CHAPTER III

EXPERIMENTAL SECTION

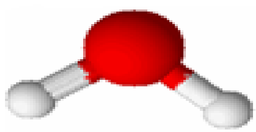
III.1. NAME, STRUCTURE, PHYSICAL AND CHEMICAL PROPERTIES, PURIFICATION AND APPLICATIONS OF THE CHEMICALS USED IN THE RESEARCH WORK

III.1.1. SOLVENTS

The details of the aqueous and non-aqueous solvents used in the research work are given below:

Water (H_2O):

Water is an omnipresent chemical substance is composed of hydrogen and oxygen and is essential for all known forms of life. In typical usage, water refers only to its liquid form or state, but the substance also exists as solid state, ice, and a gaseous state, water vapour or steam. Water is a good solvent and is often referred to as the universal solvent.



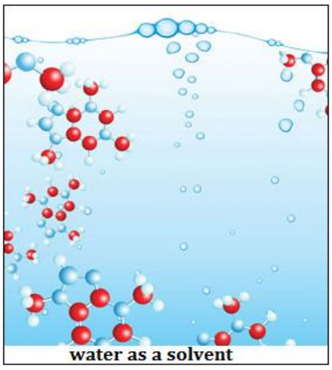
WATER

State	*: Liquid*
Molecular Formula	*: H_20*
Molecular Weight	*: 18.02 $g{\cdot}mol^{-1}$*
Density	*:0.99713 $g{\cdot}cm^3$*
Viscosity	*:0.891 mP·s*
Refractive Index	*:1.3333*
Dielectric Constant	*:78.35 at 298.15K*

Source: Distilled water, distilled from fractional distillation method in Lab.

Purification: Water was first deionised and then distilled in an all glass distilling set along with alkaline $KMnO_4$ solution to remove any organic matter therein. The doubly distilled water was finally distilled using an all glass distilling set. Precautions were taken to prevent contamination from CO_2 and other impurities. The triply distilled water had specific conductance less than 1×10^{-6} $S{\cdot}cm^{-1}$.

Application: Water is widely used in chemical reactions as a solvent or reactant and less commonly as a solute or catalyst. In inorganic reactions, water is a common solvent, dissolving many ionic compounds. Supercritical water has recently been a topic of research. Oxygen saturated supercritical water combusts organic pollutants efficiently. It easily forms hydrogen bond with other molecules and has suitable polarity to dissolve a numerous number of molecules and hence, it is called the universal solvent. Water is the main component of life in this earth. Not only a high percentage of living bodies, both plants and animals are found in water, all life on earth is thought to have arisen from water and the bodies of all living organisms are composed mostly of water. About 70 to 90 percent of all organic substance is water. The chemical reactions in all plants and animals that support life take place in water medium. Water not only provides the medium to make these life sustaining reactions possible, but water itself is often an important reactant or product of all these reactions. In short, the chemistry of life is nothing but the "water chemistry."



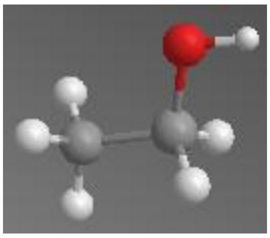
water as a solvent

Ethanol (EtOH):

Ethanol, also known as methyl alcohol, wood alcohol, wood naphtha or wood spirits, is the simplest alcohol, and is a light, volatile, colourless, flammable, liquid with a distinctive odour that is very similar to but slightly sweeter than ethanol (drinking alcohol).

Source: Merck, India.

Purification: It was passed through Linde Å molecular sieves and then distilled [1]



Ethanol

Appearance	*:Colourless Liquid*
Molecular Formula	*:C_2H_6O*
Molecular Weight	*:46.07 $g{\cdot}mol^{-1}$*
Density (30^0C)	*:0.8029 $g{\cdot}cm^3$*
Viscosity	*:0.948mP·s (303.15K)*
Refractive Index	*:1.361(298.15 K)*
Dielectric Constant	*: 24.3 (298.15 K)*

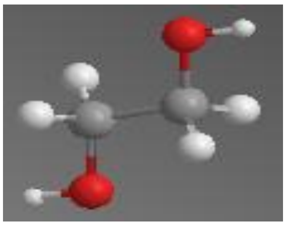
Application: The largest use of methyl alcohol by far is in making other chemicals. Methanol is a traditional denaturant for ethanol, thus giving the term methylated spirit. Methanol is also used as a solvent, and as an antifreeze in pipelines. In some waste water treatment plants, a small amount of methanol is added to waste water to provide a food source of carbon for the denitrifying bacteria, which converts nitrates to nitrogen to reduce the denitrification of sensitive aquifers. Methanol is used on a limited basis to fuel internal combustion engines. Methanol is also useful as an energy carrier. It is easier to store than hydrogen, burns cleaner than fossil fuels, and is biodegradable.

Ethylene glycol: ($C_2H_6O_2$):

Ethylene glycol is a hygroscopic liquid which has no colour, no odour, low-volatility and low-viscosity. It is completely miscible with water and many organic liquids. The hydroxyl groups on glycols undergo the usual alcohol chemistry, giving a wide variety of possible derivatives.

Source: Merck, India.

Purification: It was dried with anhydrous $CaSO_4$ and distilled under vacuum. The distillate was passed through linde type 4 Å molecular sieves. [1] [2]



Ethylene glycol

Appearance:	*Liquid*
Molecular Formula:	$C_2H_6O_2$
Molecular Weight:	*62.07 g/mol*
Boiling Point:	*197.3^{0}C*
Melting Point:	*-12.9^{0}C*
Dielectric Constant:	*40.97 at 25^{0}C*

Methanol (MeOH):

Methanol is the first alcohol of its homologues series. It is named as methyl alcohol or methanol, wood naphtha or wood alcohol. It is light, volatile, flammable, Colourless which has a distinctive odour.

Source: Merck, India.

Purification: It was passed through Linde 4Å molecular sieves and then distilled [1].

Application: The largest use of methyl alcohol by far is in making other chemicals. About 40% of methanol is converted to

Methanol

Appearance:	*Liquid*
Molecular Formula:	CH_3OH
Molecular Weight:	*32.04 g/mol*
Boiling Point:	*176 K*
Melting Point:	*337.8 K*
Dielectric Constant:	*32.70 at 298.15 K*

formaldehyde, and from there into products as diverse as plastics, plywood, paints, explosives, and permanent press textiles. Methanol is a usual denaturant for ethanol and hence it is named as methylated spirit.

Methanol is well known as solvent and another wide use is antifreeze substance in pipelines. In some waste water treatment plants, a small amount of methanol is added to waste water to provide a food source of carbon for the denitrifying bacteria, which converts nitrates to nitrogen to reduce the de nitrification of sensitive aquifers. Methanol is used on a limited basis to fuel internal combustion engines. Methanol is also useful as an energy carrier. It is easier to store than hydrogen, burns cleaner than fossil fuels, and is biodegradable.

III.1.2 ELECTROLYTES AND NON-ELECTROLYTES

The electrolytes ionic liquids, and non-electrolytes amino acids, respectively, and other chemicals than these two categories that are used in the research work have been describing follow:

III.1.2.1 Ionic Liquids

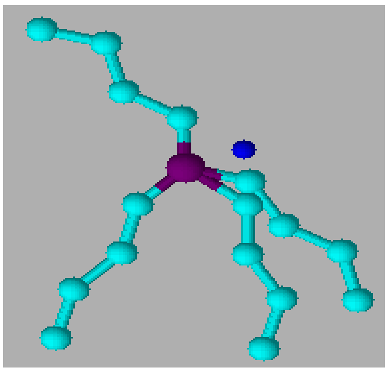
Tetrabutylphosphonium methanesulfonate:

Tetrabutylphosphonium methanesulfonate is an ionic liquid with molecular formula $C_{16}H_{36}PCH_3SO_3$. Phosphonium-based ILs is less toxic, thermally more stable, and readily available in bulk quantities and less expensive.

Source: Sigma-Aldrich, Germany

Purification: Used as purchased

Application: Phosphonium based ionic liquids are now days used as extraction solvents, chemical synthesis solvents, electrolytes in batteries and super-capacitors, and in corrosion protection Tetrabutylphosphonium methanesulfonate



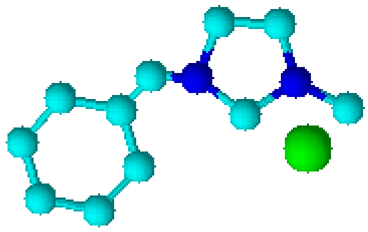
Tetrabutylphosphonium methanesulfonate

Appearance:	*White powder*
Molecular Formula:	*$C_{16}H_{36}PCH_3SO_3$*
Molecular Weight:	*354.53 g/mol*
Melting Point:	*59-62^{0}C*

ionic liquid is now appearing in applications as phase transfer catalysts , organic synthesis and electrochemical media [3].

1-Benzyl-3-methylimidazolium chlorides:

It is an imidazolium based compound compounds. The nitrogen atom is joined to benzyl group which is hydrophobic in nature. They show a variety of physical, chemical, and biological properties and most compounds are soluble in water and strong electrolytes.



1-Benzyl-3-methylimidazolium chlorides

Appearance:	*Crystalline*
Molecular Formula:	$C_{11}H_{13}ClN_2$
Molecular Weight:	*208.69 g·mol-1*

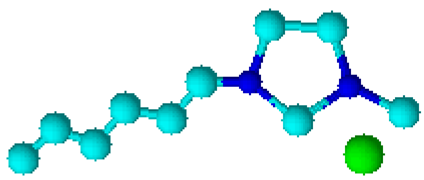
Source: Sigma Aldrich, Germany

Purification: Used as purchased. The purity of the chemical is >99.0%

Application: The ionic liquid 1-Benzyl-3-methylimidazolium chlorides used for synthesis, antistatic Agent, detergent sanitisers, softner for textiles and paper products, phase transfer catalyst, antimicrobials, disinfection agents And sanitizers, Slimicidal Agents, Algaecide, Emulsifying Agents, Pigment Dispersers

1-hexyl-3-methylimidazolium chloride:

1-hexyl-3-methylimidazolium chloride is an imidazolium based ionic liquid with a hexyl group a methyl group attached to the two active nitrogen atoms.



1-hexyl-3-methylimidazolium chloride

Appearance:	*Pale yellow liquid*
Molecular Formula:	$C_{10}H_{19}ClN_2$
Molecular Weight:	*202.72 g·mol*$^{-1}$
Relative Density:	*Not available*

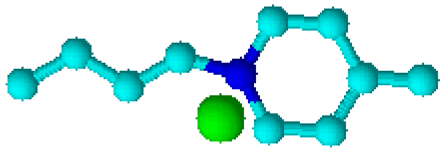
Source: Sigma Aldrich, Germany

Purification: Used as purchased. The purity of the chemical is >98.50%

Application: 1-hexyl-3-methylimidazolium chloride is widely used in industry as it can be recycled infinitely and amenability to solvation at room temperature, making them excellent green solvents.

1-butyl-4-methylpyridinium hexafluorophosphate:

1-butyl-4-methylpyridinium hexafluorophosphate is a pyridinium based ionic liquid, of molecular formula $C_{10}H_{16}F_6NP$, containing methyl, and butyl group with an active nitrogen atom. It exists in solid form.



1-butyl-4-methylpyridinium hexafluorophosphate

Appearance:	*Solid*
Molecular Formula:	$C_{10}H_{16}F_6NP$
Molecular Weight:	*295.20* $g \cdot mol^{-1}$
Melting point:	*318.15 K*

Source: Sigma Aldrich, Germany

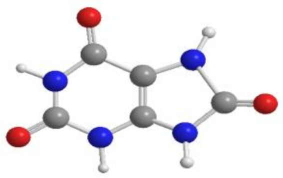
Purification: Used as purchased. The purity of the chemical is >99.0%

Application:

1-butyl-4-methylpyridinium hexafluorophosphate is used as solvents for polymerchemistry. The ionic liquid is good examples of neoteric solvents, new types of solvents,or older materials that are finding new applications as solvents, which is envi nisms, used as recyclable solvents for organic reactions and separation processes, lubricating fluids, heat transfer fluids for processing biomass and electrically conductive liquids as electrochemical device in the field of electrochemistry (batteries andsolar cells) and so forth. In the modern technology, industry, and also in academic researchfield, the vast application is frequently increases.ronmentally friendly (or eco-friendly) because they are less hazardous for human body as well as less toxic for living organisms, used as recyclable solvents for organic reactions and separation processes, lubricating fluids, heat transfer fluids for processing biomass and electrically conductive liquids as electrochemical device in the field of electrochemistry (batteries andsolar cells) and so forth. In the modern technology, industry, and also in academic researchfield, the vast application is frequently increases.

Uric acid:

The molecular structure of uric acid is the combination of carbon, nitrogen, oxygen and hydrogen with the formula $C_5H_4N_4O_3$. It forms ions and salts known as urates and acid urates, such as ammonium acid urate. Uric acid is produced by the metabolic break of purine nucleotides, and is found normally in urine. High blood concentrations of uric acid can lead to gout and are associated with other medical conditions including diabetes and the formation of ammonium acid urate kidney stones.



Uric acid

Appearance	*White crystals*
Molecular Formula	$C_5H_4N_4O_3$
Molecular Weight	*168.11 g·mol^{-1}*
Melting point	*573.15 K*

The molecular structure of uric acid is the combination of carbon, nitrogen, oxygen and hydrogen with the formula $C_5H_4N_4O_3$. It forms ions and salts known as urates and acid urates, such as ammonium acid urate. Uric acid is produced by the metabolic break of purine nucleotides, and is found normally in urine. High blood concentrations of uric acid can lead to gout and are associated with other medical conditions including diabetes and the formation of ammonium acid urate kidney stones.

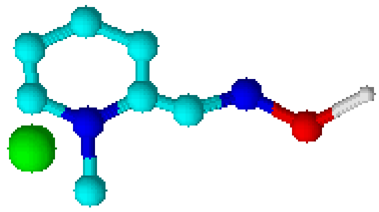
Source: Sigma Aldrich, Germany

Purification: Used as purchased. The purity of the chemical is >99.0%

III.1.2.2 Drug molecules

Pyridine-2-aldoxime methochloride:

Parlidoxime is an important drug and act as a nerve agent for the treatment of organophosphorus poisoning in the nervous system. Parlidoxime or 2-pyridine aldoximemethochloride (2-PAM) is an oxime based drug molecule. It has a suitable charged pyridine moiety and an oxime part resides at C-2 position of pyridine ring.



Appearance	*: white crystaline solid*
Molecular Formula	: $C_7H_9N_2O\,^{\cdot}Cl$
Molecular Weight	*172.61 g·mol^{-1}*
Melting Point	*: 503.15 K*

Source: Sigma Aldrich, Germany

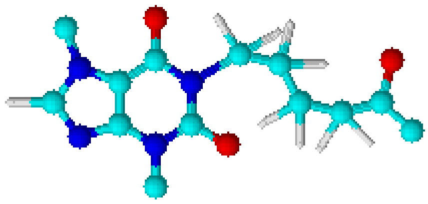
Purification: Used as purchased. The purity of chemical is more than 95.0% pure.

Pentoxifylline:

Pentoxifylline, $C_{13}H_{18}N_4O_3$, (3,7-dimethyl-1-(5-oxohexyl)purine-2,6-dione), (PTX), is a nonselective methyl xanthine phosphodiesterase inhibitor, which improves the blood flow by decreasing viscosity. PTX, at the position 3, the C atom contains aliphatic chain with polar electrophilic carbonyl group.

Source: Sigma Aldrich, Germany.

Purification: Used as purchased. The purity of the chemical is >98.0%.



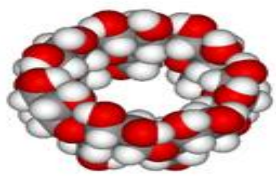
Pentoxifylline

Appearance	*:Amorphous*
Molecular Formula	*: $C_{13}H_{18}N_4O_3$*
Molecular Weight	*: 278.31 g·mol-1*
Solubility in water	*: 43mg.mol^{-1}*

III.1.2.3 Non-electrolytes

α-Cyclodextrin (α-CD):

α-Cyclodextrin is a cyclic oligosaccharide composed of 6 glucose groups. This is white amorphous solid with a cylinder like molecular structure.

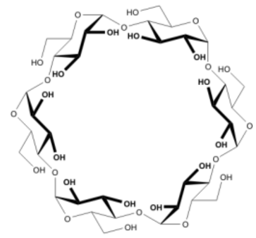


The structural arrangement makes it versatile in different fields. The properties are widely used in industry for various purposes.

Source: Sigma Aldrich, Germany.

Purification: Used as parched. The purity is 99.98%.

Application: α-Cyclodextrin is a new substance with high solubility in water and which has wide application in medicinal chemistry, food processing industry. Moreover, it is extensively used in modification of cosmetics, food stuffs etc.; whose function is to improve stability, solubility and good smell. In the production of medicine, it can strengthen the stability of medicine without being oxidized and resolving. On the other hand, it can improve the solubility. And the effect on living of medicine, lower the toxic and side-effect of medicine and cover the strange and bad smell. In the food industry, it is used to cover strange and bad smell of food, improve the stability of perfume and the *α-Cyclodextrin*



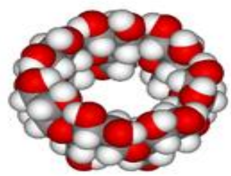
α-Cyclodextrin

condiment and keep food dry or wet at will. α-CD with a cavity diameter of 4.7-5.3Å, is of the good interest because it is easily available in market and its cavity size allows to encapsulate many common guest moieties like hormones, vitamins, and many compounds. This capability has also been of assistance for different applications in medicines, cosmetics, food technology, pharmaceutical, and chemical industries as well as in agriculture and environmental engineering as an encapsulating agent to protect sensitive molecules in hostile environment.

Appearance	*:Crystalline Powder*
Molecular Formula	$C_{42}H_{70}O_{35}$
Molecular Weight	*:1134.98 g·mol^{-1}*
Melting Point	*:563.15-573.15 K*
Boiling Point	*:1814.33 K*
Relative Density	*:1.44 g·cm^{-3} at 20°C*
Refractive Index	*:1.59 (n_D^{20})*

β-Cyclodextrin (β-CD):

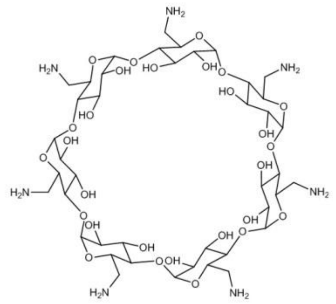
β-Cyclodextrin is white amorphous solid compound composed of 7 glucose groups having a cylinder like molecular structure. The function of β- Cyclodextrin depends on its molecular structure which can be easy to integrate other materials. That feature is applied widely in industry



Source: Sigma Aldrich, Germany.

Purification: Used as parched. The purity is 99.98%.

Application: β-Cyclodextrin is a new stuff which can be widely applied in production of medicine and food. It can be applied widely in production of medicine, food and cosmetics, whose function is to improve stability, solubility and good smelled. In the production of medicine, it can strengthen the stability of medicine without being oxidized and resolving. On the other hand, it can improve the solubility. And the effect on living of medicine, lower the toxic and side-effect of medicine and cover the strange and bad smell. In the production of food, it can mainly cover strange and bad smell of food, improve the stability of perfume and condiment and keep food dry or wet at will. β-CD with a cavity diameter of 6.4-7.5 Å, is the most interest because its cavity size allows for the best special fit for many common guest moieties. For this reason, β-cyclodextrin is widely used as host in the host-guest chemistry with various biologically active molecules such as hormones, vitamins, drug molecules and various compounds commonly used in tissue and cell-culture applications. This capability has also been of assistance for different applications in medicines, cosmetics, food technology, pharmaceutical, and chemical industries as well as in agriculture and environmental engineering as an encapsulating agent to protect sensitive molecules in hostile environment.



β-Cyclodextrin

Appearance	*:Crystalline Powder*
Molecular Formula	*:$C_{42}H_{70}O_{35}$*
Molecular Weight	*:1134.98 $g{\cdot}mol^{-1}$*
Melting Point	*:563.15-573.15 K*
Boiling Point	*:1814.33 K*
Relative Density	*:1.44 $g{\cdot}cm^{-3}$ at 20°C*
Refractive Index	*:1.59 (n_D^{20})*

III. 2 EXPERIMENTAL METHODS

III.2.1 PREPARATION OF SOLUTIONS

A sock solution for each salt was prepared by mass (digital electronic analytical balance, Mettler Toledo, AG 285, Switzerland), and the working solutions were obtained by mass dilution of the stock solution. The uncertainty of concentration (molarity or molality) of different working solutions was evaluated to be ±0.0002.

III.2.2. PREPARATION OF MULTICOMPONENT LIQUID MIXTURES

The binary and multicomponent liquid mixtures can be prepared by any one of the methods discussed below:

(a) Mole fraction
(b) Weight fraction
(c) Volume fraction

(a) Mole fraction: The mole fraction (x_i) of the multicomponent liquid mixtures can be prepared using the following relation:

$$x_i = \frac{(w_i / M_i)}{\sum_{i=1}^{n}(w_i / M_i)}$$

Where, w_i, and M_i are weight and molecular weight of i^{th} component, respectively. The values of *i*depends on the number of components involved in the formation of a mixture.

(b) Weight fraction: The mole fraction (w_i) of the multicomponent liquid mixtures can be prepared using the following relation:

$$w_i = \frac{(x_i / M_i)}{\sum_{i=1}^{n}(x_i M_i)}$$

(c) Volume fraction: The volume fraction (ϕ_i) of the multicomponent liquid mixtures can be prepared by following employing three methods:

i. ***Using volume:*** The volume fraction (ϕ_i) of the multicomponent liquid mixtures can be prepared by following relation

$$\phi_i = \frac{V_i}{\sum_{i=1}^{n} V_i}$$

Where, V_i, is the volume of pure liquid *i*.

ii. ***Using molar volume:*** The volume fraction (ϕ_i^l) of the multicomponent liquid mixtures can be prepared by following relation

$$\phi_i^l = \frac{x_i V_{mi}}{\sum_{i=1}^{n}(x_i V_{mi})}$$

Where, V_{mi} is the molar volume of pure liquid *i*.

iii. ***Using excess volume:*** The volume fraction (ϕ_i^{ex}) of the multicomponent liquid mixtures can be prepared by following relation

$$\phi_i^{ex} = \frac{x_i V_i}{\sum_{i=1}^{n}(x_i V_i) + V^E}$$

Where, V^E is the excess volume of the liquid mixture.

III.2.3 MEASUREMENTS OF EXPERIMENTAL PROPERTIES

III.2.3.1 MASS MEASUREMENT

Using digital electronic analytical balance Mettler Toledo, AG 285, Switzerland, mass in different cases were measured.

It can measure mass with a very high precision and accuracy. The weighing pot is of high accuracy and precision (0.0001g) is kept inside a glass enclosure with sliding doors to protect from dust and air currents.

Instrument Specification:

Readability	*: 0.1 mg/ 0.01mg*
Maximum capacity	*: 210 g/81g/41g*
Taring range	*: 0. . . . 210 g*
Repeatability	*: 0.1 mg/ 0.05 mg*
Linearity	*: ±0.2 mg/±0.1 mg*
Stabilization time	*: 3 s/ 15 s*
Adjustment with external weights	*:200 g*
Sensitivity	*: ±0.003%*
Display	*: LCD*
Interface	*: Local CAN universal interface*
Weighing	*: Φ 85 mm, stainless steel*
Effective height above pan	*: 240 mm*
Dimensions(w/d/h)	*: 205×330×310 mm*
Net wt./with packaging	*: 4.9 kg/7.25 kg*

III.2.3.2 CONDUCTIVITY MEASUREMENT

Conductivity measurement was done using Systronics Conductivity TDS meter-308. It can provide both automatic and manual temperature compensation. Systronic Conductivity-TDS meter 308 is a microprocessor based instrument used for measuring specific conductivity of the solution. It can provide both automatic and manual temperature compensation. Provision for storing the cell constant and calibrating solution type, is provided with the help of battery back-up. This data can be further used for measuring the conductivity of an unknown solution, without re calibrating the instrument even after switching it off.

Systronic-308 Conductivity Bridge

The conductance measurements were carried out on this conductivity bridge of accuracy ±0.01%, using a dip-type immersion conductivity cell, CD-10 having a cell constant of approximately (0.1±0.001) cm^{-1}. Measurements were made in a thermostate water bath maintained at T = (298.15 ± 0.01) K. The cell was calibrated by the method proposed by Lind et al [4] and cell constant was measured based on 0.01 M aqueous KCl solution [5]. During the conductance measurements, cell constant was maintained within the range 1.10–1.12 cm^{-1}. The conductance data were reported at a frequency of 1 kHz with the accuracy of ±0.3%. The conductivity cell was sealed to the side of a 500 cm^3 conical flask closed by a ground glass fitted with a side arm through which dry and pure nitrogen gas was passed to stop admittance of air into the cell during the addition of solvent or solution. The measurements were made in a thermostatic water bath maintained at the required temperature with an accuracy of ± 0.01 K by means of mercury in glass thermoregulator [6].

Instrument Specifications

Frequency	:100 Hz or 1 KHz Automatic
Range	: 0.1 µS to 100 mS. (6 decadic range)
Accuracy	: ±1% of F.S. ±1 digit
Resolution	: 0.001 µs
TDS	
Range	:0.1 ppm to 100 ppt. (6 decadic range)
Accuracy	: ±1% of F.S. ±1 digit
Temperature	
Range	: $0^{o}C$ to $100^{o}C$ (Auto/Manual)
Accuracy	: ± 0.2 ^{o}C ±1 digit
Resolution	: 0.1 ^{o}C
Cell Constant	: Acceptable from 0.1 to 5.0
Auto Temp. Compensation	: $0^{o}C$ to $100^{o}C$ with PT 100 sensor
Manual Temp. Compensation	: $0^{o}C$ to $60^{o}C$ user selectable
Conductivity temp. Co-efficient	: 0.0% to 9.9% user selectable
Display	: 7 digits, 7 segment LEDs (3 digits for TEMP/TEMPCO 4 digits for Conductivity/TDS) With automatic decimal point selection
TDS-factor	: 0.00 to 9.99 user selectable
Printer Port	: Epson compatible 80 Column Dot Matrix
Power	: 230V AC, ±10%, 50 Hz
Dimensions	: 250(W)× 205(D)× 75(H)
Weight	: 1.25 Kg (Approx.)
Accessories	: i) Conductivity cell, cell constant 0.1 ii) Conductivity cell, cell constant 1.0 iii) Temp. Probe (PT-100 sensor) iv) Stand & Clamp

Solutions were prepared by weight precise to ± 0.02 %. The weights were taken on a Mettler electronic analytical balance (AG 285, Switzerland). The molarity being converted to molality as required. Several independent solutions were prepared and runs were performed to ensure the reproducibility of the results. Due correction was made for the specific conductance of the solvents at desired temperatures. The following figure shows the Block diagram of the Systronics Conductivity-TDS meter 308.

III.2.3.3 DENSITY MEASUREMENT

The density measurement was performed with the help of Anton Paar DMA 4500M digital density-meter with a precision of ±0.0005 g·cm^{-3}.

Anton Paar DMA 4500M digital density-meter

In the digital density meter, the mechanic oscillation of the U-tube is e.g. electromagnetically transformed into an alternating voltage of the same frequency. The period τ can be measured with high resolution and stands in simple relation to the density ρ of the sample in the oscillator:

$$\rho = A \cdot \tau^2 - B \qquad \text{(III.a)}$$

A and B are the respective instrument constants of each oscillator. The values of A and B are determined by the calibration with the solutions of two different substances of known densities ρ_1 and ρ_2. Modern instruments calculate and store the constants A and B after the two calibration measurements, which are mostly performed with air and water. They produce suitable values to balance various influences during the measurement, e.g., the influence of the sample's viscosity and the non-linearity caused by the measuring instrument's finite mass. The instrument was calibrated by triply-distilled water and dry air.

Instrument Specification:

Density	*: 0 to 1.5 g.cm^{-3}*
Temperature	*: 15°C to 25°C*
Pressure	*: 0 to 6 bar*
Repeatability Standard deviation	
Density	*: 0.00001 g.cm^{-3}*
Temperature	*: 0.01 °C*
Additional information	
Minimum sample volume	*: approx. 2 ml*
Dimensions (L×W×H)	*: 400×225×231 mm*
Weight	*: approx. 15 kg*
Automatic bubble detection	*: yes*
Interfaces	*: 2×CAN*
Power	*: Supplied by the master instrument*

III.2.3.4 VISCOSITY MEASUREMENT

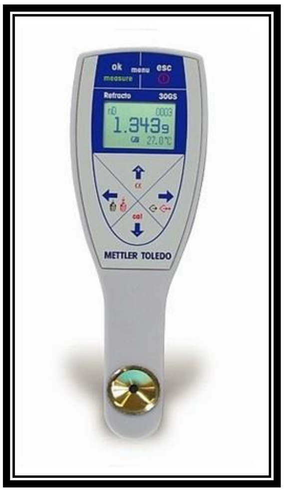
Brookfield DV-III Ultra Programmable Rheometer: The viscosities (η) were measured using a Brookfield DV-III Ultra Programmable Rheometer with fitted spindle size-42. The viscosities were obtained using the following equation

$$\eta = (100 / RPM) \times TK \times \text{torque} \times SMC$$

Where, RPM, TK (0.09373) and SMC (0.327) are the speed, viscometer torque constant and spindle multiplier constant, respectively. The calibration of the instrument was done using the standard viscosity sample solutions supplied with the instrument, water and aqueous $CaCl_2$ solutions [7]. The temperature was maintained to within ± 0.01°C using Brookfield Digital TC-500 thermostat bath. This instrument provides viscosity values with an accuracy of ± 1 %. Each measurement was reported as an average of three separate reading with a precision of 0.3 %.

Instrument Specifications:

Speed Range	*: 0-250 RPM, 0.1 RPM increments*
Viscosity Accuracy	*: ±1.0% of full scale range for a specific spindle running at a specific speed.*
Temperature sensing range	*: -100°C to 300°C (-148°F to 572°F)*
Temperature accuracy	*: ±1.0°C from -100°C to 150°C* *±2.0°C from +150°C to 300°C*
Analog Torque Output	*: 0 - 1 Volt DC (0 - 100% torque)*
Analog Temperature Output	*: 0 - 4 Volts DC (10mv / °C)*



III.2.3.5 REFRACTIVE INDEX MEASUREMENT

Refractive index was be measure with the help of Digital Refractometer (Mettler Toledo 30GS).

The instrument was calibrated using double-distilled water, toluene, cyclohexane, and carbon tetrachloride at defined temperature. The accuracy of the instrument is ±0.0005. A few drops of the sample solution were placed onto the measurement cell and the value of refractive index was taken. As refractive index is dependent on temperature, refractometer is designed to determine the temperature to produce the exact value.

Specifications-Refracto 30GS- extended RI measuring range

Model	*: Refracto 30GS*
Measurement range	*: 1.32 -1.65*
Resolution	*: 0.0001*
Accuracy	*: +/- 0.0005*
Measurement range BRIX	*: 0 - 85 Brix%*
Resolution	*: 0.1 Brix%*
Accuracy	*: +/- 0.2 Brix%*
Temperature range	*: 10 - 40°*
Resolution of temperature	*: 0.1°*
display	*: °C or °F*
Trade Name	*: 51324660*

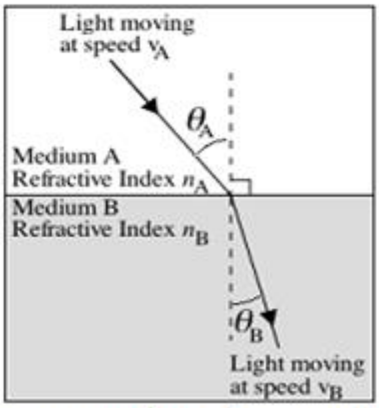
The ratio of the speed of light in a vacuum to the speed of light in another substance is defined as the index of refraction (aka refractive index or n_D) for the substance.

$$Refractive\,index\,of\,the\,substance\,(n_D) = \frac{Speed\,of\,light\,in\,a\,vacuum}{Speed\,of\,light\,in\,substance} \quad \text{(III.b)}$$

$$\frac{V_A}{V_B} = \frac{\sin\theta_A}{\sin\theta_B} = \frac{n_B}{n_A} \quad \text{(III.c)}$$

Hence, without measuring the speed of light in a sample its index of refraction can easily be determined. It measures angle of refraction with refraction index of the layer which is in contact with the solution of the sample and calculates the refractive index precisely [8] [9] Nearly all refractometers utilize this principle, but may differ in their optical design.

Light crossing from any transparent medium into another in which it has a different speed, is refracted, i.e., bent from its original path (except when the direction of travel is perpendicular to the boundary). In the case shown, the speed of light in medium A is



greater than the speed of light in medium B

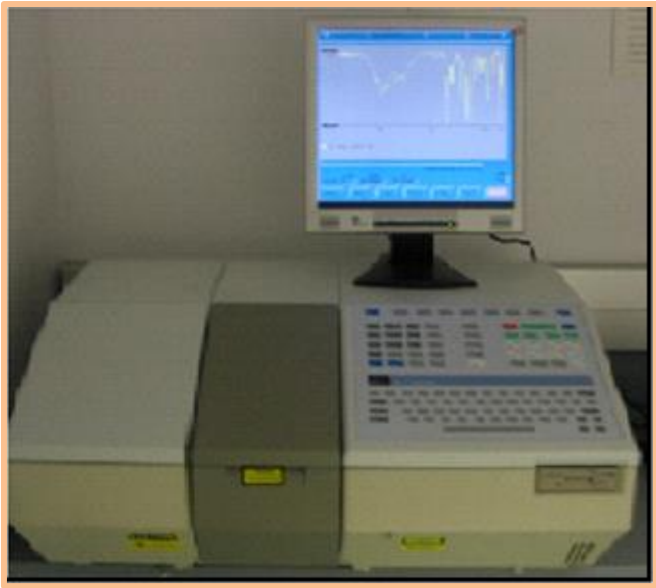
A light from its source is projected towards the illuminating prism with ground bottom surface that means roughened like a ground-glass joint so that each point on this surface can be regarded as producing light rays to be travelled in all directions. As in figure2 light propagating from point A to point B with largest angle of incidence (q_i) and consequently the largest possible angle of refraction (q_r) for a particular sample. Rest of the rays of light which go into the refracting prism with q_r and consequently get revealed to the left of point C. Thus, the detector positioned on the back side of the refracting prism would show a light region to the left and a dark region to the right.

III.2.3.6 SURFACE TENSION MEASUREMENT

Surface tension was measured by using Digital Tensiometer KRUSS K9 (Germany). The tensiometer is a precision instrument which will only perform reliably on a solid and vibration-free base. It places the same demands on its surroundings as a laboratory balance with a resolution of 0.1 mg. In addition surface tension measurements require a clean and dust-free atmosphere as atmospheric pollutants could directly falsify the results.

III.2.3.7 FT-IR MEASUREMENT

Infrared spectra were recorded in 8300 FTIR spectrometer (Shimadzu, Japan).



The intensity of light (I_0) passing through a blank is measured. The intensity is the number of photons per second. The blank solution is identical to the sample solution only differing in the case that it does not contain the substrate which absorbs light. The intensity of light (I) passing through the sample solution is measured. (In practice, instrument measures the power rather than the intensity of the light per second, which is the result of the flow of the photons per second (or intensity) and the energy per photon. The experimental data is used to calculate two quantities: the transmittance (T) and the absorbance (A).

$$T = \frac{I}{I_0}; \qquad A = -\log_{10} T \tag{III.d}$$

The fraction of light in the original beam passing through the sample and reaches the detector is the transmittance.

III.2.3.8 UV-VIS SPECTRA MEASUREMENT

Compounds that absorb Ultraviolet and/or visible light have characteristic absorbance curves as a function of wavelength. Absorbance of altered wavelengths of light arises as the molecules travel to upper energy states.

The UV-VIS spectrophotometer uses two light sources, a deuterium (D_2) lamp for ultraviolet light and a tungsten (W) lamp for visible light. After bouncing from a specially designed mirror, the light beam travels through a narrow slit and hits a diffraction grating. The grating can be rotated allowing for a specific wavelength to be selected. At any specific orientation of the grating, only monochromatic (single wavelength) successfully passes through a slit. A filter is used to remove unwanted higher orders of diffraction. There is a half mirror where half of the light is reflected and the other half passes through. Before the half mirror the light beam hits a second mirror to avoid the splitting. The spectra of the solvent is first recorded and saved to use it further as reference while recording the spectra of the sample. The intensities of the light beams are then measured at the end. Regarding this the Beer-Lambert law has been stated below[10].

Beer-Lambert Law

The change in intensity of light (dI) after passing through a sample should be proportional to the following:

(i) Path length (b), the longer the path, more photons should be absorbed

(ii) Concentration (c) of sample, more molecules absorbing means more photons absorbed

(iii) Intensity of the incident light (I), more photons means more opportunity for a molecule to see a photon. Thus, dI is proportional to bcI or $dI/I = -kbc$ (where k is a proportionality constant, the negative sign is shown because this is a decrease in intensity of the light, this makes b, c and I always positive. equation leads to Beer-Lambert's law [11]:

$$-\ln I/I_0 = kbc \qquad \text{(III.8)}$$

$$-\log I/I_0 = 2.303kbc \qquad \text{(III.9)}$$

$$\varepsilon = 2.303k \qquad \text{(III.10)}$$

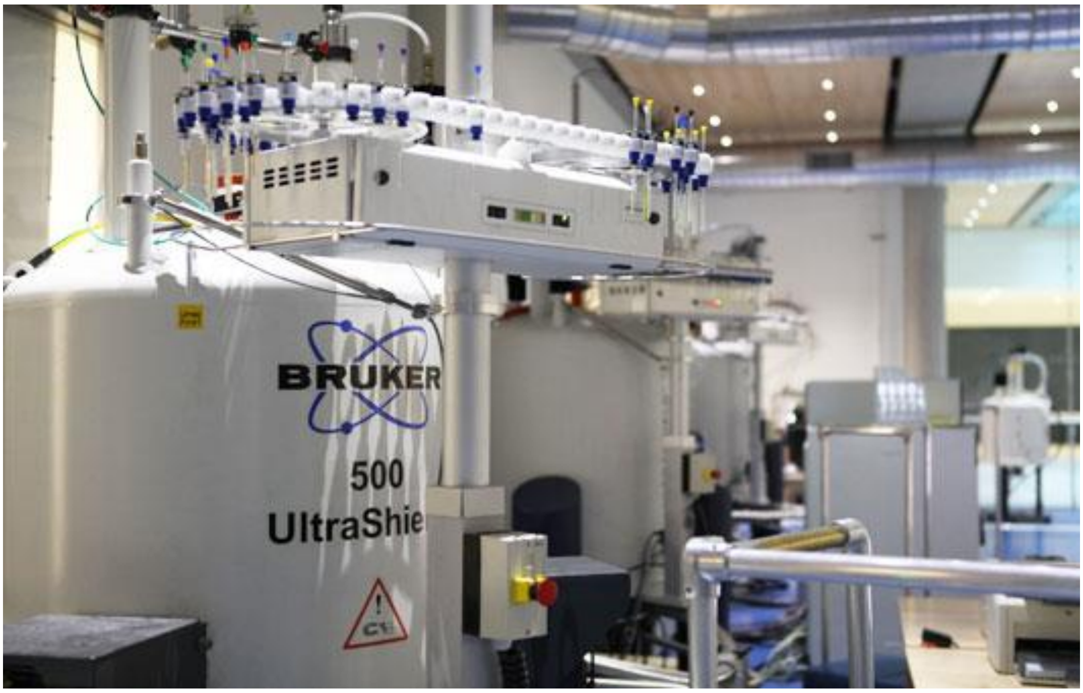
$$A = -\log I/I_0 \qquad \text{(III.11)}$$

$$A = \varepsilon bc \qquad \text{(III.12)}$$

A is defined as absorbance and it is found to be directly proportional to the path length, b and the concentration of the sample, c. The extinction coefficient is characteristic of the substance under study and of course is a function of the wavelength.

III.2.3.9. NMR SPECTRA MEASUREMENT

As on the strength of the magnetic field the resolution is mainly dependent, the NMR spectrometers are designed with very strong, big and liquid helium-cooled superconducting magnet. Less expensive machines where permanent magnets are used are also available, which still give sufficient performance for certain application such as reaction monitoring and quick checking of samples but resolution is quite low.



The protons of the solvents, as most regular solvents are hydrocarbons, are NMR active. Thus, deuterium (hydrogen-2) is substituted (99+ %). Generally deuterochloroform ($CDCl_3$) is used as a solvent. Apart from deuterochloroform deuterium oxide (D_2O) and deuterated DMSO (DMSO-d_6) are also used where applicable. While recording the NMR spectra often known solvent residual proton peak was taken as the internal standard where applicable instead of adding tetramethylsilane NMR spectra were recorded in D_2O unless otherwise stated. ^{1}H NMR spectra were recorded at 300 MHz and 400 MHz using Bruker ADVANCE 500 MHz and Bruker ADVANCE 400 MHz instruments respectively at 298.15K. Signals are quoted as δ values in ppm using residual protonated solvent signals as internal standard (D_2O : δ 4.79 ppm). Data are reported as chemical shift [12] [13].

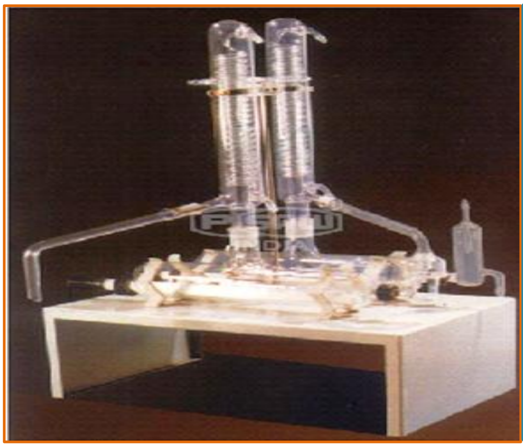
III.2.3.10. MASS SPECTROMETRY

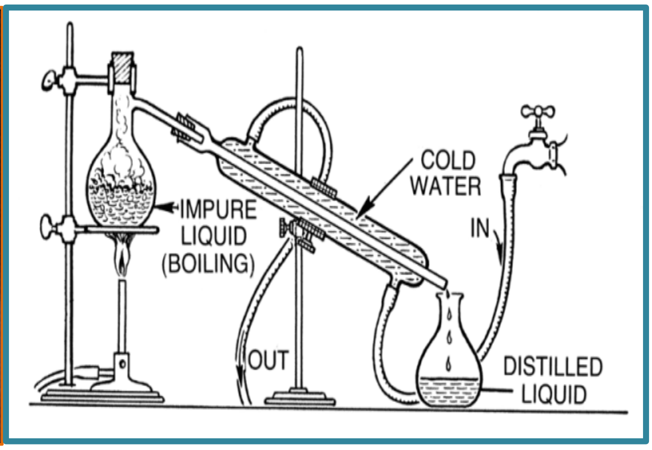
HRMS analyses were studied with Q-TOF high resolution instrument by positive mode electro-spray ionization.

Other Instruments Used:

III.2.3.10. WATER DISTILLER

Water from the natural sources is manually or automatically fed into steaming chamber of the distiller unit's. The steam arises from the steaming chamber is passed through a built-in vent to condenser where the steam gets converted into water which then passes through and store into a container. Minerals and salts due to high boiling point remains in the boiling chamber as hard deposits or scale. The distilled water is then collected in a storage tank. If the unit is an automatic model, it is set to operate to fill the storage tank. The distillation apparatus contains a flask with heating elements embedded in glass and fused in spiral type coil internally of the bottom and tapered round glass, joins at the top double walled condenser with B-40/B-50ground glass joints, suitable to work on 220 volt 50 Hz AC supply.





III.2.3.11. THERMOSTAT WATER BATH (Science India, Kolkata):

Temperature was controlled using thermostatic water bath and in which the experiments were also carried out. The temperature was maintained with an accuracy of ± 0.01 K of the desired temperature. Brookfield TC-550 water bath was used for viscometric measurements and other experiments.

Features and Benefits of Brookfield TC-550 water bath

- Provides standalone operation - no tap water required
- Easy control of set-point
- Configured to measure viscosity directly in the bath - accommodates 600 mL beaker
- Programmable Controller version is designed to automate sample temperature control.
- Built-in circulator pumps to external devices.

Laboratory water bath is a system in which a vessel containing the material to be heated is placed into or over the one containing water and to quickly heat it. These laboratory equipment supplies are available in different volumes and construction with both digital and analogue controls and greater temperature uniformity, durability, heat retention and recovery. The chambers of water bath lab products are manufactured using rugged, leak proof and highly resistant stainless steel and other lab supplies.