CHAPTER III

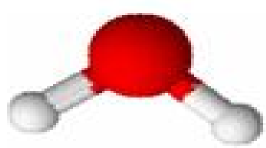
EXPERIMENTAL SECTION

3.1. NAME, STRUCTURE, PHYSICAL AND CHEMICAL PROPERTIES, PURIFICATION AND APPLICATIONS OF THE SOLVENTS AND SOLUTES USED IN THE RESEARCH WORK

3.1.1. SOLVENTS

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.



Appearance: Colourless Liquid
Molecular Formula: H_2O
Molecular Weight: 18.02 g/mol
Melting Point: 273 K
Boiling Point: 373 K
Dielectric Constant: 78.35 at 298 K

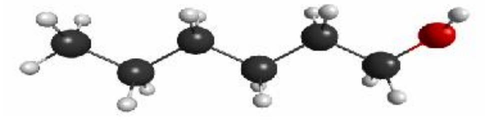
Source: Distilled water, distilled from fractional distillation method in Lab.

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·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 is also use in various industries. It is a superb solvent, generally taken as the universal solvent, due to the marked polarity of the water molecule and its tendency to form hydrogen bonds with other molecules. Life on earth totally depends on water. Not only a high percentage of living things, 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 largely of water. About 70 to 90 percent of all organic matter is water. The chemical reactions in all plants and animals that support life take place in a 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 these reactions. In short, the chemistry of life is water chemistry.

1-Hexanol:

1-Hexanol is a primary alcohol with a six-carbon chain and the molecular formula CH_3CH_2 CH_2 CH_2 CH_2 CH_2OH. This colourless liquid is slightly soluble in water. 1-Hexanol is believed to be a component of odour or freshly mown grass. Alarm pheromones emitted by the Koschevnikov gland of honey bees contain 1-hexanol.



Appearance: Colourless Liquid

Molecular Formula: $C_6H_{14}O$

Molecular Weight: 102.18 g/mol

Melting Point: 222-232 K

Boiling Point: 428-432 K

Dielectric Constant: 13.35 at 298 K

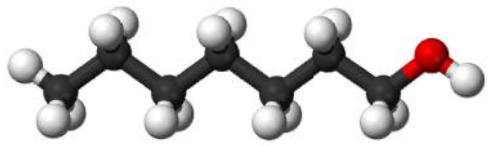
Source: sd. Fine Chemicals Ltd. Mumbai, India.

Purification: It was dried by adding drying agent $CaSO_4$ followed by filtration and then distillation [1].

Application: It is used in the perfume industry. It is used as solvent in the production of plasticizer, surfactant.

1-Heptanol:

1-Heptanol is an alcohol with a seven carbon chain and the structural formula of $CH_3(CH_2)_6OH$. It is a clear colourless liquid and very slightly soluble in water. Hydrogenations of Heptaldehyde with nickel catalysts will yield Heptyl alcohol.



Appearance: Colourless Liquid
Molecular Formula: $C_7H_{16}O$
Molecular Weight: 116.20 g/mol
Melting Point: 238.4 K
Boiling Point: 448.8 K
Dielectric Constant: 6.13 at 298 K

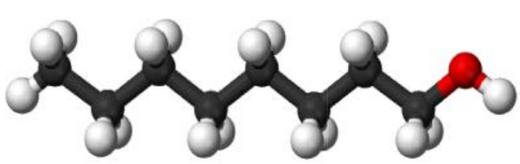
Source: sd. Fine Chemicals Ltd. Mumbai, India.

Purification: It was dried by adding drying agent $CaSO_4$ followed by filtration and then distillation [1].

Application: Heptanol is commonly used in cardiac electrophysiology experiments to block gap junctions and increase axial resistance between myocytes. It has an pleasant smell and used in cosmetic for its fragrance. It is also used in flavour compositions for imitation of coconut, and in various fruits.

1-Octanol:

1-Octanol also known as Octan-1-ol is the organic compound with molecular formula $CH_3(CH_2)_7OH$. It is a fatty alcohol. It is manufactured for the synthesis of esters for use in perfumes and flavouring. Esters of Octanol, such as octyl acetate, occur as components of essential oils. It is used to evaluate the lipophilicity of pharmaceutical products.



Appearance: Colourless Liquid
Molecular Formula: $C_8H_{18}O$
Molecular Weight: 130.23 g/mol
Melting Point: 257 K
Boiling Point: 468 K
Dielectric Constant: 5.14 at 298 K

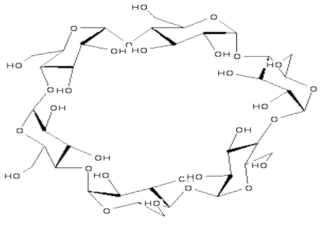
Source: sd. Fine Chemicals Ltd. Mumbai, India

Purification: It was dried by adding drying agent $CaSO_4$ followed by filtration and then distillation [1].

Application: 1-Octanol is mainly consumed as a precursor of perfumes. It has been examined for controlling essential tremor and other types of involuntary neurological tremors.

α- Cyclodextrin:

α- Cyclodextrin is a cyclic oligosaccharide having six glucopyranose units bounded by connections establishing a abbreviated narrowed structure, which has hydrophobic interior and hydrophilic frames [2]. Their whole molecular shape is that of a truncated cone, with a hydrophobic hollow and a polar interface, categorized by hydroxylated, hydrophilic frames (a narrower, primary border and a wider, secondary border)[3].



Appearance: Crystalline

Molecular Formula: $C_{36}H_{60}O_{30}$

Molecular Weight: 972.84 g/mol

Melting Point: 551 K

Source: Sigma Aldrich, Germany

Purification: Used as purchsed without further purification. The purity is 96%.

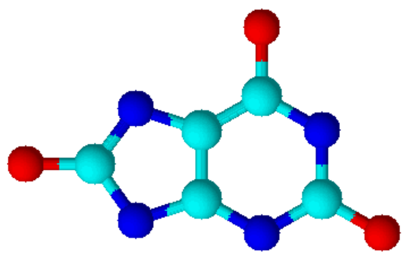
Application: α- Cyclodextrin is a multifunctional component, marketed as a fibre ingredients, an odour masking agent, as well as emulsifying applications. α-Cyclodextrin is marketed for a range of medical, healthcare and food and beverage applications which rely on its ability to bind to fats and reduce their bioavailability both in the body and in food and beverage products. As a fiber dietary supplement,α-cyclodextrin binds to fat when taken with a meal and is a natural alternative to other synthetic anti-obesity medications. α-Cyclodextrin has the unique ability among known fibers to bind nine times its own weight in fat. α-Cyclodextrin can also be used as whipping fiber, for example in desserts and confectionery applications. Tests showed that alpha cyclodextrins can cause a volume effect in various different food compositions with or without fat and at a very broad pH range. This can be used for fat free or fat containing dessert compositions and for the reduction or the replacement of egg white in confectionery and bakery applications.

Uric acid:

Uric acid is a heterocyclic compound with the molecular formula $C_5H_4N_4O_3$, i.e. 7,9-Dihydro-1H –purine-2,6,8 (3H)-trione. It is a diprotic acid. It was first isolated from kidney stones in 1776 by Scheele [4]. In general, the water solubility of uric acid is low. This low solubility is significant for the etiology of gout. Uric acid is a strong reducing agent and potent antioxidant.

Source: sd. Fine Chemicals Ltd. Mumbai, India

Purification: Used as purchased without further purification. The purity is 99%.



Appearance: Crystalline

Molecular Formula: $C_5H_4N_4O_3$

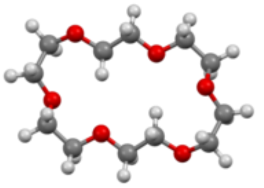
Molecular Weight: 168.11 g/mol

Melting Point: 573 K

Application: Uric acid may be a marker of oxidative stress and may have the potential therapeutic role as an antioxidant[5]. On the other hand, like other strong reducing substances such as ascorbate, uric acid can also act as a prooxidant.

18-Crown-6:

18-Crown-6 is an organic compound with the formula $[C_2H_4O]_6$ and the IUPAC name of 1,4,7,10,13,16-hexaoxacyclooctadecane. It is a white, hygroscopic crystalline solid with a low melting point. Like other crown ethers, 18-crown-6 functions as a ligand for some metal cations with a particular affinity for potassium cations (binding constant in methanol: 10^6 M^{-1}). The dipole moment of 18-crown-6 varies in different solvent and under different temperature. Under 25 °C, the dipole moment of 18-crown-6 is 2.76 ± 0.06 D in cyclohexane and 2.73 ± 0.02 in benzene. The synthesis of the crown ethers led to the awarding of the Nobel Prize in Chemistry to Charles J. Pedersen[6,7].



Appearance: Crystalline

Molecular Formula: $C_{12}H_{24}O_6$

Molecular Weight: 264.32 g/mol

Melting Point: 315-318 K

Source: Sigma Aldrich, Germany

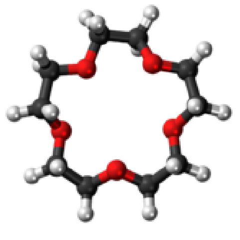
Purification: Used as purchased without further purification. The purity is >99%.

Application: 18-Crown-6 binds to a variety of small cations, using all 6 oxygens as donor atoms. Crown ethers can be used in the laboratory as phase transfer catalysts. For example, potassium permanganate dissolves in benzene In the presence of 18-crown-6, giving the so-called "purple benzene", which can be used to oxidize diverse organic compounds. Various substitution reactions are also accelerated in the presence of 18-crown-6, which suppresses ion-pairing. The anions thereby become naked nuclophiles. For example, using 18-crown-6, potassium acetate is a more powerful nucleophile in organic solvents:

$[K(18\text{-crown-}6)^+]OAc^- + C_6H_5CH_2Cl \rightarrow C_6H_5CH_2OAc + [K(18\text{-crown-}6)^+]Cl^-$

15-Crown-5:

15-Crown-5 is a crown ether with the formula $(C_2H_4O)_5$. It is a cyclic pentamer of ethylene oxide that forms complex with various cations [8], including sodium (Na^+) and potassium (K^+), however, it is complementary to Na^+ and thus has a higher selectivity for Na^+ ions.



Appearance: Liquid

Molecular Formula: $C_{10}H_{20}O_5$

Molecular Weight: 220.27 g/mol

Boiling Point: 366-369 K

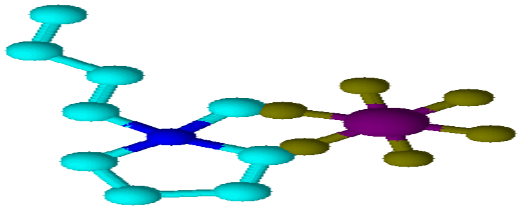
Source: Sigma Aldrich, Germany

Purification: Used as purchased without further purification. The purity is >98%.

Application: 15-Crown-5 is used as complexing agent. It has also been used to isolate oxonium ions. For example, from a solution of tetrachloroauric acid, the oxonium ion $[H_7O_3]^+$ has been isolated as the salt $[(H_7O_3)(15\text{-crown-}5)_2][AuCl_4]$.

1-butyl-1-methylpyrrolidinium hexafluorophosphate:

1-butyl-1-methylpyrrolidinium hexafluorophosphate is the pyrrolidinium based ionic liquid, of molecular formula $C_9H_{20}F_6NP$, containing butyl, methyl group with one nitrogen atom in the pyrrolidole or five-membered ring.



Appearance: Crystalline

Molecular Formula: $C_9H_{20}F_6NP$

Molecular Weight: 287.23 g/mol

Source: Sigma Aldrich, Germany

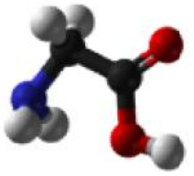
Purification: Used as purchased without further purification. The purity is >97.5%.

Application: It is 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.

3.1.2. SOLUTES (Electrolytes and Non-Electrolytes)

Glycine:

Glycine (abbreviated as Gly or G) is an organic compound with the formula NH_2CH_2COOH. Having a hydrogen substituent as its side-chain, glycine is the smallest of the 20 amino acids commonly found in proteins. Its codons are GGU, GGC, GGA, GGG of the genetic code. Glycine is a colourless, sweet-tasting crystalline solid. It is unique among the proteinogenic amino acids in that it is not chiral. It can fit into hydrophilic or hydrophobic environments, due to its minimal side chain of only one hydrogen atom.



Appearance: White Powder

Molecular Formula: $C_2H_5NO_2$

Molecular Weight: 75.07 g/mol

Melting Point: 506 K

Source: Sigma Aldrich, Germany

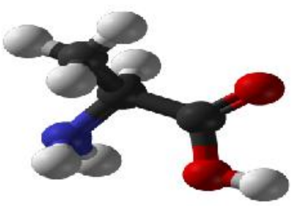
Purification: It was used after recrystallization from (ethanol + water) mixture and dried over P_2O_5 in a desiccator for about 72 h before use. The purity is 99%.

Application: Pharmaceutical grade glycine is produced for or some pharmaceutical applications, as intravenous injections. Technical grade glycine is solid for use in industrial applications, e.g. as an agent in metal complexing and finishing. For humans, glycine is solid as a sweetner/taste enhancer. Certain food supplements and protein drinks containing glycine, for drug formulations it used to improve gastic absoption. Glycine serves as a buffering agent in antacids, analgesics, antiperspirants, cosmetics, and toiletries. Many miscellaneous products use glycine or its derivatives, such as the roduction of rubber sponge products, fertilizers, metal complexants.

L-Alanine:

Alanine (abbreviated as Ala or A) is an α-amino acid with the chemical formula $CH_3CH(NH_2)COOH$. The L-isomer is one of the 20 amino acids encoded by the genetic

code. Its codons are GCU, GCC, GCA, and GCG. It is classified as a nonpolar amino acid. L-Alanine is second only to leucine in rate of occurrence, accounting for 7.8% of the primary structure in a sample of 1,150 proteins. D-Alanine occurs in bacterial cell walls and in some peptide antibiotics.



Appearance: White Powder

Molecular Formula: $C_3H_7NO_2$

Molecular Weight: 89.09 g/mol

Melting Point: 531 K

Source: Sigma Aldrich, Germany

Purification: : It was used after recrystallization from (ethanol + water) mixture and dried over P_2O_5 in a desiccator for about 72 h before use. The purity is 98%.

Application: alanine is used in dosimetric measurements in radiotherapy. Alanine plays a key role in glucose–alanine cycle between tissues and liver. In muscle and other tissues that degrade amino acids for fuel, amino groups are collected in the form of glutamate by transamination.

L-Isoleucine:

L-Isoleucine is an α-amino acid with chemical formula $C_2H_5CH(CH_3)CH(NH_2)COOH$. It is essential in humans, meaning the body cannot synthesize it, and it must be ingested in our diet. Foods that have high amounts of isoleucine include eggs, soy proteis, seaweed, turkey, chicken, lamb, cheese, and fish.

Appearance: Powder
Molecular Formula: $C_6H_{13}NO_2$
Molecular Weight: 131.18 g/mol
Melting Point: 561 K

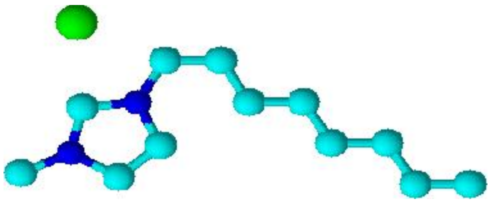
Source: Sigma Aldrich, Germany

Purification: It was used after recrystallization from (ethanol + water) mixture and dried over P_2O_5 in a desiccator for about 72 h before use. The purity is 98%.

Application: It is used as fuel by muscle cells, sparing other amino acids from being burned up. L-isoleucine is commonly used in combination with other branched chain amino acids to improve the nutritional status of patients with hepatic diseases. It plays an important role in protein synthesis, anabolism and anti-catabolism.

1-methyl-3-octylimidazolium chloride:

1-methyl-3-octylimidazolium chloride is the imidazolium based ionic liquid, of molecular formula $C_{12}H_{23}N_2Cl$, containing methyl, octyl group with two active nitrogen atoms in the imidazole or five member ring, exist as a molten liquid phase.



Appearance: Liquid

Molecular Formula: $C_{12}H_{23}N_2Cl$

Molecular Weight: 230.78 g/mol

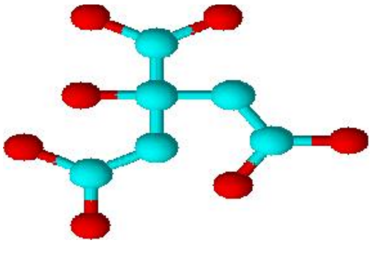
Source: Sigma Aldrich, Germany

Purification: Used as purchased without further purification. The purity is >97%.

Application: It is 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.

Citric Acid:

Citric acid, that is, 2-hydroxypropane-1,2,3-tricarboxylic acid, is a tribasic, environmentally acceptable, and versatile chemical. It is a natural preservative/conservative and is also used to add an acidic or sour taste to foods and drinks. In biochemistry, the conjugate base of citric acid, citrate, is important as an intermediate in the citric acid cycle, which occurs in the metabolism of all aerobic organisms. It consists of 3 carboxyl (R-COOH) groups. Citric acid is a commodity chemical, and more than a million tonnes are produced every year by fermentation. It is used mainly as an acidifier, as a flavoring, and as a chelating agent.



Appearance: Crystalline

Molecular Formula: $C_6H_8O_7$

Molecular Weight: 192.12 g/mol

Melting Point: 523.15 K

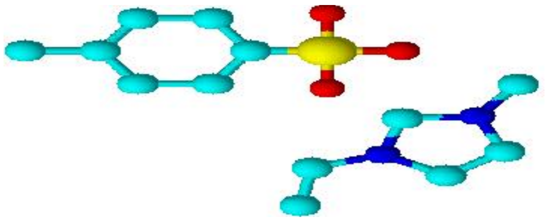
Source: Citric acid monohydrate (CA) was purchased from HiMedia.

Purification: Its mass purity as supplied is 0.99. The reagent was always placed in the desiccators over P_2O_5 to keep them in dry atmosphere.

Application: The dominant use of citric acid is as a flavouring and preservative in food and beverages, especially soft drinks [9]. Citric acid can be added to ice cream as an emulsifying agent to keep fats from separating, to caramel to prevent sucrose crystallization, or to recipes in place of fresh lemon juice. Citric acid is used with sodium bicarbonate in a wide range of effervescent formulae. Citric acid is also often used in cleaning products and sodas or fizzy drinks. Citric acid is an excellent chelating agent, binding metals. It is used to remove lime scale from boilers and evaporators. Citric acid can be used in shampoo to wash out wax and colouring from the hair. It can be used to soften water, which makes it useful in soaps and laundry detergents. Citric acid is widely used as a pH adjusting agent in creams and gels of all kinds. Citric acid is an alpha hydroxy acid and used as an active ingredient in chemical peels. Citric acid is commonly used as a buffer to increase the solubility of brown heroin. Citric acid is used as one of the active ingredients in the production of antiviral tissues. Citric acid can be used in food colouring to balance the pH level of a normally basic dye. It is used as an odourless alternative to white vinegar for home dyeing with acid dyes. Citric acid can be used as a successful alternative to nitric acid in passivation of stainless steel. Citric acid can be used as a lower-odour stop bath as part of the process for developing photographic film. As it occurs in metabolism of almost all living beings, its interactions in an aqueous solution is of great value to the biological scientists. In the pharmaceutical industry, citric acid is used as a stabilizer in various formulations, as a drug component and as an anticoagulant in blood for transfusions and also used as an acidifier in many pharmaceuticals. Citric acid can be used in food colouring to balance the pH level of the normally basic dye. Citric acid's ability to chelate metals makes it useful in soaps and laundry detergents, as well as water softening.

1-ethyl-3-methylimidazolium tosylate:

1-ethyl-3-methylimidazolium tosylate is also imidazolium based ionic liquid, of molecular formula $C_{13}H_{18}N_2O_3S$, containing ethyl, methyl groups with two active nitrogen atoms in the imidazole or five member ring, exist as a molten liquid phase with the melting point below 313K.



Appearance: Beige Crystalline

Molecular Formula: $C_6H_{11}N_3O_3$

Molecular Weight: 173.17 g/mol

Melting Point: 313.15 K

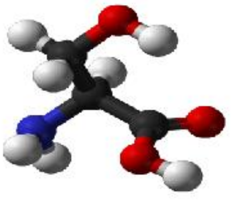
Source: Sigma Aldrich, Germany

Purification: Used as purchased without further purification. The purity is >98%.

Application: It is 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 [10] in the field of electrochemistry.

L-Serine:

Serine (abbreviated as Ser or S) encoded by the codons UCU, UCC, UCA, UCG, AGU and AGC is an α-amino acid that used in the biosynthesis of proteins. Serine was first obtained from silk protein, a particularly rich source, in 1865. Its name is derived from the Latin silk, *sericum*.



Appearance: Crystalline

Molecular Formula: $C_3H_7NO_3$

Molecular Weight: 105.09 g/mol

Melting Point: 519 K

Source: Sigma Aldrich, Germany

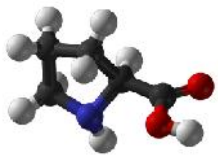
Purification: Used as purchased without further purification. The purity is 99%.

Application: Serine is important in metabolism in that it participates in the biosynthesis of purines and pyrimidines. It is the precursor to several amino acids including glycine and cysteine. Serine plays an important role in catalytic function of many enzymes.

L-Proline:

Proline (abbreviated as Pro or P) encoded by the codons CCU, CCC, CCA, and CCG is an α-amino acid that is used in the biosynthesis of proteins. Proline is the only amino acid with secondary amine. It was first isolated in 1900 by Richard Willstater.

The distinctive cyclic structure of proline's side chain gives proline an exceptional conformational rigidity compared to other amino acids.



Appearance: Crystalline

Molecular Formula: $C_5H_9NO_2$

Molecular Weight: 115.13 g/mol

Melting Point: 478-501 K

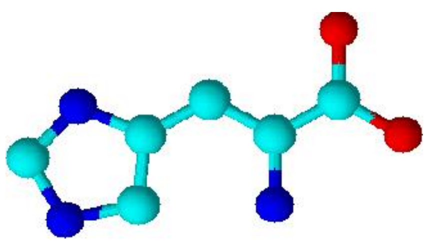
Source: Sigma Aldrich, Germany

Purification: Used as purchased without further purification. The purity is 98%.

Application: Proline and its derivatives are often used as asymmetric catalysts in proline organocatalysis reactions. In brewing, proteins rich in proline combine with polyphenols to produce haze (turbidity). The growth medium used in plant tissue culture may be supplemented with proline.

L-Histidine:

Histidine (abbreviated as His or H) encoded by the codons CAU and CAC is an α-amino acid that is used in the biosynthesis of proteins. Histidine was first isolated by German physician Albretch Kossel and Sevin Hedin in 1896.



Appearance: Crystalline

Molecular Formula: $C_6H_9N_3O_2$

Molecular Weight: 155.16 g/mol

Melting Point: 555 K

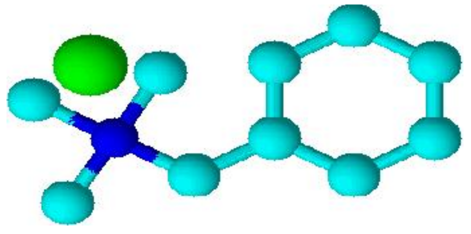
Source: Sigma Aldrich, Germany

Purification: Used as purchased without further purification. The purity is 99%.

Application: Histidine is a precursor to histamine, a vital inflammatory agent to immune responses. Histidine assists in stabilising oxyhaemoglobin and destabilising CO-bound haemoglobin.

Benzyltrimethyl ammonium chloride:

Benzyltrimethyl ammonium chloride is the ammonium based ionic liquid, of molecular formula $C_{10}H_{16}ClN$, exist as a molten solid phase.



Appearance: Crystalline

Molecular Formula: $C_{10}H_{16}ClN$

Molecular Weight: 185.69 g/mol

Melting Point: 503 K

Source: Sigma Aldrich, Germany

Purification: Used as purchased without further purification. The purity is >98%.

Application: It is used as antistatic agent, detergent sanitisers, softner for textiles and paper products, phase transfer catalyst, antimicriobial, disinfection agents and sanitizers. It is also used as emulsifying agents, pigment Dispersers.

3.2. EXPERIMENTAL METHODS

3.2.1. PREPARATION OF SOLVENT MIXTURES

For the preparation of solvent mixture, pure components were taken separately in glass stoppered bottles and thermostated at the desired temperature for sufficient time. When the thermal equilibrium was ensured, the required volumes of each component were transferred in a different bottle which was already cleaned and dried thoroughly. Conversion of required mass of the respective solvents to volume was accomplished by using experimental densities of the solvents at experimental temperature. It was then stoppered and the mixed contents were shaken well before use. While preparing different solvent mixtures care was taken to ensure that the same procedure was adopted throughout the entire work. The physical properties of different pure and mixed solvents have been presented in the respective chapters.

3.2.2. PREPARATION OF SOLUTIONS

A stock solution for each salt was prepared by mass, and the working solutions were obtained by mass dilution. The uncertainty of molarity of different salt solutions was evaluated to be ± 0.0003 mol·dm^{-3}.

3.2.3. MASS MEASUREMENT

Mass measurements were made on digital electronic analytical balance (Mettler Toledo, AG 285, Switzerland).

It can measure mass to a very high precision and accuracy. The weighing pan of a high precision (0.0001g) is inside a transparent enclosure with doors so that dust does not collect and so any air currents in the room do not affect the balance's operation.

3.2.4. DENSITY MEASUREMENT

The density was measured with the help of Anton Paar density-meter (DMA 4500M) with a precision of 0.0005 g·cm^{-3}.

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 [11]:

$$\rho = A \cdot \tau^2 - B \quad (1)$$

A and B are the respective instrument constants of each oscillator. Their values are determined by calibrating with two substances of the precisely 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 employ suitable measures to compensate various influences on the measuring result, 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 double-distilled water and dry air.

3.2.5. VISCOSITY MEASUREMENT

The viscosity (η) was also measured with the help Brookfield DV-III Ultra Programmable Rheometer with fitted spindle size-42 connected to a Brookfield Digital Bath TC-500. 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. It was calibrated against the standard viscosity samples supplied with the instrument, water and aqueous $CaCl_2$ solutions. Temperature of the experimental solution was maintained ± 0.01K using Brookfield Digital TC-500 thermostat bath. Viscosities were measured with an accuracy of ±1 %. Each measurement reported herein is an average of triplicate reading with a precision of 0.3%.

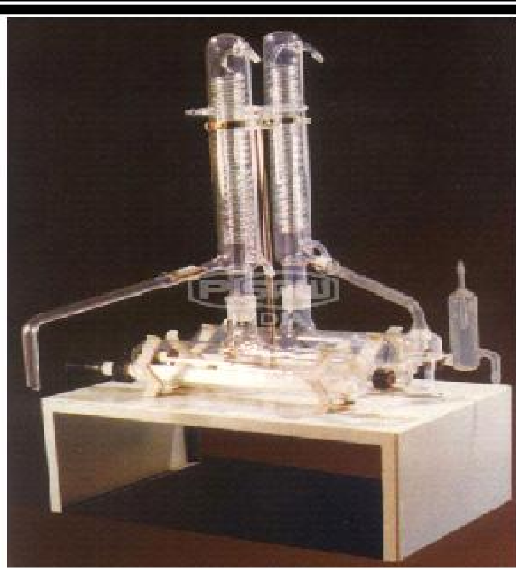
3.2.6. TEMPERATURE CONTROLLER

All the measurements were carried out in thermostatic water bath (Science India, Kolkata) maintained with an accuracy of ± 0.01 K of the desired temperature.

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 equipments 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.

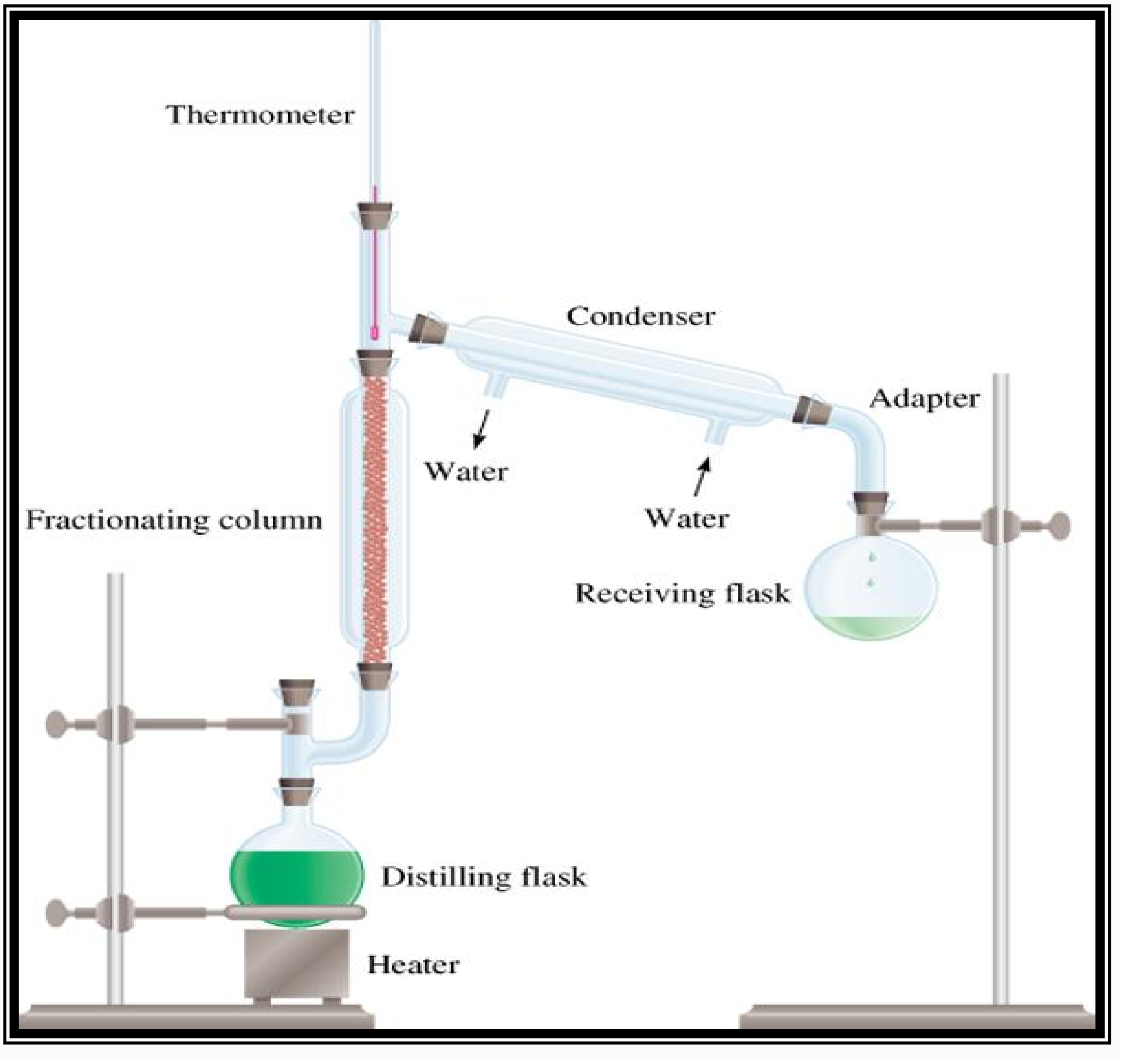
3.2.7. WATER DISTILLER

Water distillation units produce highly treated and disinfected water for laboratory usage. The distillation process removes minerals and microbiological contaminants and can reduce levels of chemical contaminants. A water distiller works by boiling water into water vapour, condensing it and then returning it to its liquid state. It is collected in a storage container.



Municipal or well water is manually or automatically fed into the distiller unit's boiling chamber. A heating element in the boiling chamber heats the water until it boils. The steam rises from the boiling chamber. Volatile contaminants (gases) are discharged through a built-in vent. Minerals and salts are retained in the boiling chamber as hard deposits or scale. The steam enters a coiled tube (condenser), which is cooled by cool water. Water droplets form as condensation occurs. The distilled water is 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 consists of flask with heating elements embedded in glass and fused in spiral type coil internally of the bottom and tapered round glass, joints at the top double walled condenser with B-40/B-50 ground glass joints, suitable to work on 220 volts, 50 cycles AC supply.

Fractional Distillation Apparatus

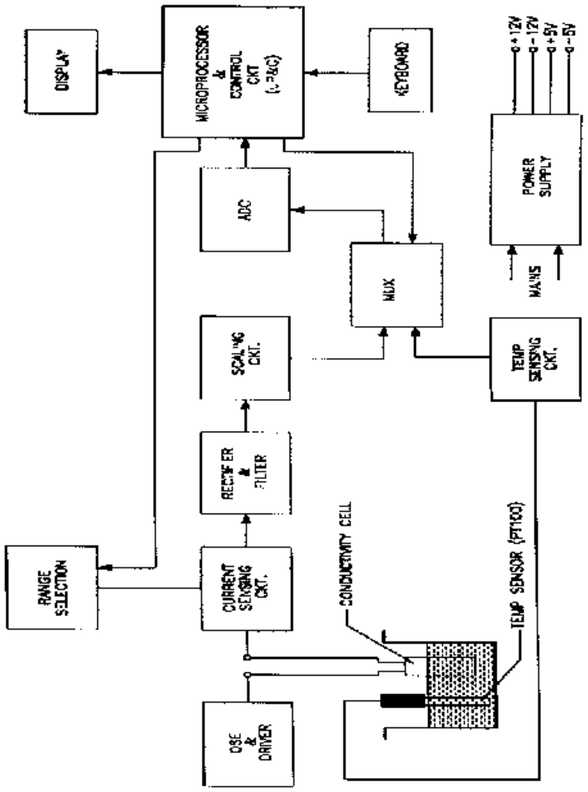


3.2.8. CONDUCTIVITY MEASUREMENT

Conductivity measurement was done using Systronics Conductivity TDS meter-308. It can provide both automatic and manual temperature compensation.

The conductance measurements were carried out on this conductivity bridge using a dip-type immersion conductivity cell of cell constant $1.11 cm^{-1}$. The entire conductance data were reported at 1 KHz and was found to be ±0.3 % precise. The instrument was standardized using 0.1(M) KCl solution. The cell was calibrated by the method of Lind and co-workers [12]. 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 prevent admission of air into the cell when solvent or solution was added. 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 [13].

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.



Block Diagram of the Instrument

3.2.9. REFRACTIVE INDEX MEASUREMENT

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

Calibration was performed by measuring the refractive indices of double-distilled water, toluene, cyclohexane, and carbon tetrachloride at defined temperature. The accuracy of the instrument is +/- 0.0005. 2-3 drops of the sample was put onto the measurement cell and the reading was taken. The refractive index of a sample depends on temperature. During measurement, refractometer determines the temperature and then corrects the refractive index to a temperature as desired by the user.

3.2.10. SURFACE TENSION

The surface tension experiments were done by platinum ring detachment mehod using a Tensiometer (K9, KRŰSS; Germany) at the experimental temperature. The accuracy of the measurement was within ±0.1mN.m^{-1}. Temperature of the system has been maintained by circulating auto-thermostated water through a double-wall glass vessel containing solution.

3.2.11. pH METER

The pH values of the experimental solutions were measured by a Mettler Toledo Seven Multi pH meter.

3.2.12. NUCLEAR MAGNETIC RESONANCE SPECTRA MEASUREMENT

Nuclear Magnetic Resonance (NMR) spectroscopy is used to study the structure of molecules, the kinetics or dynamics of molecules and the composition of mixtures of biological or synthetic solutions or composites. ^{1}H NMR spectra were recorded in D_2O . ^{1}H NMR spectra were recorded at 300 MHz using Bruker Avance

300 MHz instrument. 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.

3.2.13. ULTRAVIOLET-VISIBLE SPECTRA MEASUREMENT

Compounds that absorb Ultraviolet and or visible light have characteristic absorbance curves as a function of wavelength. Absorbance of different wavelengths of light occurs as the molecules move to higher energy states.

The UV-VIS spectrophotometer uses two light sources, a deuterium lamp for ultraviolet light and a tungsten lamp for visible light. After bouncing of a mirror, the light beam passes through a 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) light successfully passes through a slit. A filter is used to remove unwanted higher order diffraction. The light beam hits the second mirror before it gets split by a half mirror (half of the light is reflected, other half passes through). One of the beams is allowed to pass through the reference cuvette (which contains the solvent only), the other passes through the sample cuvette. The intensities of the light beams are then measured at the end. Regarding this the Beer-Lambert law has been obeyed.

Beer-Lambert law

The change in intensity 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 the sample, more molecules absorbing means more photons absorbed,

(iii) Intensity of incident light (I), more photons mean more opportunity for a molecule to see a photon.

Thus, dI is proportional to bcI or dI/I = -kbc (where k is the proportionality constant, the negative sign is shown due to a decrease in intensity of the light, this makes b, c and I always positive. Integration of the above equation leads to Beer-Lambert's law:

$$-\ln I/I_0 = kbc \qquad (2)$$

$$-\log I/I_0 = 2.303\, kbc \qquad (3)$$

$$\varepsilon = 2.303\, k \qquad (4)$$

$$A = -\log I/I_0 \qquad (5)$$

$$A = \varepsilon bc \qquad (6)$$

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 wavelength.

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