CHAPTER III

EXPERIMENTAL SECTION

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

3.1.1. SOLVENTS

1,3-Dioxolane

Dioxolane or 1,3-dioxolane is a heterocyclic acetal. No unusual toxic effects have been associated with the use 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.

Appearance: Liquid
M.F. C₆H₄O₂
M.W. 74.08 g/mol
M.P. 178 K
B.P. 348 K
D.C. 7.34 at 298.15 K

Source: Sd. Fine Chemicals, India.

Purification: It is dried with KOH and then distilled from sodium.[1]

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

Nitromethane is one of the simplest organic nitro compounds. It is a slightly viscous, highly polar liquid.

- **Appearance:** Liquid
- **M.F.:** CH₃NO₂
- **M.W.:** 61.04 g/mol
- **M.P.:** 244.15 K
- **B.P.:** 373-376 K
- **D.C.:** 39.4 at 293.15 K

**Source:** S.D. Fine Chemicals Ltd., Mumbai, India.

**Purification:** It is dried with CaSO₄ and then distilled [1].

**Application:** The principle use of nitromethane is as a stabilizer for chlorinated solvents, which are used in dry cleaning, semiconductor processing, and degreasing. It is also used most effectively as a solvent or dissolving agent for acrylate monomers, such as cyanoacrylates. In more specialized organic synthesis, nitromethane serves as a Michael donor, adding to α,β-unsaturated carbonyl compounds via 1,4-addition in the Michael reaction. Its acidity allows it to undergo deprotonation, enabling condensation reactions analogous to those of carbonyl compounds.

**Nitrobenzene**

Nitrobenzene is a colorless to pale yellow oily liquid with an odour resembling that of bitter almonds or "shoe polish." It represents a fire hazard, with a flash point (closed cup method) of 88 °C and an explosive limit (lower) of 1.8% by volume in air. Nitrobenzene can undergo degradation by both photolysis and microbial biodegradation.

- **Appearance:** Yellowish liquid
- **M.F.:** C₆H₅NO₂
- **M.W.:** 123.06 g/mol
- **M.P.:** 278.15 K
- **B.P.:** 484.05 K
- **D.C.:** 34.69
Experimental Section

Source: Sd. Fine Chemicals, India

Purification: It is distilled in presence of dil. H\textsubscript{2}SO\textsubscript{4} then dried with CaCl\textsubscript{2} followed by distillation from P\textsubscript{2}O\textsubscript{5} [1].

Application: Nitrobenzene, one of the major uses for nitrobenzene is for the production of aniline, [2] which is a chemical intermediate used during the manufacture of polyurethane. Nitrobenzene is also used industrially in the manufacture of some pharmaceuticals, dyes and rubbers, as a constituent in some polishes and paint solvents and as a solvent in the refining of petroleum. More specialized applications include the use of nitrobenzene as a precursor to rubber chemicals, pesticides, dyes, explosives, and pharmaceuticals. Nitrobenzene is also used in shoe and floor polishes, leather dressings, paint solvents, and other materials to mask unpleasant odors. Redistilled, as oil of mirbane, nitrobenzene has been used as an inexpensive perfume for soaps. A significant merchant market for nitrobenzene is its use in the production of the analgesic paracetamol [3].

Acetonitrile

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

![Image of Acetonitrile molecule]

**Appearance**: Liquid

**M.F.**: CH\textsubscript{3}CN

**M.W.**: 41.05 g/mol

**M.P.**: 607.5 K

**B.P.**: 628.3 K

**D.C.**: 37.5 at 294.26 K

Source: Thomas Baker, India.

Purification: It is shaken with silica gel, refluxed with CaH\textsubscript{2} and distilled over P\textsubscript{2}O\textsubscript{5} [1].

Application: It is widely used in battery applications because of its relatively high dielectric constant and ability 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 significant role as the dominant solvent used in the manufacture of DNA oligonucleotides from monomers. Industrially, it is used as a solvent in the purification of butadiene and in the manufacture of pharmaceuticals and photographic film. Acetonitrile is a common two-carbon building block in organic synthesis as in the production of pesticides to perfumes.

**n-Hexanol**

*n-Hexanol* is an organic alcohol with a six-carbon chain and a condensed structural formula of \( \text{CH}_3(\text{CH}_2)_5\text{OH} \). This colorless liquid is slightly soluble in water, but miscible with ether and ethanol. 1-Hexanol is believed to be a component of the odour of freshly mown grass. Alarm pheromones emitted by the Koschevnikov gland of honey bees contain 1-hexanol.

**Source:** Merck, India

**Purification:** It was dried by adding drying agent CaSO₄ followed by filtration and then distillation [1].

**Application:** n-Hexanol is used for Flavors & Essences. It is used as a solvent in the pharmaceutical and perfume industry. It is used in the production of plasticizer, surfactant.

**o-Toluidine**

It is an ortho isomer of toluidine. The chemical properties of the toluidines are quite similar to those of aniline and toluidines have properties in common with other aromatic amines. Due to the amino group bonded to the aromatic ring, the toluidines are weakly basic.


**Source:** Merck, India.

**Purification:** It was distilled from Zn dust at reduced pressure [1].

**Application:** Toluidines are used in the production of dyes. They are a component of accelerators for cyanoacrylate glues. They are toxic and are suspected human carcinogens. It is used as a presumptive test for blood in forensic science, is two o-toluidine molecules linked together.

**Aniline**

Aniline, phenylamine or aminobenzene is an organic compound with the formula C₆H₅NH₂. Consisting of a phenyl group attached to an amino group, aniline is the prototypical aromatic amine. Being a precursor to many industrial chemicals, its main use is in the manufacture of precursors to polyurethane. Like most volatile amines, it possesses the somewhat unpleasant odour of rotten fish. It ignites readily, burning with a smoky flame characteristic of aromatic compounds. Aniline is colorless, but it slowly oxidizes and resinifies in air, giving a red-brown tint to aged samples.

**Source:** Sd. Fine Chemicals

**Purification:** It was dried with KOH and then treated with stannous chloride and then distilled [1].

**Application:** The largest application of aniline is for the preparation of methylene diphenyl diisocyanate (MDI). The majority of aniline serves this market. Other uses include rubber processing chemicals (9%), herbicides (2%), and dyes and pigments (2%). As additives to rubber, aniline derivatives such as phenylenediamines and diphenylamine, are antioxidants. Illustrative of the drugs prepared from aniline is paracetamol (acetaminophen, Tylenol). The principal use of aniline in the dye industry is as a precursor to indigo, the blue
of blue jeans [4]. Aniline is also used at a smaller scale in the production of the intrinsically conducting polymer polyaniline.

**N, N, Dimethylformamide**

N, N, Dimethylformamide is an organic compound with the formula \((\text{CH}_3)_2\text{NC(O)H}\). Commonly abbreviated as DMF (though this acronym is sometimes used for dimethylfuran), this colourless liquid is miscible with water and the majority of organic liquids. DMF is a common solvent for chemical reactions. Pure dimethylformamide is odorless whereas 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, the amide of formic acid. DMF is a polar (hydrophilic) aprotic solvent with a high boiling point. It facilitates reactions that follow polar mechanisms, such as \(S_N^2\) reactions.

**Source:** Thomas Baker, India

**Purification:** It was dried by passing through Linde 4Å molecular sieves and then distilled [1].

**Application:** The primary use of dimethylformamide is as a solvent with low 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, and in the manufacture of adhesives, synthetic leathers, fibers, films, and surface coatings [5]. It is used as a reagent in the Bouveault aldehyde synthesis and in the Vilsmeier-Haack reaction, another useful 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 [6]. 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 component 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 utilized 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 used in the manufacturing of solvent dyes as an important raw material. It is consumed during reaction. Pure acetylene gas cannot be compressed and stored without 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 renders it safe to transport and use.

**N, N, Dimethylacetamide**

N,N-Dimethylacetamide is the organic compound with the formula CH₃C(O)N(CH₃)₂. This colorless, water-miscible, high boiling liquid is commonly used as a polar solvent in organic synthesis. DMA, as it often abbreviated, is miscible with most other solvents, although it is poorly soluble in aliphatic hydrocarbons.

Source: Thomas Baker, India

**Purification:** It was dried by passing through molecular sieves [1].

**Application:** DMA is useful solvent for reactions involving strong bases such as sodium hydroxide. Dimethylacetamide is commonly used as a solvent for fibers (e.g., polyacrylonitrile, spandex) or in the adhesive industry [7]. It is also
employed in the production of pharmaceuticals and plasticizers as a reaction medium.

**Dimethyl sulfoxide**

Dimethyl sulfoxide (DMSO) is an organosulfur compound with the formula \((\text{CH}_3\text{)}_2\text{SO}\). This colorless liquid is an important polar aprotic solvent that dissolves both polar and non-polar compounds and is miscible in a wide range of organic solvents as well as water. It penetrates the skin very readily, giving it the unusual property for many individuals of being secreted onto the surface of the tongue after contact with the skin and causing a garlic-like taste in the mouth.

Although it has some niche medicinal uses it also has significant known side effects. It has been promoted as a fake cure for cancer and other conditions.

**Source:** Thomas Baker, India

**Purification:** It was dried by passing through Linde 4Å molecular sieves.

**Application:** DMSO is frequently used as a solvent for chemical reactions involving salts, most notably Finkelstein reactions and other nucleophilic substitutions. It is also extensively used as an extractant in biochemistry and cell biology. Because of its ability to dissolve many kinds of compounds, DMSO plays a role in sample management and high-throughput screening operations in drug design. DMSO is used in PCR to inhibit secondary structures in the DNA template or the DNA primers. It is added to the PCR mix before reacting, where it interferes with the self-complementarity of the DNA, minimizing interfering reactions. In medicine, DMSO is predominantly used as a topical analgesic, a vehicle for topical application of pharmaceuticals, as an anti-inflammatory, and an antioxidant [8]. Because DMSO increases the rate of absorption of some
compounds through organic tissues, including skin, it can be used as a drug delivery system. It is frequently compounded with antifungal medications, enabling them to penetrate not just skin but also toe and fingernails. It is also used as veterinary medicines.

**Ethylene Glycol**

Ethylene glycol is a colourless, practically odourless, low-volatility, low-viscosity, hygroscopic liquid. It is completely miscible with water and many organic liquids. The hydroxyl groups on glycols undergo the usual alcohol chemistry, giving a wide variety of possible derivatives.

**Source:** Merck, India.

**Purification:** It was dried with anhydrous CaSO₄ and distilled under vacuum. The distillate was passed through Linde type 4 Å molecular sieves [1].

**Application:** The major uses of ethylene glycol are as an antifreeze, which accounts for over 50% of ethylene glycol’s commercial uses, and as raw material in the production of polyester fibers, mainly PET, which accounts for 40% of total ethylene glycol consumption. Because this material is cheaply available, it finds many niche applications [9]. It is widely used as an intermediate in the synthesis of many varieties of organic chemical compounds. Industrially it is employed in the manufacture of synthetic drugs and dyes.

**Methanol**

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

**Purification:** It was passed through Linde Å molecular sieves and then distilled.

**Application:** The largest use of methyl alcohol by far is in making other chemicals. About 40% of methanol is converted to formaldehyde, and from there into products as diverse as plastics, plywood, paints, explosives, and permanent press textiles. Methanol is a traditional denaturant for ethanol, thus giving the term methylated spirit. Methanol is also used as a solvent, and as an antifreeze in pipelines. In some waste water treatment plants, a small amount of methanol is added to waste water to provide a food source of carbon for the denitrifying bacteria, which converts nitrates to nitrogen to reduce the denitrification of sensitive aquifers. Methanol is used on a limited basis to fuel internal combustion engines. Methanol is also useful as an energy carrier. It is easier to store than hydrogen, burns cleaner than fossil fuels, and is biodegradable.

### 3.1.2. SOLUTES

**Sodium Tetraphenyl Borate**

Sodium tetraphenylborate is the organic compound with the formula NaB(C₆H₅)₄. It is a salt, wherein the anion consists of four phenyl rings bonded to boron.

**Appearance:** White solid

**M.F.:** (C₆H₅)₄BNa

**M.W.:** 342.216 g/mol

**M.P.:** >573.15K
**Source:** Sigma Aldrich, Germany  
**Purification:** Used as purchased.  
**Application:** This white crystalline solid is used to prepare other tetraphenylborate salts, which are often highly soluble in organic solvents. The compound is used in inorganic and organometallic chemistry as a precipitating agent.

**Tetrabutylammonium tetraphenylborate**

![Tetrabutylammonium tetraphenylborate](image)

**Source:** Sigma Aldrich, Germany  
**Purification:** Used as purchased.  
**Application:** It is widely used as supporting electrolytes in electrochemical measurements when control of electrode potentials is required.

**Tetrabutylammonium Hexafluorophosphate**

![Tetrabutylammonium Hexafluorophosphate](image)

**Source:** Sigma Aldrich, Germany  
**Purification:** Used as purchased.
**Application**: It is used as reference electrolyte because both ions have about the same volume and half the limiting conductance is assumed to give the single ion conductances of $\text{Bu}_4\text{N}^+$ and $\text{F}_6\text{P}^-$. 

**Lithium Hexafluoroarsenat**

Source: Sigma Aldrich, Germany

Purification: Used as purchased.

Application: Used as electrolytes in lithium-ion batteries

**1-Ethyl 3-Methylimidazolium Bromide**

1-Ethyl 3-Methylimidazolium Bromide is an ionic liquid. The cation consists of a five-membered ring with two nitrogen and three carbon atoms, i.e. a derivative of imidazole, with ethyl and methyl groups substituted at the two nitrogen atoms.

Source: Sigma Aldrich, Germany

Purification: Used as purchased.

Application: 1-Ethyl-3-methylimidazolium salts are used in cellulose processing. They are also used as use as non-aqueous electrolytes in electrochemical applications.
Quaternary ammonium iodides

Quaternary ammonium compounds (R₄N⁺) are a group of ammonium salts in which organic radicals have been substituted for all four hydrogens of the original ammonium cation. They have a central nitrogen atom which is joined to four organic radicals (for e.g. butyl group) and one acid radical (for e.g. iodide ion). They are prepared by treatment of an amine with an alkylating agent. They show a variety of physical, chemical, and biological properties and most compounds are soluble in water and strong electrolytes. In addition to their tendency of locating at the interface of two phases (liquid–liquid or solid–liquid) to introduce continuity between the two different phase, they have properties of disrupting micro-organisms' cell processes.

Source: Sigma Aldrich, Germany

Purification: dissolving in mixed alcohol medium and recrystallised from solvent ether medium [1].

Application: Quaternary ammonium salts are used as disinfectants, surfactants, fabric softeners, and as antistatic agents (e.g. in shampoos). In liquid fabric softeners, the chloride salts are often used. Indryer anticling strips, the sulfate salts are often used. Spermicidal jellies also contain quaternary ammonium salts. Quaternary ammonium compounds have
also been shown to have antimicrobial activity [10]. Certain quaternary ammonium compounds, especially those containing long alkyl chains, are used as antimicrobials and disinfectants. Quaternary ammonium salts are popular chemicals used in foodservice industry as sanitizing agents. In organic synthesis, quaternary ammonium salts are employed as phase transfer catalysts (PTC).

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 earlier measured by Ostwald- Sprengel type Pycometer having a bulb volume of 25cm$^3$ and an internal diameter of the capillary of about 1mm. The pycometer was calibrated at 298.15K with doubly distilled water and THF. The total uncertainty in the density value was ±0.0001gcm$^{-3}$.

The density was measured later 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 $\tau$ can be measured with high resolution and stands in simple relation to the density $\rho$ of the sample in the oscillator[11]:

$$\rho = A \cdot \tau^2 - B$$  \hspace{1cm} (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 $\rho_1$ and $\rho_2$. 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

Solvent viscosities were measured by means of a suspended Ubbelohde-type viscometer,

The kinematic viscosity ($\gamma$) and the absolute viscosity ($\eta$) are given by the following equations.

$$\gamma = k \cdot t - 1/t$$  \hspace{1cm} (2)

$$\eta = \gamma \cdot \rho$$  \hspace{1cm} (3)
where, \( t \) is the time of flow, \( \rho \) is the density and \( k \) and \( l \) are the characteristic constants of the particular viscometer. The precision of the viscosity measurement was \( \pm 0.003 \% \). In all cases, the experiments were performed in at least three replicates and the results were averaged.

Relative viscosities (\( \eta_r \)) were obtained using the equation:

\[
\eta_r = \frac{\eta}{\eta_0} = \frac{\rho t}{\rho_0 t_0}
\]

(4)

where \( \eta, \eta_0, \rho, \rho_0 \) and \( t, t_0 \) are the absolute viscosities, densities and flow times for the solution and solvent respectively.

The viscosity was also measured with the help Brookfield DV-III Ultra Programmable Rheometer fitted to a Brookfield Digital Bath TC-500.

3.2.6. TEMPERATURE CONTROLLER

All the measurements were carried out in thermostatic water bath (Science India, Kolkata) maintained with an accuracy of \( \pm 0.01 \) 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. 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.

The principle used in the measurement of the ultrasonic speed \( \nu \) is based on the accurate determination of the wavelength \( \lambda \) in the medium. Ultrasonic waves of known frequency \( f \) are produced by a quartz crystal fixed at the bottom 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 exactly a whole multiple of the sound wavelength, standing waves are formed in the medium. This acoustic resonance gives rise to an electrical reaction on the generator driving the quartz crystal and the anode current of the generator becomes a maximum.

If the distance is now 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 ($\lambda$), the speed ($u$) can be obtained by the relation.

\[
\text{Ultrasonic speed } (u) = \text{Wave Length } (\lambda) \times \text{Frequency } (f) \quad (5)
\]

The ultrasonic interferometer consists of the following two parts, (i) the high frequency generator, and (ii) the measuring cell. The measuring cell is connected to the output terminal of the high frequency generator through a shielded cable. The cell is filled with the experimental liquid before switching on the generator. The ultrasonic waves move normal from the quartz crystal till they are reflected back from the movable plate and the standing waves are formed in the liquid in between the reflector plate and the quartz crystal. The micrometer is slowly moved till the anode current on the meter on the high 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 ($\lambda$) with the following relation.

\[
d = n \times \lambda / 2 \quad (6)
\]

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

\[
K_S = 1 / (u^2. \rho) \quad (7)
\]

where $\rho$ is the density of the experimental liquid.

Figure. 1 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 effect the value of $\lambda/2$) and (c) Electronic circuit diagram of the instrument)
Figure 1: The Multifrequency Ultrasonic Interferometer

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

Figure 1(b): Position of Reflector versus Crystal Current
Figure 1(c): Electronic Circuit Diagram of the Instrument
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 $1.11 \text{cm}^{-1}$. 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 $\text{cm}^3$ 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.
3.2.9. REFRACTIVE INDEX MEASUREMENT

Refractive index was measured 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. FT-IR MEASUREMENT

Infrared spectra were recorded in 8300 FT-IR spectrometer (Shimadzu, Japan).

The intensity of light ($I_0$) passing through a blank is measured. The intensity is the number of photons per second. The blank 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 measured. (In practice, instrument measures the power rather than the intensity of the light. The power is the energy per second, which is 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 ($A$).

$$T = \frac{I}{I_0} \quad \text{and} \quad A = -\log_{10} T$$

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


