CHAPTER-III

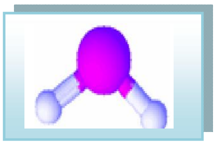
EXPERIMENTAL SECTION

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

III.1.1 SOLVENTS

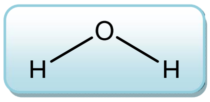
The details of the aqueous and non-aqueous solvents used in the research work are given below:

Water (H_2O):



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

WATER

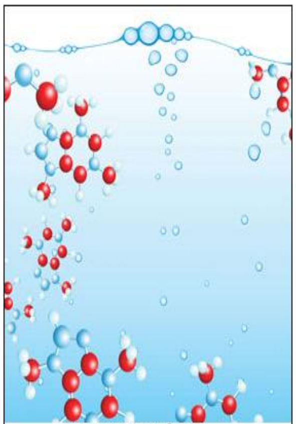


Appearance	*:Liquid*
Molecular Formula	*:H_20*
Molecular Weight	*:18.02 g·mol^{-1}*
Boiling Point	*:373 K*
Melting Point	*:273 K*
Dielectric Constant	*:78.35 at 298.15K*

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

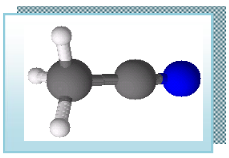
Purification: Water was first deionised and then distilled in an all glass distilling set along with alkaline $KMnO_4$ solution to eradicate any organic matter therein. The doubly distilled water was finally distilled using an all glass distilling set. Precautions were taken to prevent contamination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

Application: Water is extensively used in chemical reactions as a solvent or reactant and fewer commonly as a solute or catalyst. In inorganic reactions, water is a common solvent, dissolving many ionic compounds. In recent times s has been a topic of research. Oxygen saturated supercritical water combusts organic pollutants proficiently. It is also use in various industries. It is a superb solvent, generally taken as the widespread solvent, due to the discernible 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 originate in water, all life on earth is thought to have arisen from water and the bodies of all living organisms are composed chiefly of water. About 70 to 90 percent of all organic substance is water.


water as a solvent

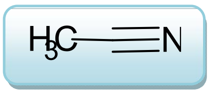
The chemical reactions in all plants and animals that support life take place in a water medium. Water not only provides the medium to compose these life sustaining reactions possible, but water itself is often an important reactant or product of these reactions. In short, the chemistry of life is water chemistry.

Acetonitrile (ACN):



Acetonitrile is the colourless liquid and of the simplest organic nitrile. It is produced mainly as a byproduct of acrylonitrile manufacture.

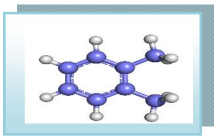
Source: Merck, India.

Acetonitrile	
	
Appearance	*:Colourless Liquid*
Molecular Formula	*:CH_3CN*
Molecular Weight	*:41.05 g·mol^{-1}*
Boiling Point	*:628.3 K*
Melting Point	*:607.5 K*
Dielectric Constant	*:35.95 at 298.15 K*

Purification: Acetonitrile (ACN) obtained from Merck, India was used after further purification. It was distilled from P_2O_5 and then from CaH_2 in an all-glass distillation apparatus [1]. The middle fraction was collected. About 99% purified acetonitrile with specific conductivity 0.8 – 1.0×10^{-8} S cm^{-3} was obtained. The purity of the liquid was checked by measuring it's density and viscosity which were in good agreement with the literature values [1,2] as shown in Table X.1.

Application: It is widely used in battery applications because of its relatively high dielectric constant and capability to dissolve electrolytes. For similar reasons it is a popular solvent in cyclic voltammetry. Its low viscosity and low chemical reactivity make it a popular choice for liquid chromatography. Acetonitrile plays a noteworthy role as the dominant solvent used in the manufacture of DNA oligonuleotides from monomers. Industrially, it is used as a solvent in the purification of butadiene and in the construct of pharmaceuticals and photographic film. Acetonitrile is a common two-carbon building block in organic synthesis as in the production of pesticides to perfumes.

o-Xylene:

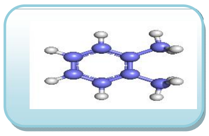


o-Xylene (ortho-xylene) is an aromatic hydrocarbon, based on benzene with two methyl substituents bonded to adjacent carbon atoms in the aromatic ring (the ortho configuration).

Source: Sisco Research Laboratory Pvt. Ltd., Mumbai, India.

Purification: It was dried by passing through molecular sieves. [3]

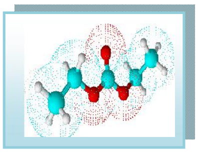
O-Xylene



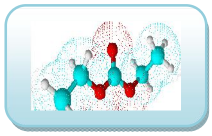
Appearance	*:Colourless Liquid*
Molecular Formula	*:C_8H_{10}*
Molecular Weight	*:106.17 g·mol^{-1}*
Boiling Point	*:417.5 K*
Melting Point	*:249 K*
Dielectric Constant	*:2.60 at 298.15 K*

Application: *o*-Xylene is largely used in the production of phthalic anhydride, and is generally extracted by distillation from an assorted xylene stream in a plant primarily designed for *p*-xylene production.

Diethyl carbonate (DEC):



Diethyl carbonate (DEC) is a carbonate ester of carbonic acid and ethanol having the formula $OC(OCH_2CH_3)_2$. At room temperature (25 °C) diethyl carbonate is a clear liquid with a low flash point.

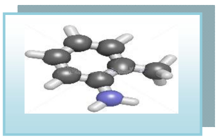
Diethyl carbonate 	
Appearance	*:Clear Liquid*
Molecular Formula	*:$C_5H_{10}O_3$*
Molecular Weight	*:118.13 g·mol^{-1}*
Boiling Point	*:401 K*
Melting Point	*:230 K*
Dielectric Constant	*:2.83 at 298.15 K*

Source: Sisco Chem., India.

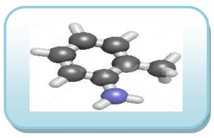
Purification: It was dried by passing through molecular sieves. [3]

Application: Diethyl carbonate is used as a solvent such as in erythromycin intramuscular injections. It can be used as a component of electrolytes in lithium batteries.

o-Toluidine:



o-Toluidine (ortho-toluidine) is an organic compound with the chemical formula C_7H_9N. This arylamine is a colorless to pale-yellow liquid with a poor solubility in water.

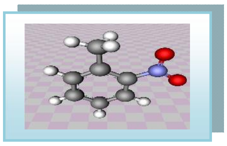
O-Toluidine 	
Appearance	*:Colourless or Pale Yellow Liquid*
Molecular Formula	*:C_8H_9N*
Molecular Weight	*:107.16 g·mol^{-1}*
Boiling Point	*:475K*
Melting Point	*:249.5 K*
Dielectric Constant	*:6.14 at 298.15 K*

Source: Merck, Mumbai, India.

Purification: It was dried by passing through molecular sieves. [3]

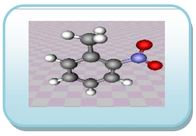
Application: *o*-Toluidine is used or applied in different circumstances. It is used mainly for dye, especially for coloring hair. The other usages of *o*-toluidine are specific determination of glucose in blood and the most recent one, the separation of toxic metal ions, which is still in the research phase.

2-Nitrotoluene:



2-Nitrotoluene or ortho-nitrotoluene is a pale yellow liquid. It can be made by nitrating toluene at above -10 °C. 2-Nitrotoluene is a nitroaromatic compound. Multi-functional magnetic resins having elevated surface area and ion exchange efficiency were developed and were tested for desorption of 2-nitrotolene. Its dioxygenation by nitrobenzene dioxygenase has been studied to assess the isotope fractionation. It has been reported as nitroaromatic explosive. Terbium-based magnetic metal-organic framework (MOF) nanospheres have been fabricated, which can detect the trace quantities of 2-nitrotoluene.

2-Nitrotoluene



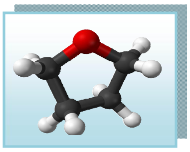
Appearance	*:Yellow Liquid*
Molecular Formula	*:*$C_7H_7NO_2$
Molecular Weight	*:137.14 g·mol*$^{-1}$
Boiling Point	*:495K*
Melting Point	*:262.8 K*
Dielectric Constant	*:26.10 at 298.15 K*

Source: Sisco Chem. Industries, Mumbai, India.

Purification: It was dried by passing through molecular sieves. [3]

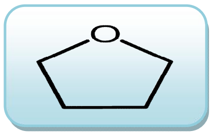
Application: 2-Nitrotoluidine solvent is largely used in the production of phthalic anhydride, pigments and antioxidants. 2-Nitrotoluene may be employed as nitrogen supplement in the culture medium of Pseudomonas sp. strain ClS1. It may be utilized as carbon and energy supplement in the culture medium of Pseudomonas sp. strain JS42.

Tetrahydrofuran (THF):



Tetrahydrofuran (**THF**) is an organic compound having formula $(CH_2)_4O$. The compound is classified as heterocyclic compound, specifically a cyclic ether. It is a colorless, water-miscible organic liquid with low viscosity. THF has an odor akin to acetone. It is mainly used as a precursor to polymers. Being polar and having a wide liquid range, THF is a versatile solvent.

Tetrahydrofuran



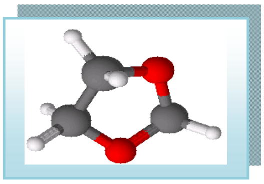
Appearance	*:Colourless Liquid*
Molecular Formula	*:C_4H_8O*
Molecular Weight	*:72.11 $g{\cdot}mol^{-1}$*
Boiling Point	*:339 K*
Melting Point	*:164.8 K*
Dielectric Constant	*:7.58 at 298.15 K*

Source: Merck, Indian.

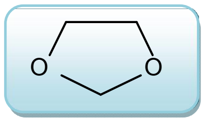
Purification: Tetrahydrofuran (THF), Merck, Indian was kept several days over potassium hydroxide (KOH), refluxed for 24 h and distilled over lithium aluminium hydride ($LiAlH_4$) described earlier [4]. The purified solvent had a boiling point of 339 K and a specific conductance of 0.81×10^{-6} S cm^{-3}. The density and viscosity of the purified solvent were in good agreement with the literature data [5,6] as shown in Table X.1. The purity of the solvent was ≥98.9%.

Application: The main application of THF is as an industrial solvent for PVC and in varnishes. It is an aprotic solvent with a fairly polar solvent and can dissolve a wide range of non-polar and polar chemical compounds. THF is a popular solvent in the laboratory when a moderately higher-boiling ethereal solvent is required and its water miscibility is not an issue. Hence, like diethyl ether, THF can be used in hydroboration reactions to synthesize primary alcohols, and as a solvent for organometallic compounds such as organolithium and Grignard reagents. THF is often used in polymer science as dissolve polymers prior to determining their molecular mass using gel permeation chromatography, to PVC as well and thus it is the main ingredient in PVC adhesives. It can be used to liquefy old PVC cement, and is often used industrially to degrease metal parts. THF is also used as a component in mobile phases for reversed-phase liquid chromatography.

1,3-Dioxolane (1,3-DO):



Dioxolane or 1,3-dioxolane is a heterocyclic acetal. No unusual toxic effects have been associated with the exploit of 1,3-dioxolane. The product is not explosive, not spontaneously flammable and has no disagreeable odour. Dioxolanes are a group of organic compounds sharing the dioxolane ring structure.

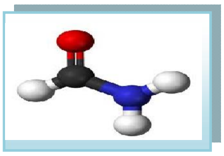
1,3-Dioxolane	
	
Appearance	*:Colourless Liquid*
Molecular Formula	*:C_3H_6O*
Molecular Weight	*:74.08 g·mol^{-1}*
Boiling Point	*:348 K*
Melting Point	*:178 K*
Dielectric Constant	*:7.34 at 298.15 K*

Source: Merck, India.

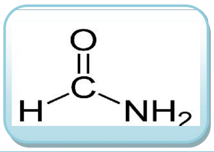
Purification: It is dried with KOH and then distilled from sodium [3].

Application: It is a very good solvent for pharmaceutical manufacturing, it is used as a substitute for many chlorinated solvents, in lithium battery electrolyte solvent component, as a copolymerization agent with trioxane and formaldehyde for manufacturing polyacetal resins, paint stripper, water solubilizing agent for pesticides, glue stabilizer, herbicides and wood preservatives.

Formamide (FA):



Formamide, also known as **methanamide**, is an amide derived from formic acid. It is a clear liquid which is miscible with water and has an odor akin to ammonia. It is chemical feedstock for the manufacture of sulfa drugs, other pharmaceuticals, herbicides, pesticides and the manufacture of hydrocyanic acid.

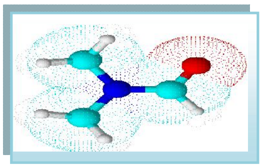
Formamide	
	
Appearance	*:Colourless Liquid*
Molecular Formula	*:$HCONH_2$*
Molecular Weight	*:45.04 g·mol^{-1}*
Boiling Point	*:483 K*
Melting Point	*:275-276 K*
Dielectric Constant	*:109.5 at 298.15 K*

Source: Sigma Adrich, Germany

Purification: The Spectrographic grade formamide used as procured, without further purification. The purity of the solvent is 99.5%.

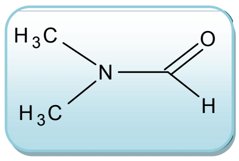
Application: It has been used as a softener for paper and fiber. It is a solvent for many ionic compounds. It has also been used as a solvent for resins and plasticizers. Formamide will commence to partially decompose into carbon monoxide and ammonia at 180°C. When heated strongly, formamide decomposes to hydrogen cyanide (HCN) and water vapor. It is also a ingredient of cryoprotectant vitrification mixtures used for cryopreservation of tissues and organs. Formamide is also used as an RNA stabiliser in gel electrophoresis by deionizing RNA. In capillary electrophoresis, it is employed for stabilizing (single) strands of denatured DNA. Another use is to add it in sol-gel solutions in order to avoid cracking during sintering. Formamide, in its pure state, has been utilized as an alternative solvent for the electrostatic self-assembly of polymer nano-films. It is used to prepare primary amines directly from ketones via their N-formyl derivatives, using the Leuckart reaction.

N,N-Dimethylformamide (DMF):



N, N, Dimethylformamide is an organ ic compound with the formula $(CH_3)_2NC(O)H$. Commonly abbreviated as DMF (though this acronym is sometimes employed for dimethylfuran), this colourless liquid is miscible with water and the majority of organic liquids. DMF is a common solvent for chemical reaction. Pure dimethyl-formamide is odorless while technical grade or degraded dimethylformamide often has a fishy smell due to impurity of dimethylamine. Its name is derived from the fact that it is a derivative of formamide,

N,N-Dimethylformamide



Appearance	*:Colourless Liquid*
Molecular Formula	$:(CH_3)_2NCHO$
Molecular Weight	*:73.09 g·mol*$^{-1}$
Boiling Point	*:425-427 K*
Melting Point	*:212.7 K*
Dielectric Constant	*:36.71 at 298.15 K*

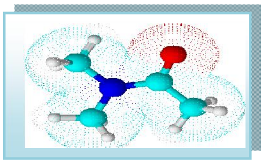
the amide of formic acid. DMF exists as polar (hydrophilic) aprotic solvent with a high boiling point. It facilitates reactions that follow polar mechanisms, such as S_N2 reactions.

Source: Thomas Baker, India

Purification: In case of purification, it was dried by passing through Linde 4Å molecular sieves and then distilled [3].

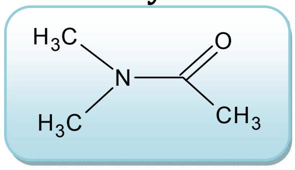
Application: The primary use of dimethylformamide is as a solvent with small evaporation rate. DMF is used in the production of acrylic fibers and plastics. It is also used as a solvent in peptide coupling for pharmaceuticals, in the development and production of pesticides, adhesives, synthetic leathers, fibers, films, and surface coatings [7]. It is used as a reagent in the Bouveault aldehyde synthesis and in the Vilsmeier-Haack reaction, another valuable method of forming aldehydes. It is also a common catalyst used in the synthesis of acyl halides, in particular the synthesis of acyl chlorides from carboxylic acids using oxalyl or thionyl chloride [8]. DMF penetrates most plastics and makes them swell. This property makes it very suitable for solid phase peptide synthesis. It also frequently occurs as a constituent of paint strippers for this purpose. DMF is very effective at separating and suspending carbon nanotubes, and is recommended by the NIST for use in near infrared spectroscopy of such. DMF can be employed as a standard in proton NMR allowing for a quantitative determination of an unknown chemical. DMF is used as a solvent to recover olefins such as 1,3-butadiene via extractive distillation. It is also utilized in the manufacturing of solvent dyes as an important raw material. It is consumed during reaction. Pure acetylene gas cannot be compressed and stored devoid of the danger of explosion. Industrial acetylene gas is, therefore, dissolved in dimethylformamide and stored in metal cylinders or bottles. The casing is also filled with agamassan, which provides it safe to transport and use.

N,N-Dimethylacetamide (DMA):



N,N-Dimethylacetamide is the organic compound with the formula $(CH_3)_2NCOCH_3$. This colorless, water-miscible, high boiling liquid is usually used as a polar solvent in organic synthesis. DMA, as it often abbreviated, is miscible with most other solvents, although it is feebly soluble in aliphatic hydrocarbons.

N,N-Dimethylacetamide



Appearance	*:Colourless Liquid*
Molecular Formula	*:$(CH_3)_2NCOCH_3$*
Molecular Weight	*:87.12 $g{\cdot}mol^{-1}$*
Boiling Point	*:438.2 K*
Melting Point	*:253 K*
Dielectric Constant	*:37.78 at 298.15 K*

Source: Thomas Baker, India

Purification: It was dried by ephemeral through molecular sieves [3].

Application: DMA is useful solvent for reactions involving strong bases such as sodium hydroxide. Dimethylacetamide is normally used as a solvent for fibers (e.g., polyacrylonitrile, spandex) or in the adhesive industry [9]. It is also employed in the production of pharmaceuticals and plasticizers as a reaction medium.

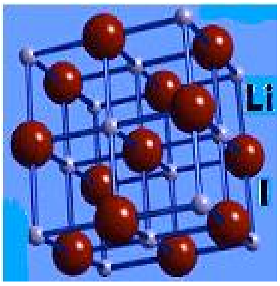
III.1.2 ELECTROLYTES AND NON-ELECTROLYTES

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

III.1.2.1 Ionic Solids

Lithium Iodide

Lithium iodide, or LiI, is a compound of lithium and iodine. When exposed to air, it becomes yellow in color, Owing to the oxidation of iodide to iodine. It crystallizes as FCC lattice.



Appearance: White crystalline solid

M.F. LiI

M.W. 133.85 g/mol

M.P. 742 K

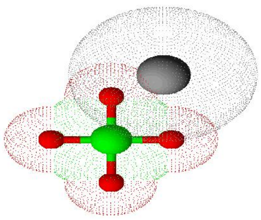
Source: Sigma Aldrich, Germany

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

Application: Lithium iodide is used as an electrolyte for high temperature batteries. It is also employed for long life batteries as required, for example, by artificial pacemakers. The solid is used as a phosphor for neutron detection. It is also used, in a complex with Iodine, in the electrolyte of dye-sensitized solar cells.

Lithium Perchlorate

Lithium perchlorate is the inorganic compound with the formula $LiClO_4$. This white or colourless crystalline salt is noteworthy for its high solubility in a lot of solvents. It exists both in anhydrous form and as a trihydrate.



Appearance: White crystalline solid

M.F. $LiClO_4$

M.W. 106.39 g/mol

M.P. 509 K

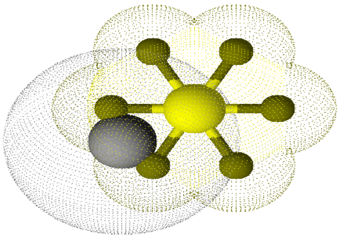
Source: Sigma Aldrich, Germany

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

Application: Lithium perchlorate is used as an electrolyte in lithium-ion batteries. Lithium perchlorate is also used as a source of oxygen in some chemical oxygen generators. In organic chemistry Lithium perchlorate is also used as a co-catalyst in the coupling of α,β-unsaturated carbonyls with aldehydes

Lithium Hexafluoarsenate

Lithium hexafluoarsenate is the inorganic compound with the formula $LiAsF_6$.



Appearance: Powder

M.F. $LiAsF_6$

M.W. 195.85 g/mol

M.P. 622 K

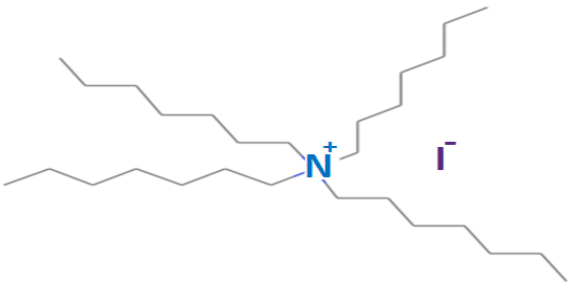
Source: Sigma Aldrich, Germany

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

Application: The surface chemistry of Li electrodes in ethereal $LiAsF_6$ was tested with new salts for potential battery systems. It is also used in the preparation of high energy batteries and lithium-ion batteries.

Tetraheptylammonium Iodide ($Hept_4NI$)

Tetrahptylammonium Iodide can be called as various other names such as, 1-Heptanaminium, N,N,N-triheptyl-, iodide; Ammonium, tetraheptyl-, iodide; Tetra-n-heptylammonium iodide etc. It contains a tertiary nitrogen atom which is linked with four heptyl (containing seven carbon atoms) groups.



Appearance: White crystalline solid

M.F. $C_{28}H_{60}NI$

M.W. 537.69 g/mol

M.P. 394 K-397 K

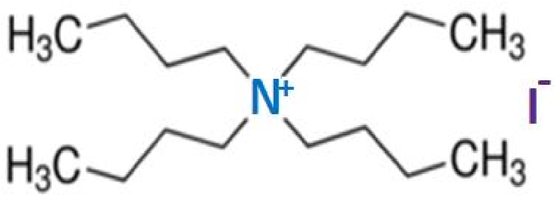
Source: Sigma Aldrich, Germany

Purification: Used as purchased. purity of the ionic solid is >99.0%

Application: $Hept_4NI$ used in this study has a number of applications such as it employed as phase transfer catalyst. Since this ionic solid has the tendency of locating at the interface of two phases (liquid–liquid or solid–liquid) for introducing the continuity between two different phases, so it positively is said as phase transfer catalyst. It is also used as osmolytes.

Tetrabutylammonium Iodide (But_4NI)

Tetrabutylammonium Iodide is an anmmonium based ionic solid having molecular formula $(CH_3CH_2CH_2CH_2)_4N(I)$. It contains a tertiary nitrogen atom which is linked with four butyl (containing four carbon atoms) groups.



Appearance: White crystalline solid

M.F. $C_{16}H_{36}NI$

M.W. 369.67 g/mol

M.P. 414 K-416 K

Source: Sigma Aldrich, Germany

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

Application: The ionic solid has immense appliances in various fields such as in chemical reactions it acts as surface-active agents (due to the hydrophobic communications between the butyl groups and water molecules), solvents, Intermediates emulsifying means, pigment disperse and phase transfer catalyst [10,11]. Since but_4NI has the tendency of locating at the interface of two phases (liquid–liquid or solid–liquid) for introducing the continuity between two different phases, so it positively is said as phase transfer catalyst. Due to low cost and low toxicity of but_4NI in recent times it has appeared as a promising substitute as a catalyst for functionalization of C–H bonds [12,13]. In industrial usage but_4NI acts as active ingredient for conditioners, antistatic agent, detergent sanitizers, softener for textiles and paper products etc. In medicinal field it uses as antimicrobials, algaecide, slimicidal agents, disinfection agents and sanitizers etc.

III.1.2.2 Ionic Liquids

1-Butyl-1-methylpyrrolidinium bromide

1-butyl-1-methylpyrrolidinium bromide is a pyrrolidinium based ionic liquid, having molecular formula $C_9H_{20}BrN$, which contains methyl and ethyl group with an active nitrogen atom in a five membered pyrrolidinium ring. This IL exists as a molten solid phase (white crystalline).

Appearance	***: White Crystalline***
Molecular Formula	***: $C_9H_{20}BrN$***
Molecular Weight	***: 222.17 $g{\cdot}mol^{-1}$***

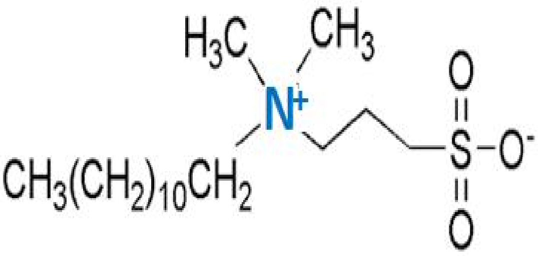
Source: Sigma Aldrich, Germany

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

Application: This ionic liquid is an excellent example of neoteric solvents, novel types of solvents, or older resources that are finding new appliances as environmentally friendly (or eco-friendly) solvents, because of having their less vulnerability for human being as well as fewer toxicity for living organisms. This IL also used as recyclable solvents for organic reactions and separation processes, lubricating fluids, heat transfer fluids for processing biomass and electrically conductive liquids as electrochemical gadget (such as batteries and solar cells) in the field of electrochemistry.

N-Dodecyl N,N-dimethyl-3-ammonio-1-propanesulfonate

N-Dodecyl N,N-dimethyl-3-ammonio-1-propanesulfonate is a ammonio group based surface active ionic liquid (SAIL) which contains a very long dodecyl (having 12 carbon atoms) alkyl chain, two methyl groups, a tertiary nitrogen atom and a propanesulfonate group. This IL exists as a molten solid phase (white crystalline).



Appearance	: White Crystalline
Molecular Formula	: $C_{17}H_{37}NO_3S$
Molecular Weight	: 335.50 g·mol^{-1}

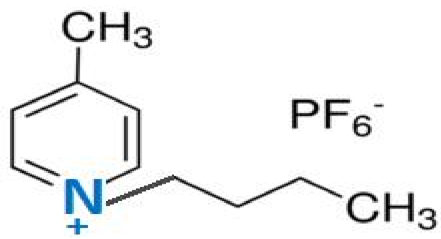
Source: Sigma Aldrich, Germany

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

Application: Their relatively rapid emergence as alternative solvents has involved a rapidly growing number of examples of the application such as organic synthesis, chemical reactions, chemical separations, and material preparations [14,15]. ILs are composed of sterically mismatched ions that hinder crystal formation, thus molecular structure can be used to tune physicochemical properties. The design and synthesis of functional ILs that integrate structural or functional groups have been reported. ILs was designed as oriented solvents which could impact selectivity in reactions by ordering reactants [16]. Furthermore, functional ILs were also used as templates for the synthesis of mesoporous and zeolitic materials [17] and in the formation of ordered thin films [18,19]. Recently, ILs having a long alkyl chain group exhibited surface active properties in their aqueous solutions. Considering the special structures and properties of IL surfactant (zwitterionic detergent employed for protein solubilization), it is of interest to investigate their complexation behaviour using different techniques.

1-Butyl-4-methylpyridinium hexafluorophosphate

1-butyl-4-methylpyridinium hexafluorophosphate is basically a pyridinium based ionic liquid. It contains one butyl and one methyl attached with the nitrogen atom of group as cationic part, then again, hexafluorophosphate as an anionic part.



Appearance	*: White Crystalline*
Molecular Formula	*: $C_{17}H_{37}NO_3S$*
Molecular Weight	*: 335.50 g·mol^{-1}*

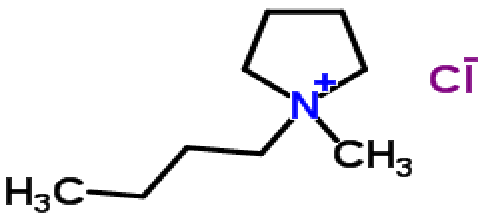
Source: Sigma Aldrich, Germany

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

Application: This ionic liquid is an excellent example of neoteric solvents, novel types of solvents, or older resources that are finding new appliances as environmentally friendly (or eco-friendly) solvents, because of having their less vulnerability for human being as well as fewer toxicity for living organisms. The IL can selectively dissolve and remove gases and could be used for air purification on submarines and spaceships. It is also used in the present widely used in solvent extraction, liquid–liquid extraction process, electrochemical studies, dye-sensitized solar cells [20-23].

1-butyl-1-methylpyrrolidinium chloride

1-butyl-1-methylpyrrolidinium chloride is a pyrrolinium based ionic liquid, having molecular formula $C_9H_{20}ClN$, which contains methyl and ethyl group with an active nitrogen atom in a five membered pyrrolidinium ring. This IL exists as a molten solid phase (white crystalline).



Appearance	*: White Crystalline*
Molecular Formula	*: $C_9H_{20}ClN$*
Molecular Weight	*: 177.71 g·mol^{-1}*

Source: Sigma Aldrich, Germany.

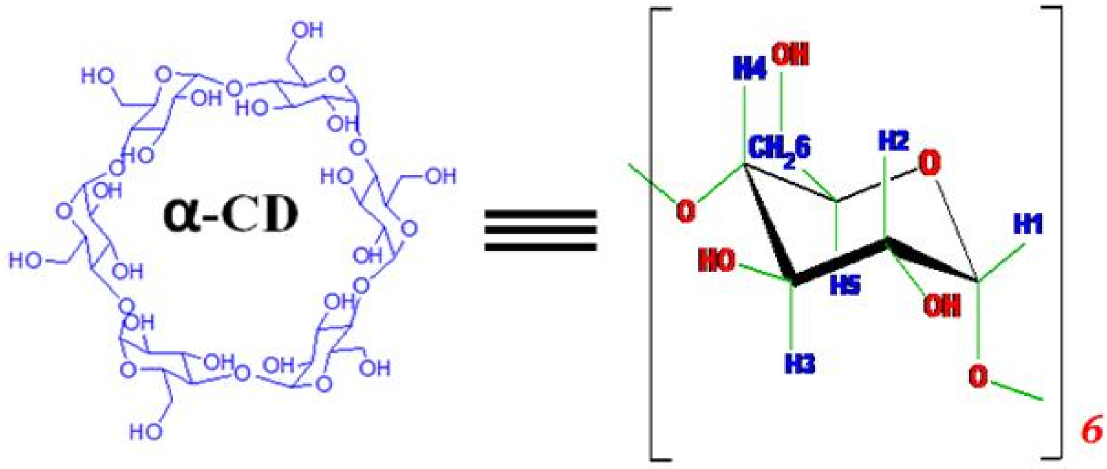
Purification: Used as purchased. The purity of the ionic liquid is >99.0%

Application: This ionic liquid is an excellent example of neoteric solvents, novel types of solvents, or older resources that are finding new appliances as environmentally friendly (or eco-friendly) solvents, because of having their less vulnerability for human being as well as fewer toxicity for living organisms. This IL also used as recyclable solvents for organic reactions and separation processes, lubricating fluids, heat transfer fluids for processing biomass and electrically conductive liquids as electrochemical gadget (such as batteries and solar cells) in the field of electrochemistry.

Non-Electrolytes

Alpha Cyclodextrin (α-CD)

α-CD is naturally occurring polysachharides of six glucose units and they are covalently attached via end to end α-1,4 linkage. It has hydrophobic inner cavity and hydrophilic outer surface. In aqueous medium hydrophobic inner core allow it to form host-guest inclusion colmplex with suitable hydrophobic molecules. Most of the cases it forms 1:1 inclusion complex due to its small cavity volume.



Appearance:	Crystalline Powder
Molecular Formula:	$C_{36}H_{60}O_{30}$
Molecular Weight:	972.85 g/mol
Melting Point:	>551 K

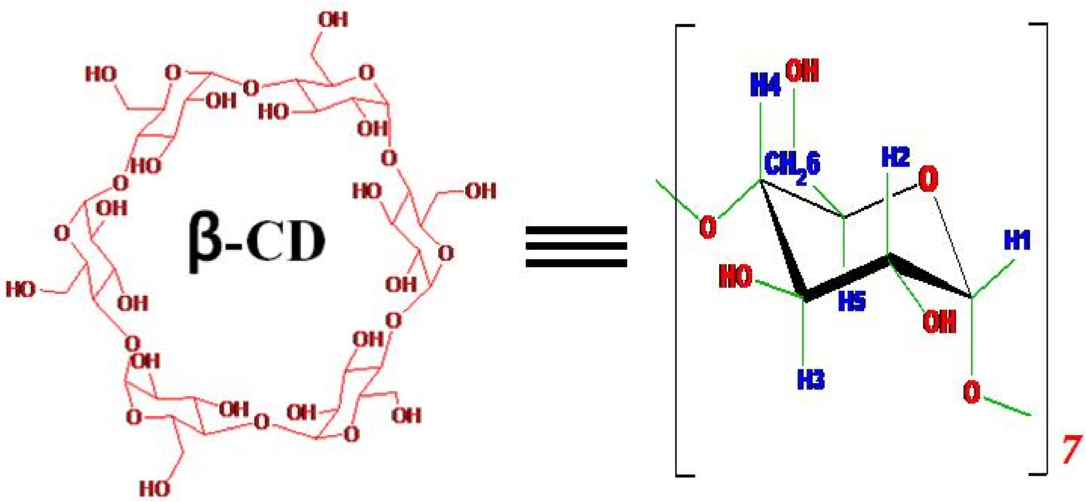
Source: Sigma Aldrich, Germany

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

Application: α-Cyclodextrin is widely applied in production of medicine and food. It also used in cosmetics, paint, textile industries. 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. α-cyclodextrin is commonly used as a complexing agent in hormones, vitamins, and many bioactive compounds frequently used in tissue and cell culture applications.

Beta Cyclodextrin (β-CD)

β-Cyclodextrin is finely made from pure provision material-starch and translate enzyme, which is white powder and whose molecule structure is like a cylinder compounded from 7 glucose group with a key of 2-1.4. The function of β-Cyclodextrin depends on its cylinder molecule structure which can be easy to integrate other materials. That feature is applied widely in industry



Appearance:	Crystalline Powder
Molecular Formula:	$C_{42}H_{70}O_{35}$
Molecular Weight:	1134.98 g/mol
Melting Point:	563.15-573.15 K
Boiling Point	1814.33 K

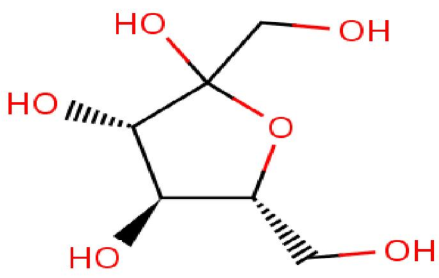
Source: Sigma Aldrich, Germany

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 improved 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. 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 most commonly used as a complexing agent in hormones, vitamins, and many compounds frequently used in tissue and cell culture applications. This aptitude 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 antagonistic environment.

D(-)Fructose

Living system of every animal and man is composed of several molecules having specific functions are termed as biomolecules. Carbohydrates are one of the main classes of biomolecules. D(-)Fructose is one of the most important biomolecule. Fructose, or fruit sugar, is a simple ketonic monosaccharide found in lots of plants, where it is often bonded to glucose to form the disaccharide sucrose.



Appearance:	**Crystalline Powder**
Molecular Formula:	$C_6H_{12}O_6$
Molecular Weight:	**180.16 g/mol**
Melting Point:	**376 K**

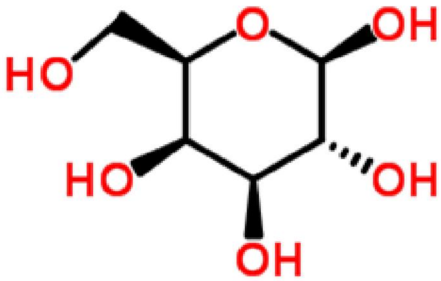
Source: Thomas Baker, Mumbai

Purification: Used as parched. The purity of the sample is 99.4%.

Application: D(-)Fructose usually act as a ubiquitous fuel for biological processes to supply necessary energy for the function of the living and their day's work. An unusual level of carbohydrate in human body fluid is a caution hint of a medical stipulation. Such as, an unbalanced concentration of carbohydrates in human blood or urine entails a biological dysfunction.

D(+)Galactose

Living system of every animal and man is composed of several molecules having specific functions are termed as biomolecules. Carbohydrates are one of the main classes of biomolecules. D(+)Galactose is one of the most important biomolecule. It is a monosaccharide sugar that is less sweet than glucose and fructose. It is a C-4 epimer of glucose. Galactan is a polymeric form of galactose found in hemicellulose.



Appearance:	**Crystalline Powder**
Molecular Formula:	**$C_6H_{12}O_6$**
Molecular Weight:	**180.16 g/mol**
Melting Point:	**440 K**

Source: Thomas Baker, Mumbai

Purification: Used as parched. The purity of the sample is 99.9%.

Application: D(+)Galactose usually act as a ubiquitous fuel for biological processes to supply necessary energy for the function of the living and their day's work. An unusual level of carbohydrate in human body fluid is a caution hint of a medical stipulation. Such as, an unbalanced concentration of carbohydrates in human blood or urine entails a biological dysfunction.

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. PREPARATION OF SOLVENT MIXTURES (MIXED SOLVENTS)

The research work has been carried out with binary or ternary solvent systems with acrylonitrile, tetrahydrofuran, methanol, ethylene glycol, 1,3-dioxolane etc. as primary solvents with some polar, weakly polar and non-polar solvents as well as with some electrolytes (ionic liquids & other electroluytes) and non-electrolytes (amino acids and other solutes).

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.

Different types of binary and ternary solutions have been prepared and used for my research studies.

III.2.4 MEASUREMENTS OF EXPERIMENTAL PROPERTIES

III.2.4.1 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.

Mettler Toledo, AG 285

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	*: LocalCAN 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.2.4.2 CONDUCTIVITY MEASUREMENT

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

Systronics Conductivity-TDS meter 308 is a microprocessor based instrument used for measuring specific conductivity of solutions. It can provide both automatic and manual temperature compensation. The instrument shows the conductivity of the solution under test at the existing temperature or with temperature compensation. Provision for storing the cell constant and the 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 recalibrating the instrument even after switching it off.

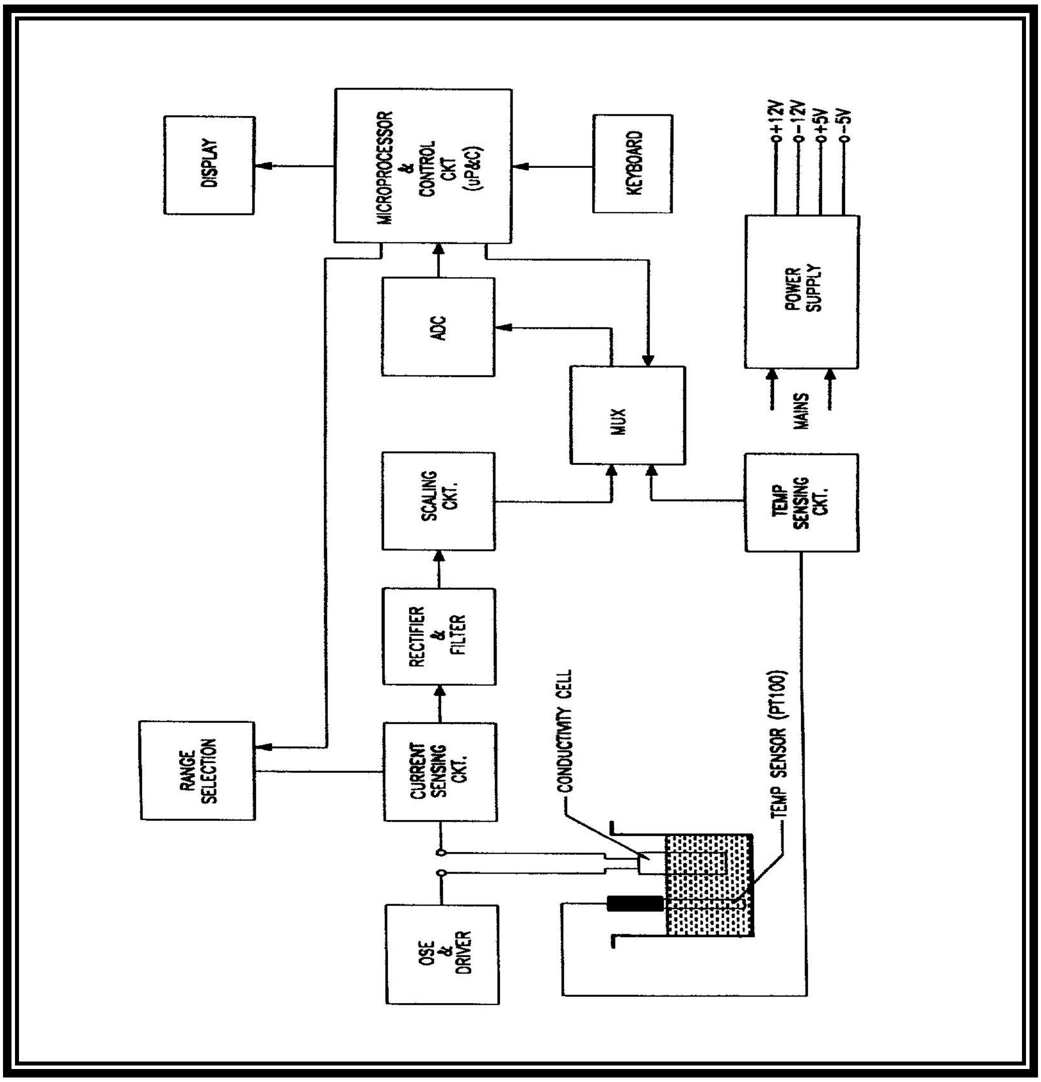
Systronic-308 Conductivity Bridge

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 [24]. and cell constant was measured based on 0.01 M aqueous KCl solution [25]. 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 and the accuracy was ±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 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 [26].

Instrument Specifications:

Frequency	*:100 Hz or 1 KHz Automatic*
Conductivity	
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^oC to 100^oC (Auto/Manual)*
Accuracy	*: ± 0.2 ^oC ±1 digit*
Resolution	*: 0.1 ^oC*
Cell Constant	*: Acceptable from 0.1 to 5.0*
Auto Temp. Compensation	*: 0^oC to 100^oC with PT 100 sensor*
Manual Temp. Compensation	*: 0^oC to 60^oC 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*

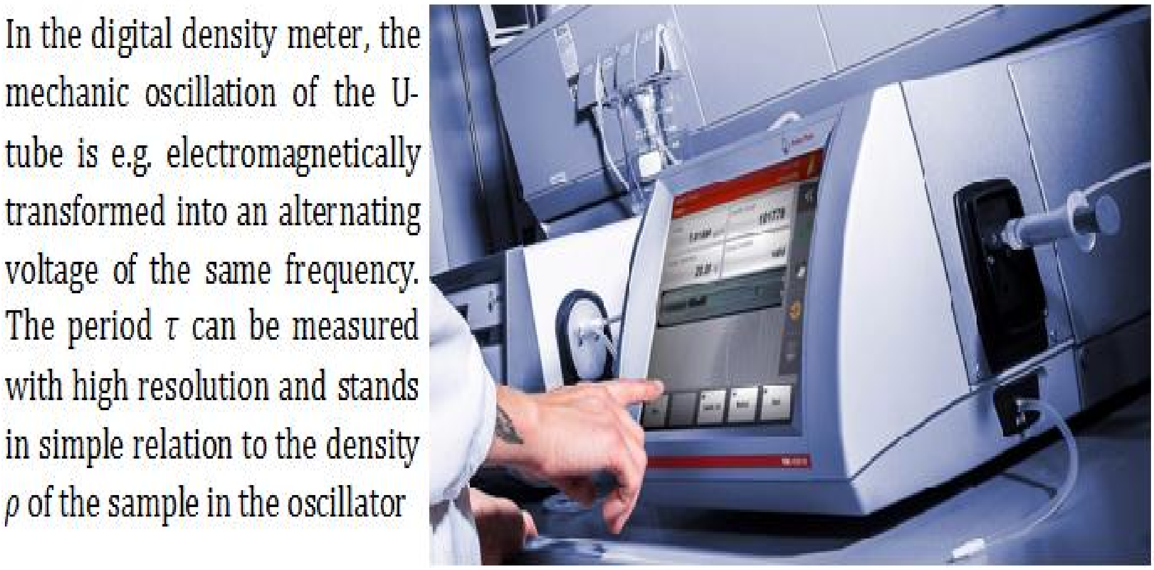
Solutions were prepared by weight precise to ± 0.02 %. The weights were taken on a Mettler electronic analytical balance (AG 285, Switzerland). The molarity being converted to molality as required. Several independent solutions were prepared and runs were performed to ensure the reproducibility of the 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.



Block Diagram of the Instrument

III.2.4.3 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, 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:

$$\rho = A \cdot \tau^2 - B \tag{III.1}$$

A and B are the respective instrument constants of each oscillator. Their values are resolute 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 typically 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 triply-distilled water and dry air.

III.2.4.3 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 instrument was calibrated against the standard viscosity samples supplied through the instrument, water and aqueous $CaCl_2$ solutions [27]. The temperature was maintained to within ± 0.01°C using Brookfield Digital TC-500 thermostat bath. The viscosities were measured with an accuracy of ± 1 %. Each measurement reported herein is an average of triplicate reading with a precision of 0.3 %.

Instrument Specifications:

Range of Speed	*: 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)*
Accuracy of temperature	*: ±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.2.4.5 ULTRASONIC SPEED MEASUREMENT

The ultrasonic speed was measured with an accuracy of 0.2% using single-crystal variable-path ultrasonic interferometer (Model M-81 Mittal Enterprises, New Delhi) operating at 4MHz which was calibrated with water, methanol and benzene at required temperature.

Ultrasonic speeds were measured, with an accuracy of 0.2 %, using a single-crystal variable-path ultrasonic interferometer (Model M-81, Mittal Enterprise, New Delhi) operating at 4 MHz, which was calibrated with water, methanol and benzene at required temperature. The temperature stability was maintained within ± 0.01 K by circulating thermostatic water around the cell by a circulating pump.

Multifrequency Ultrasonic Interferometer

Insrument Specification:

High Frequency Generator	*: Single and Multi-frequency*
Model No.	*: M-81 Mittal Enterprises*
Measuring Cell	*: Four cell (1, 2,3, & 4 MHz)*
Max. displacement of the reflector	*: 20 mm*
Required Quantity of liquid	*: 10 c.c.*
Least Count of micrometer	*: 0.01mm/0.001 mm*

Accuracy	*: 0.2%*
Shielded Cable Impedance	*: 50 O*

Working Principle of Ultrasonic Interferometer

The principle used in the measurement of the ultrasonic speed (u) is based on the accurate determination of the wavelength (λ) in the medium. Ultrasonic waves of well-known frequency (f) are produced by a quartz crystal fixed at the base of the cell. These waves are reflected by a movable metallic plate kept parallel to the quartz crystal. If the separation between these two plates is accurately a whole multiple of the sound wavelength, standing waves are fashioned in the medium. This acoustic resonance provides rise to an electrical reaction on the generator pouring the quartz crystal and the anode current of the generator becomes a maximum.

If the distance is currently increased or decreased and the variation is exactly one half of wave length ($\lambda/2$) or integral multiples of it, anode current becomes maximum. From the knowledge of the wave length (λ), the speed (u) can be obtained by the relation.

$$\text{Ultrasonic speed } (u) = \text{Wave Length } (\lambda) \times \text{Frequency } (f) \qquad \text{(III.5)}$$

Experimental set-up - ultrasonic interferometer consists of the following parts,

a. One high frequency generator.
b. Measuring cell, 1, 2, 3 and 4 MHz.
c. Shielded cable

The measuring cell is connected to the output terminal of the high frequency generator through a shielded cable. The cell is filled through the experimental liquid before switching on the generator. The ultrasonic waves shift normal from the quartz crystal till they are reflected back as of the movable plate and the standing waves are formed in the liquid in between the reflector plate and the quartz crystal. The micrometer is gradually moved till the anode current on the meter on the elevated frequency generator shows a maximum. A number of maxima readings of anode current are passed and their number (n) is counted. The total distance (d)

thus moved by the micrometer gives the value of the wavelength (λ) with the following relation.

$$d = n \times \lambda/2 \tag{III.6}$$

Further, the velocity is determined from which the isentropic compressibility (β_S) is calculated by the following formula:

$$\beta_S = 1 / (u^2 . \rho) \tag{III.7}$$

where ρ is the density of the experimental liquid.

The following Figure shows the Multifrequency Ultrasonic Interferometer i.e.

(a) Cross-section of the measuring cell,

(b) Position of reflector vs. crystal current (Note: The extra peaks in between minima and maxima occurs due to a number of reasons, but these do not affect the value of $\lambda/2$) and)

(c) Electronic circuit diagram of the instrument

The Multifrequency Ultrasonic Interferometer

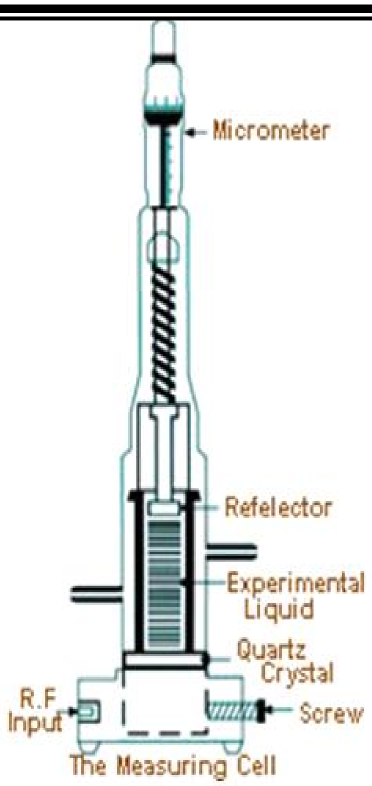


Figure 1(a): Cross-Section of the Measuring Cell

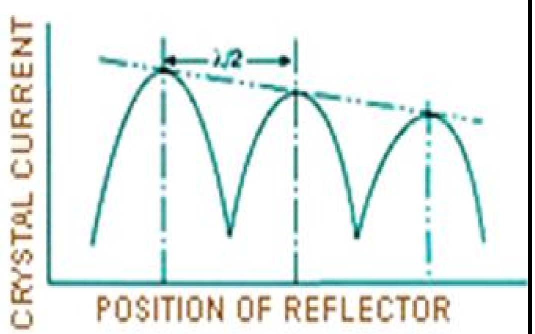
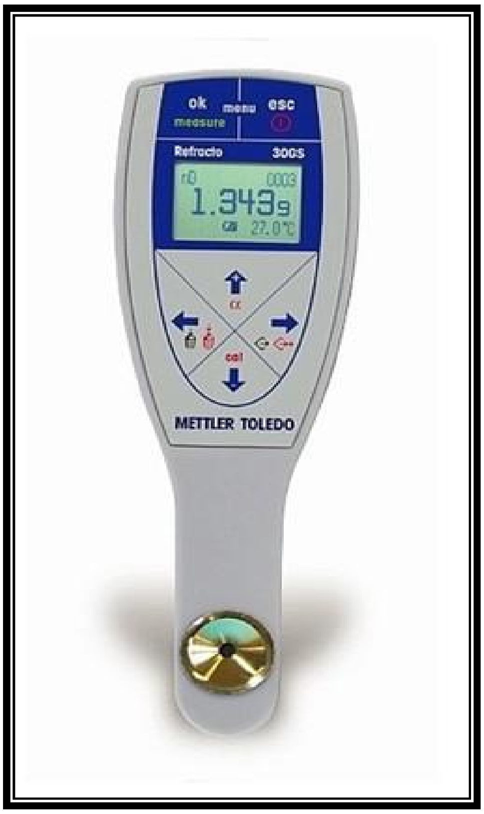


Figure 1(b): Position of Reflector versus Crystal Current

III.2.4.6 REFRACTIVE INDEX MEASUREMENT



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

Calibration was executed 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.

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	*: 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 (also known as refractive index or n_D) for the substance.

$$Refractive\,index\,of\,the\,substance\,(n_D) = \frac{Speed\,of\,light\,in\,a\,vacuum}{Speed\,of\,light\,in\,substance} \qquad (III.8)$$

Whenever light changes speed as it crosses a boundary from one medium into another its direction of travel also changes, i.e., it is refracted (Figure 1). (In the special case of the light traveling perpendicular to the boundary there is no change in direction upon entering the new medium.) The relationship between light's speed in the two mediums (v_A and v_B), the angles of incidence (q_A) and refraction (q_B) and the refractive indexes of the two mediums (n_A and n_B) is shown below:

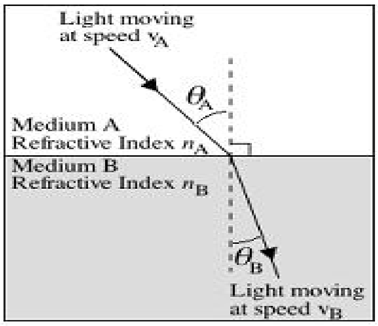


Figure 1. Light crossing from any transparent medium into another in which it has a different speed, is refracted, i.e., bent from its original path (except when the direction of travel is perpendicular to the boundary). In the case shown, the speed of light in medium A is greater than the speed of light in medium B

$$\frac{V_A}{V_B} = \frac{\sin\theta_A}{\sin\theta_B} = \frac{n_B}{n_A} \qquad \text{(III.9)}$$

Thus, it is not necessary to measure the speed of light in a sample in order to determine its index of refraction. Instead, by means of measuring the angle of refraction, and knowing the index of refraction of the layer to be precise in contact with the sample, it is probable to determine the refractive index of the sample quite accurately [28]. Nearly all refractometers utilize this principle, but may differ in their optical design.

A light source is projected through the illuminating prism, the bottom surface of which be ground (i.e., roughened like a ground-glass joint), so every point on this surface can be thought of as generating light rays traveling in all directions. Assessment of Figure 1 shows that light traveling from point A to point B will have the biggest angle of incidence (q_i) and therefore the largest possible angle of refraction (q_r) for that sample. All other rays of light inflowing the refracting prism will have smaller q_r and hence lie to the left of point C. As a consequence, a detector located on the back side of the refracting prism would show a light region to the left and a dark region to the right.

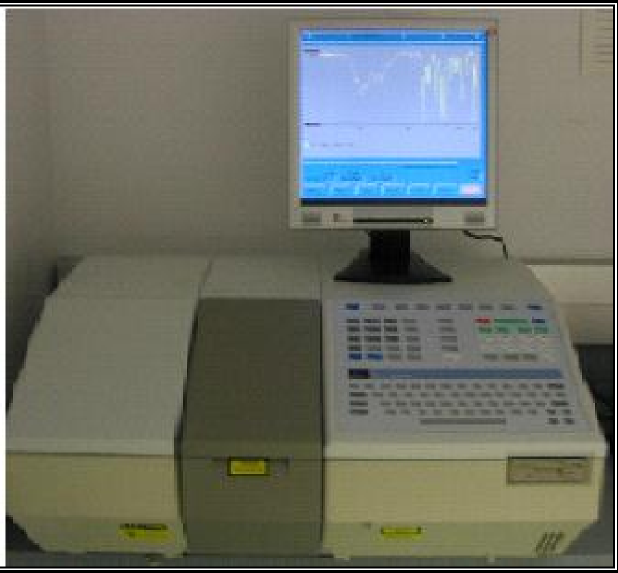
III.2.4.7 SURFACE TENSION MEASUREMENT

Surface tension was measured by using Digital Tensiometer KRŰSS 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.2.4.8 FTIR MEASUREMENT

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

It measures the intensity of light passing through the blank and measures the intensity of light passing through the sample. It is useful to calculate the transmittance and the absorbance



The intensity of light (I_0) passing through a blank is considered. The intensity is the number of photons per second. The blank is a solution that is identical to the sample solution except that the blank does not contain the solution that absorbs light. The intensity of light (I) passing through the sample solution is calculated. (In practice, instrument measures the power somewhat than the intensity of the light. The power is the energy per second, which be the product of the intensity (photons per second) and the energy per photon. The experimental data is used to calculate two quantities: the transmittance (T) and the absorbance (A).

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

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

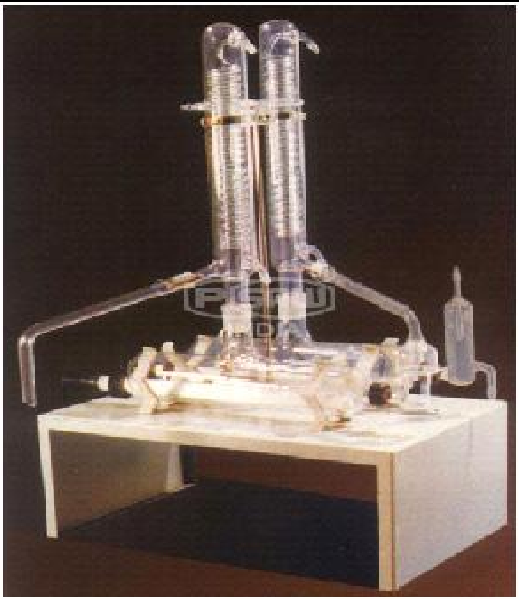
III.2.4.7 NMR MEASUREMENT

Nuclear Magnetic Spectra (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 ^1H NMR spectra were recorded at 400MHz and 500 MHz using Bruker Advance instrument. Signals are quoted as δ values in ppmusing residual protonated solvent signals as internal standard (D_2O: δ 4.79 ppm). Data are reported as chemical shift.

OTHER INSTRUMENTS USED:

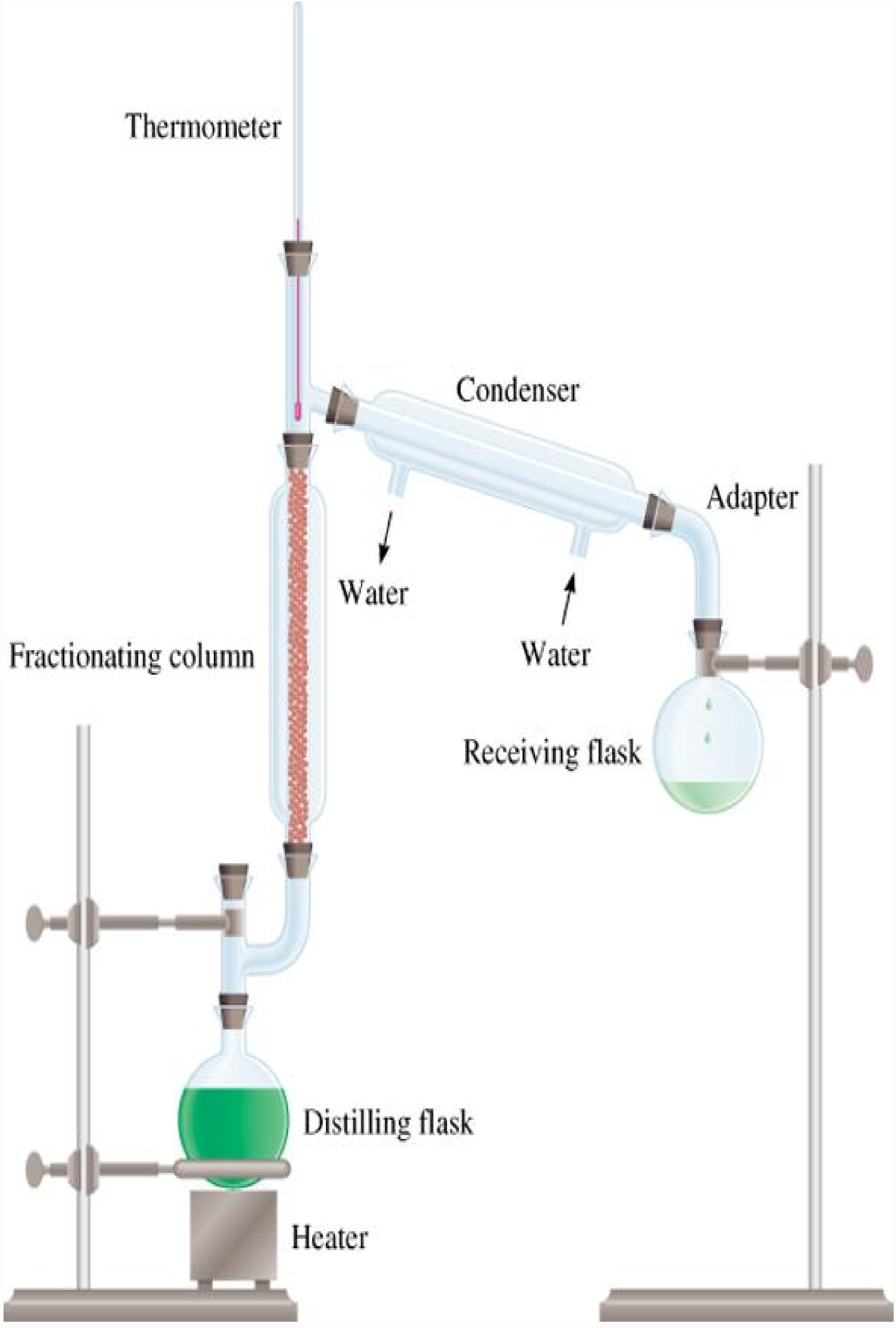
III.2.4.8 WATER DISTILLER

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 physically or automatically fed into the distiller unit's boiling chamber. A heating element inside the boiling chamber heats the water awaiting it boils. The steam increases from the boiling chamber. Volatile contaminants (such as, gases) are discharged through a built-in vent. Minerals and salts are retained in the boiling chamber as hard deposits or scale. The steam penetrates a coiled tube (condenser), which is cooled by cold 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 activate to fill the storage tank. The distillation apparatus consists of flask with heating elements embedded in glass and fused in spiral category coil internally of the base and tapered round glass, joints at the apex double walled condenser with B-40/B-50 ground glass joints, suitable to work on 220 volts, 50 cycles AC supply.

Fractional Distillation Apparatus



III.2.4.9 THERMOSTAT WATER BATH (Science India, Kolkata):

The measurements were carried out in thermostatic water bath maintained with an accuracy of ± 0.01 K of the desired temperature.

Laboratory water bath is a system in which a vessel containing the substance to be heated is placed into or over the one containing water and to rapidly heat it. These laboratory apparatus 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.