CHAPTER -III
Experimental Section

3.1. Name, Structure, Physical properties, purification and application of the used solvents and solutes.

3.1.1. Solvents

Water:

Water is a ubiquitous chemical substance that 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 has a solid state, ice, and a gaseous state, water vapor or steam. Water is a good solvent and is often referred to as the universal solvent.

Source: Distilled water.

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 $1 \times 10^{-6}$ S.cm$^{-1}$. $^1$

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. In organic reactions, it is not usually used as a reaction solvent, because it does not dissolve the reactants well and is amphoteric (acidic and basic) and nucleophilic. Nevertheless, these properties are

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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<tbody>
<tr>
<td>Appearance:</td>
<td>Liquid</td>
</tr>
<tr>
<td>Molecular Formula:</td>
<td>H$_2$O</td>
</tr>
<tr>
<td>Molecular Weight:</td>
<td>18.02g/mol</td>
</tr>
<tr>
<td>Boiling Point:</td>
<td>100 °C</td>
</tr>
<tr>
<td>Melting Point:</td>
<td>0 °C</td>
</tr>
<tr>
<td>Dielectric Constant:</td>
<td>78.35 at 25°C</td>
</tr>
</tbody>
</table>
sometimes desirable. Also, acceleration of Diels-Alder reactions by water has been observed. Supercritical water has recently been a topic of research. Oxygen-saturated supercritical water combusts organic pollutants efficiently. It is also use in various industries.

2-Methoxyethanol:

2-Methoxyethanol, or methyl cellosolve, is an organic compound that is used mainly as a solvent. It is a clear, colourless liquid with an ether-like odour. It is in a class of solvents known as glycol ethers which are notable for their ability to dissolve a variety of different types of chemical compounds and for their miscibility with water and other solvents.

Source: Sd. fine chemicals Ltd., Mumbai, India.

Purification: Peroxides was removed by refluxing with stannous chloride. It was dried with silica gel crystals, with a final distillation with sodium².

Application: 2-Methoxyethanol is used as a solvent for many different purposes such as varnishes, dyes, and resins. It is also used as an additive in airplane deicing solutions.
Carbon Tetrachloride:

Carbon tetrachloride, also known by many other names (notably, carbon tet in the cleaning industry, and as a Halon or Freon in HVAC) is the organic compound with the formula CCl₄. In the carbon tetrachloride molecule, four chlorine atoms are positioned symmetrically as corners in a tetrahedral configuration joined to a central carbon atom by single covalent bonds. Because of this symmetrical geometry, CCl₄ is non-polar. Source: Merck, India.

Purification: It was dried with CaCl₂ and distilled over P₂O₅.

Application: As it has no C-H bonds, carbon tetrachloride does not easily undergo free-radical reactions. Hence it is a useful solvent for halogenations. In organic chemistry, carbon tetrachloride serves as a source of chlorine. It is used as a solvent in synthetic chemistry research. It is sometimes useful as a solvent for infrared spectroscopy. Because carbon tetrachloride does not have any hydrogen atoms, it was historically used in proton NMR spectroscopy. However, carbon tetrachloride is toxic, and its dissolving power is low. Its use has been largely superseded by deuterated solvents, which offer superior solvating properties and allow for deuterium lock by the spectrometer.

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<thead>
<tr>
<th>Property</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Liquid</td>
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<tr>
<td>Molecular Formula</td>
<td>CCl₄</td>
</tr>
<tr>
<td>Molecular Weight</td>
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<tr>
<td>Boiling Point</td>
<td>77°C</td>
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<tr>
<td>Melting Point</td>
<td>-23°C</td>
</tr>
<tr>
<td>Dielectric Constant</td>
<td>2.2 at 20°C</td>
</tr>
</tbody>
</table>
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.

**Source:** Merck, India

**Purification:** It can be purified by fraction distillation. Impure nitrobenzene is taken in a round bottomed flask fitted with a condenser and heated in a water bath maintained at 207-210°C. Nitrobenzene is obtained as yellow vapours which is cooled by condenser and collected back.

**Application:** As it is very stable in Friedel-Crafts reaction and in presence of oxidizing agent, it is used in the manufacture of aniline, benzidine etc. It is also used to provide scent in cheap quality soap and shoe-polish. It is used in the manufacture of polyurethanes; nitrobenzene is also used as a solvent in petroleum refining, as a solvent in the manufacture of cellulose ethers and acetates, in the manufacture of dinitrobenzene and dichloroaniline, and in the synthesis of other organic compounds, including acetaminophen.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Liquid</td>
</tr>
<tr>
<td>Molecular Formula</td>
<td>C₆H₅NO₂</td>
</tr>
<tr>
<td>Molecular Weight</td>
<td>123.06 g/mol</td>
</tr>
<tr>
<td>Boiling Point</td>
<td>210.9°C</td>
</tr>
<tr>
<td>Dielectric Constant</td>
<td>34.8 at 25°C</td>
</tr>
</tbody>
</table>
3.1.2. Solutes

**Catechol:**

Catechol, formerly known as pyrocatechol, is 1, 2-dihydroxybenzene, an organic compound with the feathery white crystals which are very rapidly soluble in water. It is the ortho isomer of one of the three isomeric benzenediols.

**Source:** Sd. fine chemicals Ltd., Mumbai, India.

**Purification:** Commercial sample of catechol was purified by repeated crystallization from mixture of chloroform-methanol. The sample was dissolved in chloroform in hot condition, filtered and to the filtrate dried & distilled methanol was added dropwise. Fine plat like crystal separated and recovered by rapid filtration & ready for use.

**Application:** It is used mainly as a precursor to pesticides, flavors and fragrances. Catechol is consumed in the production of pesticides, the remainder being used as a precursor to fine chemicals such as perfumes and pharmaceuticals. It is a common building block in organic synthesis. Several industrially significant flavors and fragrances are prepared starting from catechol.
Glycine:

Glycine (abbreviated as Gly or G) is the organic compound with only a hydrogen atom as its side chain, glycine is the smallest of the 20 amino acids commonly found in proteins. Glycine is unique among the proteinogenic amino acids in that it is not chiral.

**Source:** Analar, BDH.

**Purification:** It was purified by re-crystallizing from methanol-water mixture and dried at 100°C for 12 h in a vacuum desiccator over P₂O₅ before use.

**Application:** Pharmaceutical grade glycine is produced for use in some pharmaceutical applications, such as intravenous injections, Technical grade glycine is used in industrial applications; e.g., as an agent in metal complexing and finishing. Glycine serves as a buffering agent in antacids, analgesics, antiperspirants, cosmetics, and toiletries. Glycine is an intermediate in the synthesis of a variety of chemical products.

D-Glucose:

Glucose (C₆H₁₂O₆), a simple sugar (monosaccharide), is an important carbohydrate in biology. Cells use it as a source of energy and a metabolic intermediate. Glucose is one of the main products of photosynthesis and starts cellular respiration. Starch and cellulose are polymers derived from the dehydration of glucose.
**Source**: Sd. fine chemicals, India

**Purification**: Recrystallised slowly from aqueous 80 % ethanol and then vacuum dried over P_2O_5.

**Application**: Glucose circulates in the blood, providing energy to organs, glands, muscles, indeed to every cell. Glucose is used in oxidation. More complex sugars have to be changed to glucose first before they can be broken down to release energy in respiration. Glucose is a ubiquitous fuel in biology. It is used as an energy source in most organisms, from bacteria to humans. Use of glucose may be by either aerobic respiration, anaerobic respiration, or fermentation. Glucose is a primary source of energy for the brain, and hence its availability influences psychological processes. When glucose is low, psychological processes requiring mental effort (e.g., self-control, effortful decision-making) are impaired.

**D-Mannitol**:

Mannitol is a sugar alcohol; that is, it is derived from a sugar by reduction. Other sugar alcohols include xylitol and sorbitol. Aqueous solutions of mannitol are mildly acidic and sometimes such solutions are treated to lower the pH.

**Source**: Sd. fine chemicals, India
Purification: Crystallised from ethanol and dried at 100°C.

Application: Mannitol is used clinically to reduce acutely raised intracranial pressure until more definitive treatment can be applied, e.g., after head trauma. It is also used to treat patients with oliguric renal failure. It is administered intravenously, and is filtered by the glomeruli of the kidney, but is incapable of being resorbed from the renal tubule, resulting in decreased water and Na\(^+\) reabsorption via its osmotic effect. Consequently, mannitol increases water and Na\(^+\) excretion, thereby decreasing extracellular fluid volume.

Mannitol can also be used to open the blood-brain barrier by temporarily shrinking the tightly coupled endothelial cells that make up the barrier. This makes mannitol indispensable for delivering various drugs directly to the brain (e.g., in the treatment of Alzheimer's disease). Mannitol is commonly used in the circuit prime of a heart lung machine during cardiopulmonary bypass. The presence of mannitol preserves renal function during the times of low blood flow and pressure, while the patient is on bypass. The solution prevents the swelling of endothelial cells in the kidney, which may have otherwise reduced blood flow to this area and resulted in cell damage.

**D-Sucrose:**

Sucrose has hydrogen to oxygen ratio of 2:1. It consists of two monosaccharide, glucose and fructose, joined by a glycosidic bond between carbon atom 1 of the glucose unit and carbon atom 2 of the fructose unit. What is notable about sucrose is that unlike most polysaccharides, the glycosidic bond

<table>
<thead>
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<th>Appearance:</th>
<th>White Crystalline Solid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular Formula:</td>
<td>C(<em>{11})H(</em>{22})O(_{11})</td>
</tr>
<tr>
<td>Molecular Weight:</td>
<td>342.3 g/mol</td>
</tr>
<tr>
<td>Boiling Point:</td>
<td>(160-186)(^\circ)C</td>
</tr>
<tr>
<td>Melting Point:</td>
<td>186 (^\circ)C</td>
</tr>
</tbody>
</table>
is formed between the reducing ends of both glucose and fructose, and not between
the reducing end of one and the nonreducing end of the other. The effect of this
inhibits further bonding to other saccharide units. Since it contains no free anomeric
carbon atom, it is classified as a nonreducing sugar. Sucrose melts and decomposes
at 186 °C to form caramel, and when combusted produces carbon, carbon dioxide,
and water. Water breaks down sucrose by hydrolysis; however the process is so
gradual that it could sit in solution for years with negligible change. If the enzyme
sucrose is added however, the reaction will proceed rapidly.

**Source:** Sd. fine chemicals, India

**Purification:** Crystallised from water.

**Application:** Sucrose is an easily assimilated macronutrient that provides a quick
source of energy to the body, provoking a rapid rise in blood glucose upon ingestion.
However, pure sucrose is not normally part of a human diet balanced for good
nutrition, although it may be included sparingly to make certain foods more
palatable. Over-consumption of sucrose has been linked with some adverse health
effects. The most common is dental caries or tooth decay, in which oral bacteria
convert sugars (including sucrose) from food into acids that attack tooth enamel.
Sucrose, as a pure carbohydrate, has a high food energy content (4 kilocalories per
gram or 17 kilojoules per gram), and thus can make a diet hypercaloric even in small
amounts, contributing to obesity.
Silver sulphate:

Silver sulfate is an ionic compound of silver. It is minimally soluble in water. 

**Source:** Loba Chemie, India.

**Purification:** Silver sulphate was recrystallized from concentrated sulfuric acid, cooled, then diluted with de-ionized water and the precipitate was filtered, washed and dried at 120°C.

**Application:** Silver sulfate is used in silver plating and as a non-staining substitute to silver nitrate. This sulfate is stable under ordinary conditions of use and storage, though it darkens upon exposure to air or light.

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Thorium nitrate:

Thorium Nitrate is a white crystalline mass, mildly toxic and not combustible but accelerates burning of combustible materials. It may explode in a fire if in large quantities or if the combustible material is finely divided. Toxic oxides of nitrogen are produced in fires. It is mildly radioactive.

**Source:** Thomas Baker, India, purity 99%
**Application**: Thorium is a lanthanide (rare earth) material with potential nuclear power applications. It is also used in several other high temperature glass applications, such as in the mantle of lamps and to produce crystal growth crucibles and ampules. The nitrate is extensively used in the commercial production of gas mantles. Such mantles are made by impregnating cotton or synthetic fibres with a 25 to 50 percent solution of Th(NO$_3$)$_4$.

**Oxalic acid**: Oxalic Acid (also called Ethanedioic Acid) is a toxic organic compound belonging to the family of dicarboxylic acids. It occurs in the form of its metal salts (usually calcium or potassium) in many plants. It is commercially manufactured by heating sodium formate in the presence of an alkali catalyst to form sodium oxalate, which should be converted to free oxalic acid when treated with sulfuric acid. It is also prepared by oxidizing carbohydrates with nitric acid, by heating saw dust with caustic alkalies or by fermentation of sugar solutions in the presence of certain molds. Oxalic acid is the only possible compound in which two carboxyl groups are joined directly; for this reason oxalic acid is one of the strongest acids in organic compounds.

**Source**: Analytical Reagent Grade.

**Purification**: Oxalic acid was used after drying over P$_2$O$_5$ in a desiccator for more than 24 hours.

**Application**: Unlike other carboxylic acids, oxalic acid is readily oxidized and combines with calcium, iron, sodium, magnesium, or potassium to form less soluble salts called oxalates. Oxalic acid and oxalates are useful as reducing agents for photography, bleaching, and rust removal. They are widely used as a purifying

<table>
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<tr>
<th>Appearance:</th>
<th>White Crystalline Solid</th>
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<tbody>
<tr>
<td><strong>Molecular Formula</strong>:</td>
<td>(COOH)$_2$.2H$_2$O</td>
</tr>
<tr>
<td><strong>Molecular Weight</strong>:</td>
<td>126.07 g/mol</td>
</tr>
<tr>
<td><strong>Boiling point</strong>:</td>
<td>(149-160)$^\circ$C</td>
</tr>
<tr>
<td><strong>Melting Point</strong>:</td>
<td>(101-102)$^\circ$C</td>
</tr>
</tbody>
</table>
agent in pharmaceutical industry, precipitating agent in rare-earth metal processing, bleaching agent in textile and wood industry, rust-remover for metal treatment, grinding agent, waste water treatment, acid rinse in laundries and removing scale from automobile radiators. Plasticizer for polymers, biodegradable solvents and lubricants, engineering plastics, epoxy curing agent, adhesive and powder coating, corrosion inhibitor, perfumery and pharmaceutical, and also as an electrolyte

**Sodium Nitrate:**

A nitrate mineral having chemical composition NaNO$_3$ (sodium nitrate); also known as nitratite, it is by far the most abundant of the nitrate minerals. It sometimes occurs as simple rhombohedral crystals but is usually massive granular.

**Source:** Ranbaxy Laboratories limited.

**Purification:** Sodium nitrate was purified as crystallised from hot water by cooling to 0°C Dried under vacuum at 140°C.

**Application:** Sodium nitrate is used as an ingredient in fertilizers, pyrotechnics, as an ingredient in smoke bombs, as a food preservative, and as a solid rocket propellant, as well as in glass and pottery enamels.

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<thead>
<tr>
<th><strong>Sodium Nitrate</strong></th>
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<td><strong>Appearance:</strong></td>
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<td><strong>Molecular Formula:</strong></td>
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<tr>
<td><strong>Molecular Weight:</strong></td>
</tr>
<tr>
<td><strong>Boiling Point:</strong></td>
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<tr>
<td><strong>Melting Point:</strong></td>
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</tbody>
</table>
**Potassium Nitrate:**

Potassium nitrate has an orthorhombic crystal structure at room temperature, which transforms to a trigonal system at 129 °C. Upon heating to temperatures above 560 °C, it decomposes into potassium nitrite, generating oxygen. Potassium nitrate is a strong oxidizer which burns and explodes with organics. It occurs as a mineral niter and is a natural solid source of nitrogen.

**Source:** Sd. fine chemicals, India.

**Purification:** Potassium nitrate was crystallised from hot water by cooling. Dried for 12hr under vacuum at 70°C.

**Application:** Potassium nitrate is mainly used in fertilizers, as a source of nitrogen and potassium—two of the macro nutrients for plants. When used by itself, it has an NPK rating of 13-0-44. It is used in the manufacture of gunpowder. It is also used in explosives, matches, and fertilizers, and as a preservative in foods especially meats. It is sometimes used in medicine as a diuretic. Potassium nitrate prill type is used to produce kinescope, optics glass, high grade craft glassware rocket propellants and fireworks.

Potassium nitrate is also one of the three components of black powder, along with powdered charcoal (substantially carbon) and sulfur, where it acts as an oxidizer.

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<thead>
<tr>
<th>Property</th>
<th>Value</th>
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<tbody>
<tr>
<td>Appearance</td>
<td>White Crystalline Solid</td>
</tr>
<tr>
<td>Molecular Formula</td>
<td>KNO₃</td>
</tr>
<tr>
<td>Molecular Weight</td>
<td>101.1 g/mol</td>
</tr>
<tr>
<td>Boiling Point</td>
<td>400°C</td>
</tr>
<tr>
<td>Melting Point</td>
<td>333°C</td>
</tr>
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</table>
Lithium nitrate: Lithium nitrate is an inorganic compound with the formula LiNO₃. It is the lithium salt of nitric acid. Upon thermal decomposition, LiNO₃ gives lithium oxide (Li₂O), nitrogen dioxide, and oxygen. Because of its relatively small size, the lithium cation is very polarizing, which favors the formation of the oxide.

**Source:** Thomas Baker, India.

**Purification:** Lithium nitrate was crystallised from ethanol. It was dried at 180°C for several days by repeated melting under vacuum.

**Application:** Lithium nitrate is used as an electrolyte for high temperature batteries. It is also used for long life batteries as required, for example, by artificial pacemakers. The solid is used as a phosphor for neutron detection. Mainly used as aqueous ammonia stabilizer in refrigeration facility, rocket propellant, oxidizer for fireworks, etching agent for glass, static inhibitors, and as component of molten salts in metallurgical industry.

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<tr>
<th>Property</th>
<th>Value</th>
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<tr>
<td><strong>Appearance:</strong></td>
<td>White Crystalline Solid</td>
</tr>
<tr>
<td><strong>Molecular Formula:</strong></td>
<td>LiNO₃</td>
</tr>
<tr>
<td><strong>Molecular Weight:</strong></td>
<td>68.95 g/mol</td>
</tr>
<tr>
<td><strong>Boiling Point:</strong></td>
<td>255°C</td>
</tr>
</tbody>
</table>
**Sodium Molybdate:**

Sodium molybdate is useful as a source of molybdenum\(^3\) It is often found as the dihydrate, Na\(_2\)MoO\(_4\)\(\cdot\)2H\(_2\)O. The molybdate (VI) anion is tetrahedral. Two sodium cations coordinate with every one anion\(^4\).

**Source:** E. Merck, India

**Purification:** Sodium molybdate was purified by re-crystallizing twice from conductivity water and then dried in a vacuum desiccator over P\(_2\)O\(_5\) for 24 hours before use.

**Application:** The agriculture industry uses 1 million pounds per year as a fertilizer. In particular, its use has been suggested for treatment of whiptail in broccoli and cauliflower in molybdenum-deficient soils. However, care must be taken because at a level of 0.3 ppm sodium molybdate can cause copper deficiencies in animals, particularly cattle.

It is used in industry for corrosion inhibition, as it is a non-oxidizing anodic inhibitor. The addition of sodium molybdate significantly reduces the nitrite requirement of fluids inhibited with nitrite-amine, and improves the corrosion protection of carboxylate salt fluids.

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**Sodium Molybdate**

| Appearance: | white crystalline solid |
| Molecular Formula: | Na\(_2\)MoO\(_4\)\(\cdot\)2H\(_2\)O |
| Molecular Weight: | 241.95 g/mol |
| Melting Point: | 687°C |
**Phosphomolybdic acid:**

Phosphomolybdic acid, also known as dodeca molybdophosphoric acid or PMA is a component of Masson's trichrome stain. It is a yellow-green compound, freely soluble in water and polar organic solvents such as ethanol

**Source:** Thomas Baker, India

**Application:** Phosphomolybdic acid is used as a reagent in thin layer chromatography for staining phenolics, hydrocarbon waxes, alkaloids and steroids. Conjugated, unsaturated compounds reduce PMA to molybdenum blue. The colour intensifies with increasing number of double bonds in the molecule being stained.

<table>
<thead>
<tr>
<th><strong>Appearance</strong></th>
<th>Solid yellow crystals</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Molecular Formula</strong></td>
<td>$\text{H}<em>3\text{Mo}</em>{12}\text{O}_{40}\text{P}$</td>
</tr>
<tr>
<td><strong>Molecular Weight</strong></td>
<td>1825.25 g/mol</td>
</tr>
<tr>
<td><strong>Melting Point</strong></td>
<td>(78-90)°C</td>
</tr>
</tbody>
</table>

**Cetrimonium bromide:**

Cetrimonium bromide is one of the components of the topical antiseptic. It is a cationic surfactant. As any surfactant, it forms micelles in aqueous solutions. At 303 K (30 °C) it forms micelles with aggregation number 75-120 (depending on method of determination, usually average ~95) and degree of ionization $\alpha$ (fractional charge) 0.2 - 0.1 (from low to high concentration). The standard constant of Br-counterion binding to the micelle at 303 K (30 °C) is $K^\circ \approx 400$. This value is calculated from Br- and CTA+ ion selective electrode measurements and conductometry data by using literature data for micelle size ($r = \sim 3$ nm),
extrapolated to the critical micelle concentration. However, it varies with total surfactant concentration so it is extrapolated to the point at which the concentration of micelles is zero.

**Source:** Thomas Baker, India >99%

**Application:** Cetrimonium bromide is widely used as active ingredient for hair conditioners, detergent sanitizers, disinfection agents and softener for textiles and paper products. Cetrimonium bromide ((C_{16}H_{33})N(CH_{3})_{3}Br) is one of the components of the topical antiseptic cetrimide. The cetrimonium (or hexadecyltrimethylammonium) cation is an effective antiseptic agent against bacteria and fungi. Its uses include providing a buffer solution for the extraction of DNA. It has been widely used in synthesis of gold nanoparticles (e.g., spheres, rods, bipyramids). It is also widely used in hair conditioning products. The closely related compounds cetrimonium chloride and cetrimonium stearate are also used as topical antiseptics, and may be found in many household products such as shampoos and cosmetics, while cetrimonium bromide, due to its high cost, is only found in select cosmetics.

**Nicotinamide:**

Nicotinamide, commonly known as vitamin B_{3}, is a water soluble vitamin, an essential micronutrient, and a reactive moiety of the coenzyme nicotinamide adenine dinucleotide (NAD). It is an essential part of the coenzyme nicotinamide adenine dinucleotide phosphate (NADP).

**Source:** ACROS Organics Company, purity > 98 %
**Application**: Nicotinamide plays a very important role to maintain the normal function of the digestive systems and cholesterol levels in the human body. The combination of nicotinic acid and nicotinamide is clinically referred to as niacin, since nicotinic acid is converted in the body into the amide very fast, and for nutritional purposes, both of them have equal biological activities. Nicotinamide is an interesting molecule because of its two nitrogen atoms – one in the heterocyclic ring and the other as the amide group.

<table>
<thead>
<tr>
<th>Nicotinamide</th>
<th>Appearance:</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Molecular Formula:</td>
<td>C₆H₆N₂O</td>
<td></td>
</tr>
<tr>
<td>Molecular Weight:</td>
<td>122.13 g/mol</td>
<td></td>
</tr>
<tr>
<td>Boiling Point:</td>
<td>(150-160)°C</td>
<td></td>
</tr>
<tr>
<td>Melting Point:</td>
<td>130 °C</td>
<td></td>
</tr>
</tbody>
</table>

**Resorcinol**: There are three isomeric compounds of dihydroxybenzene molecule structure, which all have traditional names. The meta (1,3) isomer is resorcinol (also known as resorcin), which forms clear needle crystals. Resorcinol is a dihydroxy phenol, which are readily soluble in water, alcohol and ether, but insoluble in chloroform and carbon disulfide. It reduces Fehling’s solution, and ammoniacal silver solutions. It does not form a precipitate with lead acetate solution, as the isomeric pyrocatechol does. Iron(III) chloride

<table>
<thead>
<tr>
<th>Resorcinol</th>
<th>Appearance:</th>
<th>White crystalline solid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular Formula:</td>
<td>C₆H₄(OH)₄</td>
<td></td>
</tr>
<tr>
<td>Molecular Weight:</td>
<td>110.1 g/mol</td>
<td></td>
</tr>
<tr>
<td>Boiling Point:</td>
<td>277°C</td>
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<tr>
<td>Melting Point:</td>
<td>110 °C</td>
<td></td>
</tr>
</tbody>
</table>
colors its aqueous solution a dark violet, and bromine water precipitates tribromoresorcin. These properties are what give it its use as a colouring agent for certain chromatography experiments.

**Source:** Sd. fine Chemical Limited, India.

**Purification:** Resorcinol was purified by standard methods and the compound was dried and stored in a vacuum desiccator.

**Application:** Resorcinol is an important organic compound, used externally as an antiseptic and disinfectant. It is also used as a chemical intermediate for the synthesis of pharmaceuticals and some organic compounds. An emerging use of resorcinol is as a template molecule in supramolecular chemistry. The -OH groups on resorcinol form hydrogen bonds to target molecules holding them in the proper orientation for a reaction resorcinol is readily soluble in water through hydrogen bonding.

**Caffeine:**

Caffeine is an alkaloid. Alkaloids are a broad category of nitrogen containing organic metabolites produced by plants. Caffeine- an additive to popular carbonated drinks exists widely in the leaves, seeds, and fruits of a large number of plants. Among them, cocoa beans, tea, coffee, cola, and guarana are the best known.

**Source:** Loba Chemie, India.

**Purification:** Caffeine was purified by dissolving it in aqueous ethanol and then recrystallized.

**Application:** Three naturally occurring purine base xanthine alkaloids, caffeine is the most significant stimulant to central nervous system. It has a wide

<table>
<thead>
<tr>
<th>Appearance:</th>
<th>White crystalline</th>
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<tbody>
<tr>
<td>Molecular Formula:</td>
<td>C$<em>{6}$H$</em>{10}$N$<em>{4}$O$</em>{2}$</td>
</tr>
<tr>
<td>Molecular Weight:</td>
<td>194.19 g/mol</td>
</tr>
<tr>
<td>Melting Point:</td>
<td>178°C</td>
</tr>
<tr>
<td>Boiling Point:</td>
<td>238°C</td>
</tr>
</tbody>
</table>
range of effects on cardiovascular activity including vasoconstriction and so forth. It produces acute elevations in both systolic and diastolic blood pressure in most individuals but as a complex forming agent decreases the effective concentration of the anti-tumor antibiotic actinomycin D. It is teratogenic and causes inhibition of DNA repair, inhibition cyclic AMP phosphodiesterase activity and inhibits seed germination. It can be a cause of cancer, heart diseases and complications in pregnant women and ageing. It is a diuretic, has applications in bilirubin determination in neonates with a clearing influence on the turbidity of human sera. As drug it finds extensive applications in pharmacological preparations including analgesics, diet aids, and cold/flu remedies. However, it may produce confusion, tremours, insomenia and excitement leading to mild delirium. Caffeine also finds applications as a chemical marker in detecting sources of domestic water pollution and as antioxidants.

**Quaternary ammonium iodide:**

Quaternary ammonium compounds \((\text{R}_4\text{N}^+)\) are any of 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). The organic radicals \(R\) may be alkyl, aryl, or aralkyl, and the nitrogen can be part of a ring system. They are prepared by treatment of an amine with an alkylation 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 microorganisms’ cell processes.

**Source:** Sigma Aldrich, Germany.

**Application:** These compounds are used as, Surface-active agents, solvents, intermediates, active ingredient for conditioners, antistatic agent, detergent sanitizers, softener for textiles and paper products, Phase Transfer Catalyst, Antimicrobials, Disinfection Agents and Sanitizers, Slimicidal Agents, Algaecide, Emulsifying Agents, Pigment Dispersers

### 3.2. Experimental Method

#### 3.2.1. Measurement of Density

Densities ($\rho$) were measured with an Ostwald-Sprengel type pycnometer having a bulb volume of 25 cm$^3$ and an internal diameter of the capillary of about 1 mm. The pycnometer was calibrated at 298.15 K with doubly distilled water and THF. The total uncertainty in density was estimated to be ±0.0001 g cm$^{-3}$.

The measurements were carried out in a thermostatic water bath (Science India, Kolkata) maintained with an accuracy of ± 0.01 K of the desired temperature. A 60W heating element and a toluene-mercury thermo-regulator were used to maintain the temperature of the experimental thermostat which was placed in a hot-cum-cold thermostat. The temperature of the hot-cum-cold thermostat was preset at the desired temperature using a contact thermometer and relay system. The absolute temperature was determined by a calibrated platinum resistance thermometer and Muller bridge $^6$-$^8$. 
Experimental Section

Density meter\(\text{(Anton Paar(DMA 4500 M) GmbH, Austria-Europe)}\)

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.

\[ \rho = A\tau^2 - B \] ..........................(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 parasitic 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.

3.2.2. Measurement of Viscosity

The kinematic viscosities were measured by means of a suspended-level Ubbelohde viscometer. The time of flow was measured with a stop watch. The viscometer was always kept in a vertical position in the water-bath. The viscometer needed no correction for kinetic energy.
The kinematic viscosity ($\gamma$) and the absolute viscosity ($\eta$) are given by the following equations:

$$\gamma = kt - 1 / t \quad \text{...............}(2)$$

$$\eta = \gamma \cdot \rho \quad \text{...............}(3)$$

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 $\pm 0.004 \%$. 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:

$$\eta_r = \eta / \eta_0 = \rho t / \rho_0 t_0 \quad \text{...............}(4)$$

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 measurements were carried out in a thermostatic water bath maintained with an accuracy of $\pm 0.01 \text{ K}$ of the desired temperature.

3.3. Instruments available in our Department:

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**
Rotary Vacuum Flash Evaporator

A rotary evaporator (or rotavap) is a device used in chemical laboratories for the efficient and gentle removal of solvents from samples by evaporation. When referenced in the chemistry research literature, description of the use of this technique and equipment may include the phrase "rotary evaporator", though use is often rather signaled by other language (e.g. "the sample was evaporated under reduced pressure").

Rotary evaporation is most often and conveniently applied to separate "low boiling" solvents such as n-hexane or ethyl acetate from compounds which are solid at room temperature and pressure. However, careful application also allows removal of a solvent from a sample containing a liquid compound if there is minimal co-evaporation (azeotropic behavior), and a sufficient difference in boiling points at the chosen temperature and reduced pressure.

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

**Digital electronic analytical balance:**

The mass measurements accurate to ±0.01 mg were made on a digital electronic analytical balance (Mettler Toledo, AG 285, Switzerland). An analytical balance is used to measure mass to a very high degree of precision and accuracy. The weighing pan(s) of a high precision (.01 mg or better) analytical balance are 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.

The use of a vented balance safety enclosure, which has uniquely designed acrylic airfoils, allows a smooth turbulence-free airflow that prevents balance fluctuation and the weighing of mass down to 1 µg without fluctuations or loss of product. Also, the sample must be at room temperature to prevent natural convection from forming air currents inside the enclosure, affecting the weighing.
Ultrasonic Interferometer

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.

The principle used in the measurement of the ultrasonic speed \( u \) 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.

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

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:

\[
d = n \times \frac{\lambda}{2}
\]

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

\[
K_s = \frac{1}{u^2 \rho}
\]

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
Conductivity Bridge

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.
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\(^\text{10}\). 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\(^\text{11}\).

Solutions were prepared by weight precise to ± 0.02 %. The weights were taken on a Mettler electronic analytical balance (AG 285, Switzerland). The molarities being converted to molalities 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
Abbe Refractometer (Cyberlab, MA01527, USA)

The refractive indices of pure liquids and their binary mixture were measured by using a thermostated Abbe refractometer. The values of refractive index were obtained using sodium D light. The uncertainty of refractive index measurements was within 0.0001. The thermostat temperature was constant to ±0.01 K. Water was circulated into the prism of the refractometer by a circulation pump connected to an external thermostated water bath.

Calibration was performed by measuring the refractive indices of double-distilled water, toluene, cyclohexane, and carbon tetrachloride at defined temperature. The sample mixtures were directly injected into the prism assembly of the instrument using an airtight hypodermic syringe, and an average of four measurements was taken for each mixture.

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

\[
\text{refractive index (n) of substance} = \frac{\text{speed of light in a vacuum}}{\text{speed of light in substance}}
\]
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:

\[
\frac{V_A}{V_B} = \frac{\sin \theta_A}{\sin \theta_B} = \frac{n_B}{n_A}
\]

Thus, it is not necessary to measure the speed of light in a sample in order to determine its index of refraction. Instead, by measuring the angle of refraction, and knowing the index of refraction of the layer that is in contact with the sample, it is possible to determine the refractive index of the sample quite accurately. Nearly all refractometers utilize this principle, but may differ in their optical design.
In the Abbe’ refractometer the liquid sample is sandwiched into a thin layer between an illuminating prism and a refracting prism (Figure 2). The refracting prism is made of a glass with a high refractive index (e.g., 1.75) and the refractometer is designed to be used with samples having a refractive index smaller than that of the refracting prism.

A light source is projected through the illuminating prism, the bottom surface of which is ground (i.e., roughened like a ground-glass joint), so each point on this surface can be thought of as generating light rays traveling in all directions. Inspection of Figure 2 shows that light traveling from point A to point B will have the largest angle of incidence ($\theta_i$) and hence the largest possible angle of refraction ($\theta_r$) for that sample. All other rays of light entering the refracting prism will have smaller $\theta_r$ and hence lie to the left of point C. Thus, a detector placed on the back side of the refracting prism would show a light region to the left and a dark region to the right.

**IR Spectrophotometer (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 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 solute that absorbs light. Second, the intensity of light \((I)\) passing through the sample solution is measured. (In practice, instruments measure 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.) Third, the experimental data is used to calculate two quantities: the transmittance \((T)\) and the absorbance \((A)\).

\[
T = \frac{I}{I_0} \quad A = -\log_{10} T \quad \text{..................(10)}
\]

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

**UV/Visible Spectrophotometer**

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-Visible spectrophotometer uses two light sources, a deuterium (D\(_2\)) lamp for ultraviolet light and a tungsten (W) lamp for visible light. After bouncing off 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) successfully passes through a slit. A filter is used to remove unwanted higher orders of diffraction. (Recall the experiment you did last semester on Atomic Spectra) The light beam hits a second mirror before it gets split by a half mirror (half of the light...
is reflected, the other half passes through). One of the beams is allowed to pass through a 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.

**Beer-Lambert Law**

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

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

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

(iii) intensity of the incident light \((I)\), more photons mean more opportunity for a molecule to see a photon. Thus, \( dl \) is proportional to \( bcl \) or \( dl/I = -kbc \) (where \( k \) is a proportionality constant, the negative sign is shown because this is a decrease in intensity of the light, this makes \( b, c \) and \( I \) always positive.

Integration of the above equation leads to Beer-Lambert’s Law:

\[
-ln\frac{I}{I_0} = kbc \quad \text{...............(11)}
\]

\[
-log\frac{I}{I_0} = 2.303kbc \quad \text{...............(12)}
\]

\[
\varepsilon = 2.303k \quad \text{...............(13)}
\]

\[
A = -log\frac{I}{I_0} \quad \text{...............(14)}
\]

\[
A = \varepsilon bc \quad \text{...............(15)}
\]

A is defined as absorbance and it is found to be directly proportional to the path length, \( b \), and the concentration of the sample, \( c \). The extinction coefficient is characteristic of the substance under study and of course, is a function of the wavelength. Molecules strongly absorb only in some regions of the electromagnetic spectrum. The photon carries a specific amount of energy defined by its wavelength (Recall Planck’s equation: \( E = hc/\text{wavelength} \)). The molecule will only absorb a photon if the energy it carries matches a certain amount the molecule can use. In the
ultraviolet-visible region, this energy corresponds to electronic excitations (promotion of electrons from occupied orbitals to unoccupied orbitals). The longest wavelength (the least energy) therefore corresponds to the energy difference between the ground and the first excited state (or promotion of an electron from the highest filled orbital (HFO) to the lowest unfilled orbital (LUO)).
References: