s, which allows for enough time for all 3 peaks to elute through the detector. When ready to begin the trial, rotate the injector handle to the inject position, in order to inject the sample into the mobile phase. Immediately, click "Start Trial" on the data collection program. When the scan is complete, repeat the process for each of the 7 standard solutions. For each of the first 3 standards, only one of the 3 peaks appears. Note the location of the peak, which is used to identify the component.

15- Select 3 diet soda samples, and allow them to sit out in open containers overnight to remove the carbonation.

16- After overnight degassing, draw approximately 3 mL of each diet soda into a plastic syringe. Next, attach a filter tip to the syringe and push the soda through the filter into a glass vial, in order to remove any solid particulates.

17- Dilute 2 mL of each sample with 2 mL of the mobile phase to decrease the soda concentration by half.

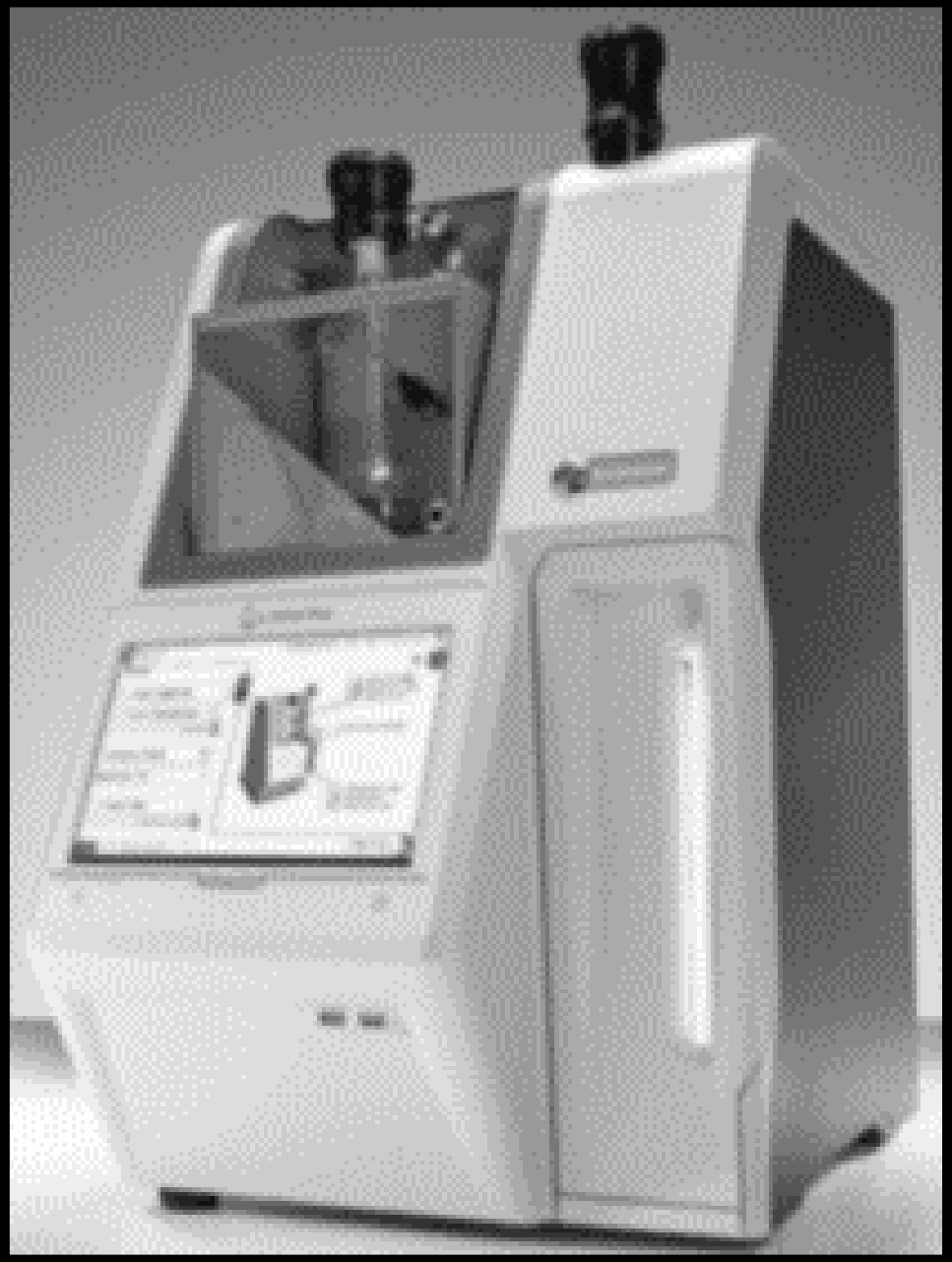
HPLC Operation

18- Draw 100 µL of one of the soda samples into a syringe, and inject it into the sample loop. Run the trial with identical parameters to the standard solutions. Repeat for each soda sample.

19- Correlate the peak areas of the standard samples to the known concentrations. To do so, determine the peak areas on the chromatographs for each standard sample using the triangular method. Calculate the peak height times with the width at half of the height, and use this value as the peak area.

20- Using the peak area and known concentrations create a calibration curve for each component, and determine the least-squares fit for each calibration curve.

21- Calculate the concentration of each component in the diet sodas from the peak areas. Remember that the sodas were all diluted by a factor of 2 prior to injection into the HPLC. Based on these results, calculate the mg of each component in a 12-oz can of soda



Atmospheric Distillation Analyzer –

Anton Paar- Diana 700

Atmospheric distillation

For distillation of hydrocarbon-based fuels. the whole process shows data and correlation curves of the temperature and volume and provide initial distillation point temperature and final distillation point temperature value, also can provide temperature and flow rate at percent point. Can detect local atmospheric pressure automatically and correct for the temperature at standard atmospheric pressure automatically. Testing and results are in full compliance with ASTM D86, D850, D1078, ISO 3405, IP 123, IP 195.

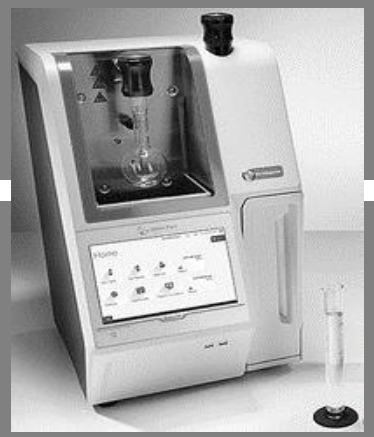
High Performance Atmospheric Distillation

- The distillation at atmospheric pressure is a crucial test to maximize the yield of different petroleum products out of crude oil based on their boiling range characteristics.
- The distillation behavior also provides important information about composition, properties, and behavior during storage and use.
- This affects the safety regulations as well as the handling and the performance of hydrocarbon-based fuels.
- Anton Paar’s distillation unit Diana 700 is the most convenient solution for performing high-precision atmospheric distillation tests on petrochemical products.
- Sophisticated temperature measurement and volume detection make sure that your results are highly accurate.

Instrument Features to ensure perfection fro the first drop

- Robust and cost-saving instrument
- Smart operator assistance
- Inbuilt features for the highest safety level
- Mobile multi-plug with indestructible temperature sensor
- Automatic positioning of heater and shield
- Intuitive and easy operation with customizable user interface
- Highly accurate volume detector
- The highest accuracy for your results

Atmospheric Distillation Analyzer – Anton Paar-Diana 700 is consisting of



1. Distillation flask, 125 mL
2. Multi-plug
3. Receiving cylinder, 100 mL
4. Drip plate
5. Boiling stones
6. Flask support board, 38 mm and 50 mm
7. Condenser tube stopper

Atmospheric Distillation Analyzer – Anton Paar - Diana 700 Operation

1. If required, perform a volume detector check.
2. Launch the "Easy Distill" feature.
3. Define a sample name.
4. Select the method “ASTM Group 1” or “ASTM Group 4”
5. Clean the condenser tube by threading the cleaning wire with the felt through the tube and pulling it out.
6. Select and place the flask support board in the heating chamber.
7. Slip the stopper onto the condenser tube.
8. Select the distillation flask.
9. Measure 100 mL of the sample in the receiving cylinder.
10. Place the cylinder into the cooling chamber.
11. Tap “Scan Volume”.
12. Pour the sample into the distillation flask.
13. Add boiling stones to the sample.
14. Mount the multi-plug into the flask.
15. Attach the flask to the condenser tube.
16. Insert the drip plate into the receiving cylinder.
17. Place the receiving cylinder into the cooling chamber if not already done so.
18. Close the cooling chamber.
19. Tap <DISTILL>.
20. After the distillation, measure the residue in the flask using the 100 mL graduated cylinder.
21. Perform an automatic residue scan by tapping “Automatic volume scanning”.
22. Tap <Edit> and enter the amount into the "Residue" field.






Automatic vacuum distillation

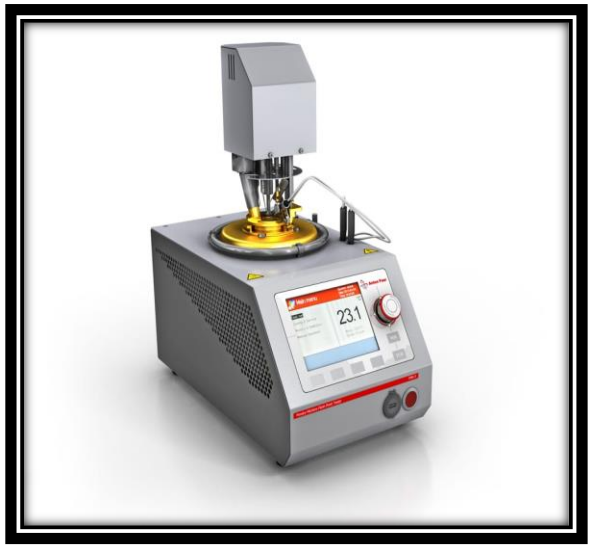
Performs distillation in accordance with ASTM D1160. MINIDIST 1160 version V6 is a stand-alone, bench-size unit designed to automatically run vacuum distillation of crude residue and high boiling mineral fractions up to 600°C/1100°F AET in accordance with ASTM D1160.

K71000 Automatic PMCC Flash Point Analyzer

The Koehler model K71000 Automated Pensky-Martens Closed Cup Flash Point Analyzer is the latest design for performing the ASTM D93 test method and related test specifications.

The K71000 determines the flash points of distillate fuels such as:

- Diesel
- Biodiesel blends
- Kerosene
- Heating oil
- Turbine fuels
- New and in-use lubricating oils
- Residual fuel oils
- Cutback residual
- Used lubricating oils
- Mixtures of petroleum liquids containing suspended solids
- Mixtures of petroleum liquids that tend to form a surface film during testing and biodiesel (B100)



Closed cup automatic detection of flash and fire Point Pensky Martens, application range up to 400 °C (°C/°F selectable).

The K71000 determines the flash points of a wide range of products by a closed cup method with stirring of the sample.

Flash point is the lowest temperature barometrically corrected to standard atmospheric pressure of 101.3 kPa (760 Torr) at which the vapors of a combustible liquid will ignite and the flame front propagate across the head-space of the sample cup under the conditions specified by the test method.

The Koehler model K71000 Automated Pensky-Martens Closed Cup Flash Point Analyzer is consisting of:

- Standard Cup and cover approx. 75 ml (with filling mark)
- Heating device
- Gas or Electric Igniter
- Thermometer
- Stirring

Method Scope:

The scope of the method covers the analysis of products in the temperature range of 40 to 360℃ but this automated apparatus extends the range from dew point + 10℃ to 400℃.

Automated Pensky-Martens Closed Cup Flash Point Analyzer Operation and Method Summary:

1. Flash point tests are conducted by filling the test cup to the full indication mark.
2. Placing the flash cup into the test sample chamber then locking the cup cover into position.
3. Next, the user selects a predefined or user programmed test method.
4. Types in or accepts the expected flash point (EFP), optionally selects a product type, then hits the start button.
5. A quick test feature allows for determination of flash points of unknown materials beginning the test from ambient and proceeding with the test at an accelerated heating rate.
6. The unit is equipped with a differential Pt-100 RTD probe designed to duplicate the response time of a mercury-in-glass thermometer as per ASTM D93-02a and E1-03a.
7. If a flash is not detected 30℃ above the expected flash point or at 405℃, then the test is automatically aborted for safety.
8. Once analysis is complete the results are displayed to the screen and saved to the instrument hard disk drive.
9. The results may be opened in a report generation program like MS Word or Excel and sent to a local or network printer.
10. Once the results have been reviewed and accepted by the operator the results may be sent directly to a network folder for processing and direct entry into LIMS.
11. Once analysis is complete the instrument actuates the rapid cool-down system.
12. The cool-down system is capable of reducing the temperature of the sample from300 to 30℃ in about 8 minutes in a laboratory with ambient conditions at 20℃.
13. User can also manually switch to standard air cooling.
14. Cooling the system will take a longer period of time however fan noise will be reduced.

K27000

Smoke Point Lamp

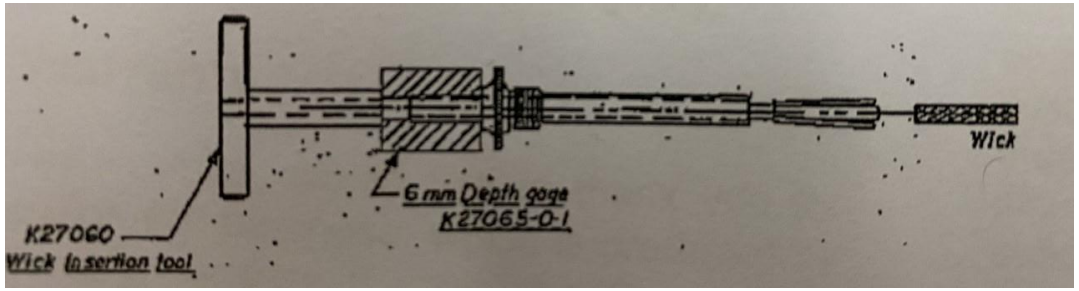
One of the oldest test methods in the petroleum industry. This test method provides an indication of the relative smoke producing properties of kerosenes and aviation turbine fuels in a diffusion flame. It is related to the hydrocarbon composition of such fuels. Conforms to ASTM D1322 and related specifications

Smoke Point is an indicator of the combustion qualities of aviation turbine fuels and kerosene. The K27000 Smoke Point Lamp measures the maximum flame height attainable without smoking.

The K27000 Smoke Point Lamp Device is consisting of:

- Smoke point lamp (Chimney and scale)
- Candle
- Wicks
- Wick tube
- Wick Insertion Tool
- Scissors or sharp razor

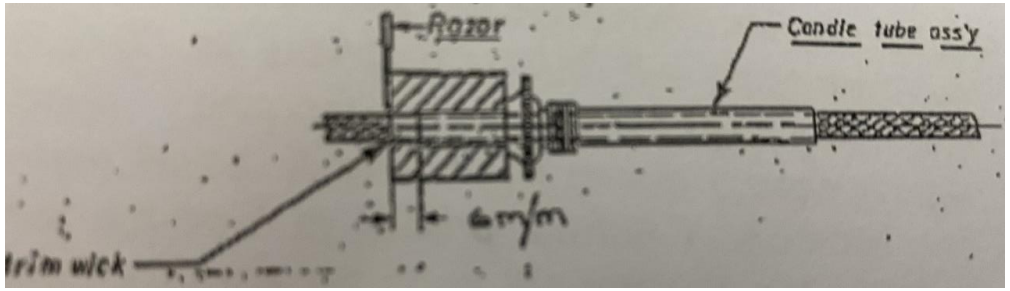
1. Be sure to read the safety and hazard warnings before operating this instrument.
2. Soak the extracted wick in a beaker of the fuel sample to be tested.



3- To insert the wick into the candle tube assembly, move the movable slide collar on the wick insertion tool over the three metal prongs causing them to slide together. Then, install the 6 mm depth gauge (K27065-0-1) onto the short end of the candle tube assembly as shown in the following diagram. Insert wick insertion tool (K27060) into the short end of the candle tube assembly and push the candle down on the wick insertion until the three metal prongs open. Insert the soaked wick into the open metal prongs and slowly pull the candle tube assembly and depth gauge out of the wick insertion.

Smoke Point Lamp Operation

4- Refer to the following figure to trim the wick. Using the razor blade, cut the wick flush with the depth gauge and remove the gauge from the candle.



5- Fill the candle with approximately 10 to 15 mL of extra fuel to ensure the wick will burn for a substantial amount of time if necessary, Pay attention while pouring the extra fuel into the candle because there is an overflow pipe. If fuel is poured over the overflow pipe, then it will leak out of the bottom. This can be avoided by knowing the location of the overflow pipe and pouring in the opposite direction.

6- Place the long end of the candle tube assembly (with the wick already inserted) into the candle. Rotate the candle tube assembly in the opposite direction of tightening (approximately 5 rotations) to allow the wick to be positioned straight inside the candle. Then tighten the candle tube assembly.

7- After inserting the wick into the candle, open the glass door simply by pulling open. Insert the candle into the candle holder with a twisting motion, thus ensuring that the wick can turn freely and will not become stuck in between the gallery and the candle tube. Then lock into position.

8- Attach the sighting device to the top of the chimney. Adjust the sighting device by turning the knurl nut to aid in measuring the height of the flame.

9- Light the candle and close the glass door.

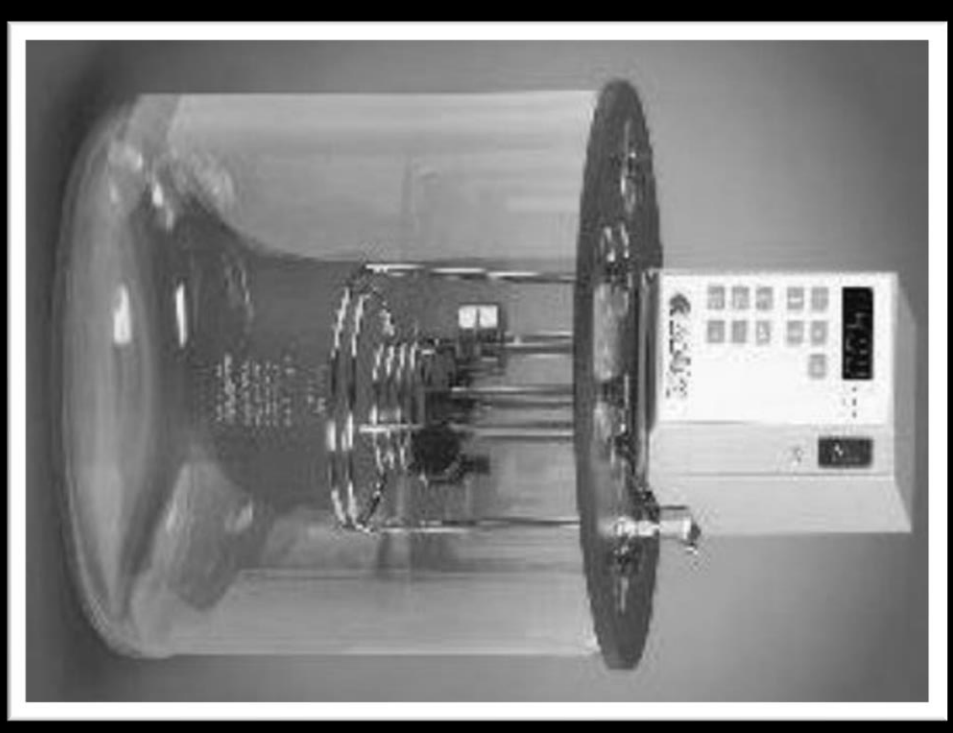
10- After burning the candle is raised until a smoky tail appears, then the candle is lowered slowly through several stages of flame appear once.

11- Determined the maximum height of flame that can be achieved without smoking.

Sulfur In Oil Analyzer

sulfur content in petrochemical products, especially in oil products (such as gasoline, diesel, lubricating oil, etc.) has become the focus of environmental protection worldwide. When oil with high sulfur content is combusted, carbon dioxide will be generated and then be cooled to generate water. Water and sulfur are combined to generate sulfurous acid or sulfuric acid which will corrode the engine. When large amount of sulfur oxides get into air, air pollution can be caused, including forming acid rain. Excessive sulfate will be formed after the sulfur in gasoline is combusted, and will be attached to the precious metal coating surface of the catalyst, resulting in excessive emissions of automobile exhaust. Therefore, it is very important to reduce the sulfur content in gasoline. The instrument complies with national standards including ASTM 7039.

KV1000 Kinematic Viscosity Bath



The KV1000 Kinematic Viscosity Bath is for performing kinematic viscosity tests with glass capillary viscometers according to the ASTM D445 test method and related test specifications.

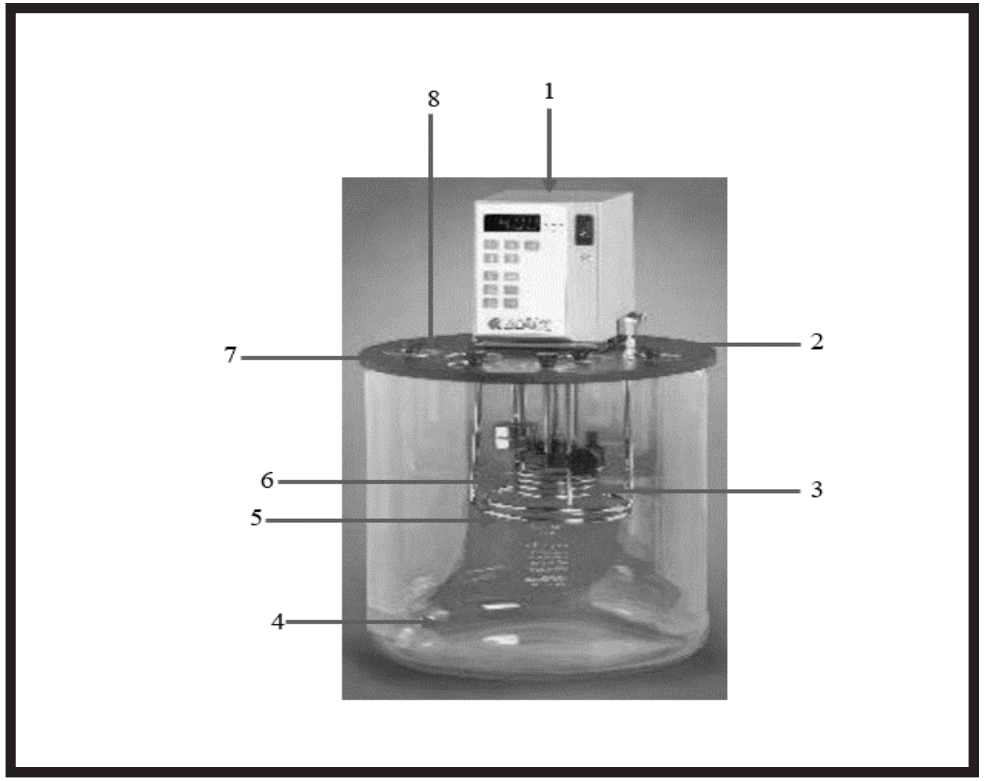
kinematic viscosity (KV): is absolute viscosity of a fluid divided by its density at the same temperature of measurement. Unit mm^2/s or centiStokes (cSt). To determine kinematic viscosity, a fixed volume of the test fluid is allowed to flow through a calibrated capillary tube (**viscometer**) that is held at a closely controlled temperature. In order to calculate viscosity index, Kinematic viscosity of oil or lubricants should be measured at specified temperature 40 and 100 ^oC.

Viscometer

Digital constant temperature bath for kinematic viscosity testing of petroleum products. Accommodates six round 2" (51mm) dia. viscometer holders.

The KV1000 Kinematic Viscosity Bath apparatus consists of:

- Viscometer
- Viscometer holder
- Temperature control bath
- Temperature measuring device
- Timing device



Instrument descriptions and controls

Temperature controller

1. Coolant Outlet/Inlet
2. Temperature Sensor
3. Pyrex Glass Bath
4. Cooling Coil
5. Heating Coil
6. Viscometer Port
7. Thermometer/Thermocouple Port

Ventilation:

A fume hood or exhaust system is required when operating the unit. Flammable vapors and/or steam are generated during accumulate. A canopy-style hood may be used if the height from the top of the unit to the canopy is 5 feet or less. The exhaust blower should have a rating of 1000 C.F.M or greater.

KV1000 Kinematic Viscosity Bath Operation

Bath:

1. Ensure the Borosilicate glass jar is not cracked or broken. Do not use the jar if there is any damage.
2. Fill the bath with the appropriate heat transfer fluid based upon the testing temperature.
3. Fill the bath with the medium to 2” (5 cm) from the top of the bath to allow room for fluid expansion.
4. This will provide the proper depth for immersing the viscometers and allow for thermal expansion.

Temperature Controller Operation

Setting the Safety Set Point

1. The Safety Set feature automatically disconnects controller power to the heater and pump in the event that the reservoir liquid level drops too low or the controller fails.
2. The Safety Set is user adjustable between approximately 40 and 210 ̊C.
3. It should be set at least 5 ̊C higher than the Software High Limit temperature.
4. Use a flat blade screwdriver to rotate the Safety Set Indicator Knob to the desired temperature.
5. Do not force the Knob beyond the stops at either end of the temperature value range.
6. Once the Safety Set temperature has been set, turn power to the controller ON by pressing the Power Switch on the front of the controller.
7. The pump will begin operating, the display will flash the current temperature set point (tx.xx), the ̊C LED will light, and the current bath temperature will appear on this display.
8. Pump speed selection is made using the Pump Speed Selection Switch on the rear of the Controller.

Selecting Temperature Units

The control set point and actual bath temperature may be displayed in either ̊C or ̊F. The factory-default is ̊C.

1- To change from ̊C to ̊F, place the Circuit Breaker/Power Switch on the rear of the Controller in the OFF position and then press and hold the **P2** Button while turning the power back ON.

2- To change from ̊F to ̊C, place the Circuit Breaker/Power Switch on the rear of the Controller in the OFF position and then press and hold the **P3** Button while turning the power back ON.

KV1000 Kinematic Viscosity Bath

General Procedure for Kinematic Viscosity

1. Adjust and maintain the viscometer bath at the required test temperature.
2. Thermometer shall be held in an upright position under the same conditions of immersion as when calibrated.
3. In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificate be used.
4. They should be viewed with a lens assembly giving approximately five times magnification and be arranged to eliminate parallax errors.
5. Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity.
6. Then a viscometer bath is maintained at a required test temperature.
7. The viscometer is charged with the fluid sample and placed in the bath where it is maintained until its temperature reaches the test temperature.
8. Once it reaches the test temperature the level of the sample in the viscometer is marked.
9. The head level is adjusted to a position (7 mm for instance) that is above the first mark.
10. The time taken by the fluid to reach the second marked position is measured.

Refractometer

The refractometers conforms to the requirements of ASTM D1218 and is used widely throughout the petroleum industry.

Octane Number (RON)

Octane/Cetane analyser. Research (RON), motor (MON) method. Anti Knocking Index AKI (Pump octane number PON).

Lab Scale Digital Analytical Electronic Balance Laboratory Weighing Balance

Weighing a Chemical

1. Always ensure that the balance is clean and is of an appropriate type for the amount that you will weigh.
2. It is not sensible to weigh 100 mg on a 5 kg balance!
3. Tare the balance with your receptacle on it and add your compound until you obtain the desired weight.
4. If it is not possible to weigh directly into the reaction flask, use an alternative container (e.g. a sample vail for liquid or a weighing boat for solids) and then transfer the substance to the reaction flask.
5. Do not weigh onto filter paper.
6. If you have pre-weighed a flask and then weigh it containing a compound, use the same balance for each weighing.
7. You must clean-up any spillages on the balance or bench.

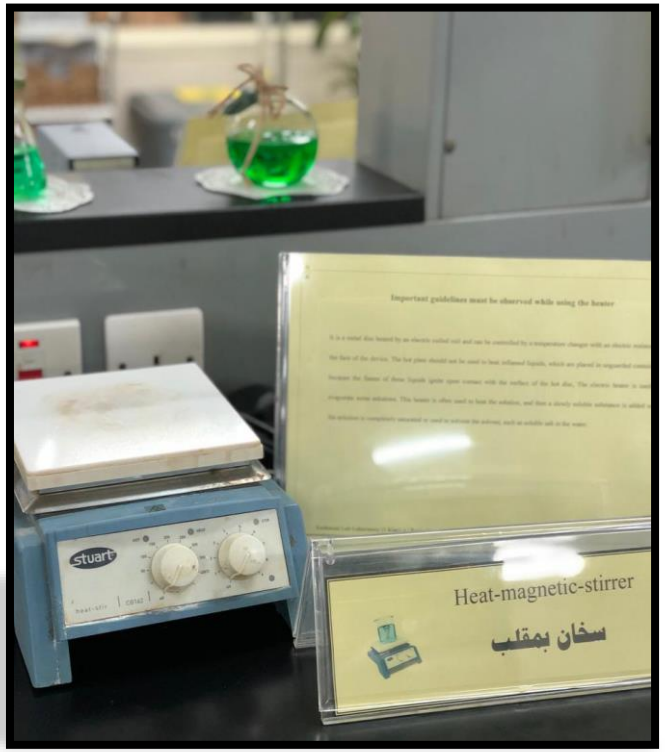
Melting Point Apparatus

Determining a melting point

1. One way of characterising a pure compound is through a melting determination.
2. Place a small amount of sample on a watch glass, press the open end of the melting point tube into the sample (picking up perhaps 2 mm worth of sample), then tap the closed end on the bench until the sample drops to the bottom.
3. Using a melting point apparatus that is cold, insert the sample tube into the holder and heat, watching the sample all of the time through the viewer.
4. As soon as the sample starts to melt, turn off the heating, and note the temperature range over which it melted.
5. If time permits you could repeat the measurement with slower heating in the vicinity of the melting point.
6. Melting points are usually reported as a range, e.g. X – Y ℃.
7. Remember to include the recrystallisation solvent, e.g. mp 54 – 56 ℃ (from dichloromethane).

Important guidelines must be observed while using the heater

It is a metal disc heated by an electric coiled coil and can be controlled by a temperature changer with an electric resistor on the face of the device. The hot plate should not be used to heat inflamed liquids, which are placed in unguarded containers because the fumes of these liquids ignite upon contact with the surface of the hot disc, The electric heater is used to evaporate some solutions. This heater is often used to heat the solution, and then a slowly soluble substance is added until the solution is completely saturated or used to solvent the solvent, such as soluble salt in the water.



Laboratory water bath

A water bath is laboratory equipment made from a container filled with heated water. It is used to incubate samples in water at a constant temperature over a long period of time. Most water baths have a digital or an analogue interface to allow users to set a desired temperature, but some water baths have their temperature controlled by a current passing through a reader. Utilisations include warming of reagents, melting of substrates or incubation of cell cultures. It is also used to enable certain chemical reactions to occur at high temperature. Water bath is a preferred heat source for heating flammable chemicals instead of an open flame to prevent ignition. Different types of water baths are used depending on application. For all water baths, it can be used up to 99.9 °C. When temperature is above 100 °C, alternative methods such as oil bath, silicone bath or sand bath may be used.

Types of water bath

Circulating water baths

Circulating water baths (also called *stirrers*) are ideal for applications when temperature uniformity and consistency are critical, such as enzymatic and serologic experiments. Water is thoroughly circulated throughout the bath resulting in a more uniform temperature.

Non-circulating water baths

This type of water bath relies primarily on convection instead of water being uniformly heated. Therefore, it is less accurate in terms of temperature control. In addition, there are add-ons that provide stirring to non-circulating water baths to create more uniform heat transfer.

Shaking water baths

This type of water bath has extra control for shaking, which moves liquids around. This shaking feature can be turned on or off. In microbiological practices, constant shaking allows liquid-grown cell cultures grown to constantly mix with the air.

Some key benefits of shaking water bath are user-friendly operation via keypad, convenient bath drains, adjustable shaking frequencies, bright LED-display, optional lift-up bath cover, power switch integrated in keypad and warning and cut-off protection for low/high temperature.

Precautions

- Use with caution.
- It is not recommended to use water bath with moisture sensitive or pyrophoric reactions. Do not heat a bath fluid above its flash point.
- Water level should be regularly monitored, and filled with distilled water only. This is required to prevent salts from depositing on the heater.
- Disinfectants can be added to prevent growth of organisms.
- Raise the temperature to 90 °C or higher to once a week for half an hour for the purpose of decontamination.
- Markers tend to come off easily in water baths. Use water resistant ones.
- If application involves liquids that give off fumes, it is recommended to operate water bath in fume hood or in a well ventilated area.
- The cover is closed to prevent evaporation and to help reaching high temperatures.
- Set up on a steady surface away from flammable materials.

Flame Photometers

The Jenway flame photometer is a low temperature, single channel meter designed for the routine determinations of Sodium (Na), Potassium (K), Lithium (Li), Barium (Ba) and Calcium (Ca). The instrument is fitted with automatic flame failure detection for user safety. An air compressor and gas .regulator are required to operate the unit

Features

Sodium, Potassium, Calcium, Barium and Lithium filters are included and factory installed

Electronic flame failure detection

Fine and coarse sensitivity controls

Operates on propane, butane, natural gas or L.P.G. supplies

Recorder output

Easily maintained

Economically priced

Warranty: 2 years

Flame Photometers

Specifications

Limits of Detection:

Na ó 0.2 ppm

K ó 0.2 ppm

Li ó 0.25 ppm

Ca ó 15 ppm

Ba ó 30 ppm

Reproducibility: ó1% Coefficient of Variation for 20 consecutive samples using 10 ppm Na set to read 50.0

Linearity: Better than 2% when concentration of 3 ppm Na and K and 5 ppm Li are set to read 100

Specificity: Interference from Na, K and Li equal in concentration to test element will be less than 0.5%

Recorder Output: Nominal 1.00 V for a reading of 100.0

Services: Electrical: 90-125 V or 190-250 V @ 50/60 Hz

Air: Moisture and oil free 6 litre/minute of 1 kg/cm2 (14 psi(

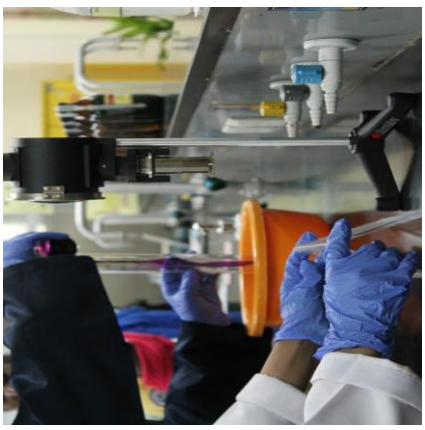
Fuel: Propane, Butane, Natural Gas or L.P.G.

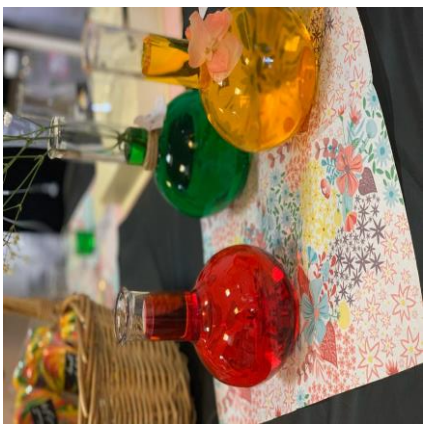
Flame Photometers

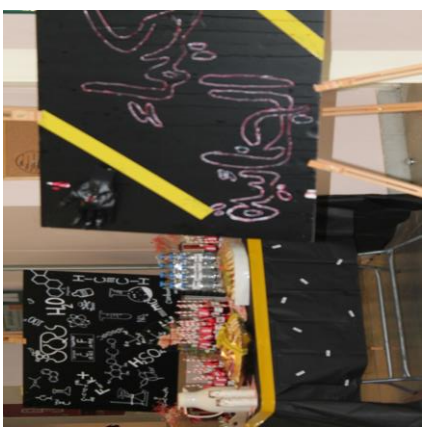
Activities in Chemistry Lab

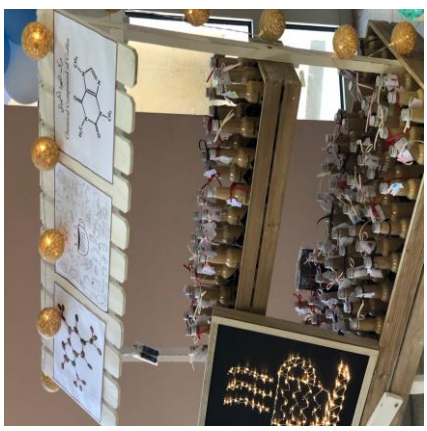
Activities in Chemistry

Activities in Chemistry Lab









Thank you for
your interest!