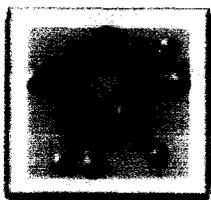


CHAPTER-III

A. SOURCE & PURIFICATION OF SOLVENTS AND CHEMICALS

III.A.1. Solvents:

1. Tetrahydrofuran

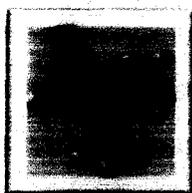


Tetrahydrofuran and its mixtures are the very important solvents widely used in various industries. This is a good industrial solvent and figures prominently in the high-energy battery technologies and has sound its application in organic syntheses as manifested from the physico-chemical studies in this medium [1-3].

Molecular formula:	C₄H₈O
Molar mass:	72.11 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.8807 g·cm⁻³ [3]

- Source:** Merck, India.
- Purification:** It was kept for several days over KOH, refluxed for 24 h, and distilled over LiAlH₄ [3,4]. The purity of the solvent finally obtained was > 99.0 %.

2. N, N-Dimethylformamide



N,N-Dimethylformamide is a colourless liquid is miscible with water and the majority of organic liquids. Dimethylformamide is a common solvent for chemical

reactions. Pure dimethylformamide is odorless. It is also used in the separation of saturated and unsaturated hydrocarbons and serves as solvents for vinyl resins, acid gases, polyacrylic fibres and catalyst in carbonylation reaction as well as in organic synthesis. It has also been used as the model of peptide linkage in studies aimed at understanding of protein denaturation studies [5-6].

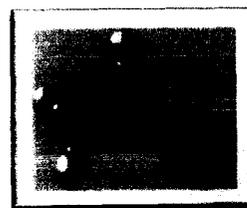
Molecular formula:	C₃H₇NO
Molar mass:	73.09 g·mol⁻¹
Appearance:	clear liquid
Density (25°C):	0.9443 g·cm⁻³ [3]

a) **Source:** Merck, India.

b) **Purification:** It was mixed with 10 % (by volume) benzene and the azeotrope were distilled off under atmospheric pressure at about 353 K. The product was dried over silica gel and distilled at reduced pressure, with the middle fraction being collected. The purified solvents were stored over P₂O₅ in a desiccator before use [7].

3. Acrylonitrile

Acrylonitrile is a pungent-smelling colorless liquid often appears yellow due to impurities. It is an important monomer for the manufacture of useful plastics. The study of mixtures of acrylonitrile in various solvents is of interest because of its wide use as an important industrial monomer for polyacrylonitrile as well as for investigating the effect of the simultaneous presence of the C=C double bond and the polar nitrile C≡N group on the molecular interactions.

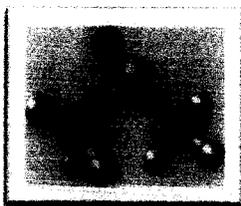


Molecular formula:	C₃H₃N
Molar mass:	73.09 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.8002 g·cm⁻³ [8]

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

b) **Purification:** It was washed with dilute H₂SO₄, then with dilute Na₂CO₃ and water, and again dried over anhydrous CaCl₂ and distilled fractionally [9].

4. Dimethylsulphoxide



Dimethylsulfoxide is an organosulphur compound. 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 has a distinctive property of penetrating the skin very readily, so that one may taste it soon after it comes into contact with the skin. Having low toxicity, it can be used in biology and medicine, especially for low-temperature preservation [10].

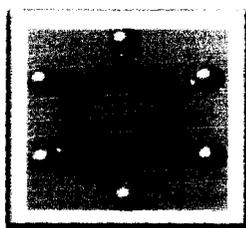
Molecular formula:	C₂H₆OS
Molar mass:	78.13 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	1.0960 g·cm⁻³ [3]

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

b) **Purification:** It was stored over 3 Å molecular sieves for 3 days before use. It is then refluxed for 4hr over CaO. The purity of the purified liquid was ascertained by Gas Liquid Chromatography and also by comparing

experimental values of densities, viscosities, and ultrasonic speeds of sound with their literature values [11].

5. Benzene



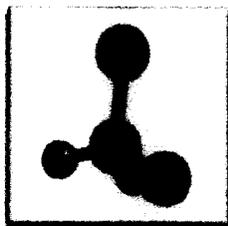
Benzene, or benzol, is an organic chemical compound which is a colorless and highly flammable liquid with a sweet smell and a relatively high melting point. Because it is a known carcinogen, its use as an additive in gasoline is now limited, but it is an important industrial solvent and precursor in the production of drugs, plastics, synthetic rubber, and dyes. Benzene is a natural constituent of crude oil, and may be synthesized from other compounds present in petroleum.

Molecular formula:	C₆H₆
Molar mass:	78.11 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.8734 g·cm⁻³ [12]

- a) **Source:** S.D. Fine Chemicals Ltd., Mumbai, India. (A. R. grade, purity > 99 %).
- b) **Purification:** It was purified by means of a simple distillation technique with the first and last 20 % of the distillate being discarded [9].

6. Dichloromethane

Dichloromethane or methylene chloride is an organic compound which is a colorless, volatile liquid with a moderately sweet aroma is widely used as a solvent. Although it is not miscible with water,

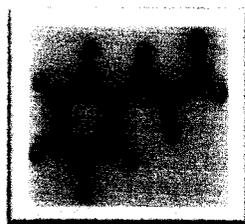


it is miscible with many organic solvents. Dichloromethane's volatility and ability to dissolve a wide range of organic compounds makes it a useful solvent for many chemical processes. It is a solvent exhibiting high density and low viscosity.

Molecular formula:	CH₂Cl₂
Molar mass:	84.93 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	1.3161 g·cm⁻³ [13]

- a) **Source:** Merck, India.
- b) **Purification:** It was purified by pre-drying with CaCl₂, and distilled from CaSO₄ and stored away from bright light over 4Å molecular sieves [9].

7. Cinnamaldehyde



Cinnamaldehyde is an organic compound that gives cinnamon its flavor and odor. This pale yellow viscous liquid occurs naturally in the bark of cinnamon trees and other species of the genus *Cinnamomum*. The essential oil of cinnamon bark is about 90 % cinnamaldehyde. The most obvious application for cinnamaldehyde is as flavoring in items like chewing gum, ice cream, candy, and beverages

Molecular formula:	C₉H₈O
Molar mass:	132.16 g·mol⁻¹
Appearance:	Yellow oil
Density (25°C):	1.0459 g·cm⁻³ [14]

- a) **Source:** S.D. Fine Chemicals Ltd., Mumbai, India.
- b) **Purification:** It was crystallized from benzene and dried at 60°C under vacuum ^[9]. The gas-liquid chromatography (GLC) analyses of these liquids indicated a mole fraction purity of > 99.0 %.

8. Benzaldehyde



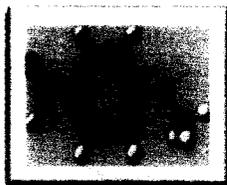
Benzaldehyde is an organic compound consisting of a benzene ring with a formyl substituent. It is the simplest aromatic aldehyde and one of the most industrially useful. This colorless liquid has a characteristic pleasant almond-like odor.

Benzaldehyde is used- (i) for flavouring purposes, in perfumery; (ii) in the manufacture of dyes; and (iii) as a starting material for the synthesis of several other organic compounds, e.g., cinnamaldehyde, cinnamic acid, benzoyl chloride, etc ^[15].

Molecular formula:	C₇H₆O
Molar mass:	106.12 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	1.0409 g·cm⁻³ [14]

- a) **Source:** S.D. Fine Chemicals Ltd., Mumbai, India.
- b) **Purification:** To prevent oxidation benzaldehyde usually contains additives such as catechol. After filtration benzaldehyde was washed with 10 % Na₂CO₃ (until no more CO₂ evolved), then with saturated Na₂SO₃ and water, followed by drying with CaCl₂ ^[9].

9. Anisaldehyde



Anisaldehyde, or anisic aldehyde, is an organic compound that consists of a benzene ring substituted with an aldehyde and a methoxy group. It is a clear colorless liquid with a strong aroma. Anisaldehyde is used as an intermediate in the synthesis of other compounds important in pharmaceuticals and perfumery. A solution of *para*-anisaldehyde in acid and ethanol is frequently used to stain thin layer chromatography plates.

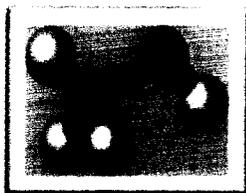
Molecular formula:	C₈H₈O₂
Molar mass:	136.15 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	1.1202 g·cm⁻³ [14]

- a) **Source:** S.D. Fine Chemicals Ltd., Mumbai, India.
- b) **Purification:** Anisaldehyde was washed with NaHCO₃, then with water, dried with anhydrous MgSO₄, and distilled [9].

ALCOHOLS

Alcohols and their aqueous and non-aqueous mixtures are widely used in pharmaceutical industry as excipients in different formulations or as solvents. Alcohols have varied applications in chemical and cosmetic industries. These are useful in enology and as an alternative energy source [16]. Knowledge of their physico-chemical characteristics helps to understand their behavior in a better way.

1. Methanol

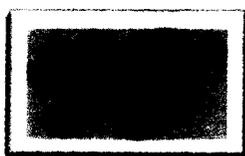


Methanol is used as solvent for paints and varnishes, antifreeze for automobile radiators, motor fuel, denaturant for ethanol, etc. It is obtainable commercially in adequate purity for most purposes, the impurity being up to 0.05 % water usually be removed by distillation, or by use of molecular sieves.

Molecular formula:	CH₄O
Molar mass:	32.04 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.7869 g·cm⁻³ [17]

- a) **Source:** Merck, India.
- b) **Purification:** It was dried over 4Å molecular sieves and then distilled fractionally. Middle fraction was collected and redistilled [18].

2. Ethanol



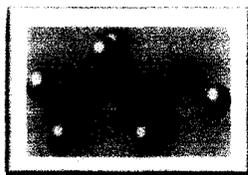
Ethanol has been used as a solvent in quantitative studies and 'Absolute' alcohol usually contains 0.01% water.

Molecular formula:	C₂H₅O
Molar mass:	46.1 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.7850 g·cm⁻³ [17]

- a) **Source:** Merck, India.

- b) **Purification:** It was dried over 4Å molecular sieves and then distilled fractionally. Middle fraction was collected and redistilled [18]

3. 1-Propanol



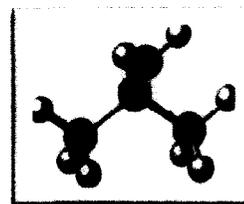
Propan-1-ol is a primary alcohol with the molecular formula of C_3H_8O . It is also known as 1-propanol, 1-propyl alcohol, n-propyl alcohol, n-propanol, or simply propanol. It is an isomer of propan-2-ol. It is used as a solvent in the pharmaceutical industry, and for resins and cellulose esters. It is formed naturally in small amounts during many fermentation processes.

Molecular formula:	C_3H_8O
Molar mass:	$60.10 \text{ g}\cdot\text{mol}^{-1}$
Appearance:	Colourless liquid
Density (25°C):	$0.7958 \text{ g}\cdot\text{cm}^{-3}$ [19]

- a) **Source:** Merck, India.
- b) **Purification:** It was dried over CaO for several hours, followed by distillation and a further drying.

4. 2-Propanol

It is a colorless, flammable chemical compound with a strong odor. It is the simplest example of a secondary alcohol. It is also known as iso-propyl alcohol and is readily available. Like acetone, it dissolves a wide range of non-polar compounds. It is

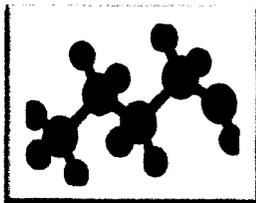


also relatively non-toxic and evaporates quickly. Thus it is used widely as a solvent and as a cleaning fluid, especially for dissolving lipophilic contaminants such as oil.

Molecular formula:	C₃H₈O
Molar mass:	60.10 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.7779 g·cm⁻³ [20]

- a) **Source:** Merck, India.
- b) **Purification:** It was dried over CaO for several hours, followed by distillation and a further drying.

5. 1- Butanol

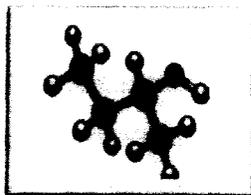


It is a primary alcohol with a 4-carbon structure. It is one of the group of “fusel alcohol” (from the German word for ‘bad liquor’), which has more than two carbon atoms and significant solubility in water. Industrial uses include the manufacture of pharmaceuticals, polymers, herbicide esters. It is also used as an ingredient in perfumes and as a solvent for the extraction of essential oils. It is also used in wide range of consumer goods.

Molecular formula:	C₄H₁₀O
Molar mass:	74.12 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.8060 g·cm⁻³ [17]

- a) **Source:** Merck, India.
- b) **Purification:** It was dried over CaO or solid NaOH followed by refluxing with, and distillation.

6. 2- Butanol



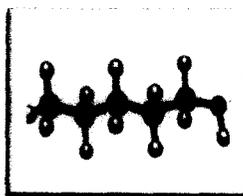
2-Butanol or sec-butanol, is a colourless liquid that is completely miscible with polar organic solvent such as ethers and other alcohols. It is produced on a large scale, primarily as a precursor to the industrial solvent methyl ethyl ketone. It occurs naturally as a product of fermentation of carbohydrates. It is used for the production of fruit essences, as a flavouring in food and as a solvent.

Molecular formula:	C₄H₁₀O
Molar mass:	74.12 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.8035 g·cm⁻³ [17]

- a) **Source:** Merck, India.
- b) **Purification:** It is dried over K₂CO₃, followed by filtration and fractional distillation refluxing with CaO, and distillation.

7. Amyl alcohol

Amyl alcohol is used fundamentally for the perfumes composition and the synthesis of fruit essences. They are also used as solvents for surfaces and lacquer baths, inks for print and dyes for wool as well as in the chemical production of photographic and pharmaceutical substances. Furthermore, they are an intermediate in the production of amyl acetate and other amyl esters.



Molecular formula:	C₅H₁₂O
Molar mass:	88.15 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.8110 g·cm⁻³ [21]

- a) **Source:** Merck, India.
- b) **Purification:** It was dried with anhydrous K₂CO₃ and distilled. The middle fractions for both the liquids were collected and kept free from humidity with 3 Å molecular sieves.

8. Iso-Amyl alcohol



Iso-amyl alcohol is used in research field for its usefulness in gas chromatography. It can isolate high quality RNA from even the hardest to isolate samples for immediate use in micro array application and it is also useful in most DNA applications [10].

Molecular formula:	C₅H₁₂O
Molar mass:	88.15 g·mol⁻¹
Appearance:	Colourless liquid
Density (25°C):	0.8071 g·cm⁻³ [21]

- a) **Source:** Merck, India.
- b) **Purification:** It was dried with anhydrous K₂CO₃ and distilled. The middle fractions for both the liquids were collected and kept free from humidity with 3 Å molecular sieves.

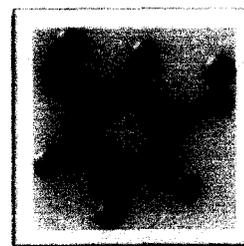
Water (H₂O, M.W. 18.016), was first deionized and then distilled in an all glass distilling set along with alkaline KMnO₄ solution to remove any organic matter [22] therein. The doubly distilled water was finally distilled using an all glass distilling set. Precautions were taken to prevent contamination from CO₂ and other impurities. The triply distilled water had specific conductance less than 1×10^{-6} S·cm⁻¹.

Densities and viscosities of the purified solvents were in good agreement with the literature values [3,12,13,14,17,19-21,23,24] and are listed in the respective chapters. The purity of most of the solvents finally obtained was better than 99.5 %. All solvents, pure or mixed, were stored over 3 Å molecular sieves for three days before use.

III.A.2. Solutes:

Resorcinol

Resorcinol (or resorcin) is a dihydroxy phenol. It is a white solid with molecular formula C₆H₆O₂. Used externally it is an antiseptic and disinfectant, and is used 5 to 10 % in ointments in the treatment of chronic skin diseases such as psoriasis and eczema. It can be included as an anti-dandruff agent in shampoo or in sunscreen cosmetics.



Resorcinol is also used as a chemical intermediate for the synthesis of pharmaceuticals and other organic compounds. It is used in the production of diazo dyes and plasticizers and as a UV absorber in resins.

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. Many such reactions are able to be carried out in the solid state thereby reducing or eliminating the use of solvents that may be harmful to the environment.

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

Purification: It 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 drop-wise. Fine plat like crystal separated and recovered by rapid filtration & ready for use.

Glycine

Glycine (abbreviated as Gly or G) ($C_2H_5NO_2$) (M.W. $75.07 \text{ g}\cdot\text{mol}^{-1}$) is an organic compound with the formula $C_2H_5NO_2$. With only a hydrogen atom as its side chain, glycine is the smallest of the 20 amino acids commonly found in proteins. Glycine is a colourless, sweet-tasting crystalline solid. It is unique among the proteinogenic amino acids in that it is not chiral. It can fit into hydrophilic or hydrophobic environments, due to its single hydrogen atom side chain. The principal function of glycine is as a precursor to proteins. It is also a building block to numerous natural products. Glycine is an inhibitory neurotransmitter in the central nervous system, especially in the spinal cord, brainstem, and retina.

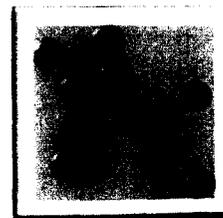


Source: Analar

Purification: It was purified by re-crystallizing from methanol-water mixture and dried at 373.15 K for 12 h in vacuum desiccator over P_2O_5 before use.

L-Alanine

Alanine (abbreviated as Ala or A) ($C_3H_7NO_2$) (M.W. $89.09 \text{ g}\cdot\text{mol}^{-1}$) is an α -amino acid with the chemical formula $C_3H_7NO_2$. The L-isomer is one of the 22 proteinogenic amino acids, i.e., the building blocks of proteins. It is classified as a non-polar amino acid. Alanine is a nonessential amino acid, meaning it can be manufactured by the human body, and does not need to be obtained directly through the diet. Alanine is found in a wide variety of foods, but is particularly concentrated in meats.

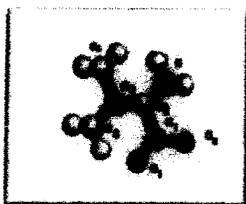


Source: S.D. Fine Chemicals Ltd., Mumbai, India.

Purification: It was purified by re-crystallizing from methanol-water mixture and dried at 373.15 K for 12 h in vacuum desiccator over P_2O_5 before use.

L-Valine

L-Valine (abbreviated as Val or V) ($C_5H_{11}NO_2$) (M.W.117.15 $g\cdot mol^{-1}$) is an α -amino acid. L-valine is one of the 22 proteinogenic amino acids, i.e., the building blocks of proteins. It is needed to keep the body in balance for greater muscle growth and recovery. It is one of the most important amino acid. When it comes to muscle building and energy, Valine is perhaps best known for its effects as a balancing agent of our bodies' nitrogen content. Valine has been shown to aid in correcting deficiencies created by drug addictions and as a supplemental treatment for those addictions.

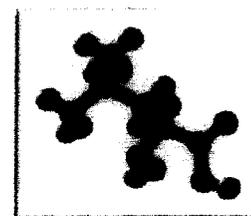


Source: Loba Chemie, India.

Purification: It was purified by re-crystallizing from methanol-water mixture and dried at 373.15 K for 12 h in vacuum desiccator over P_2O_5 before use.

L-Leucine

L-Leucine (abbreviated as Leu or L) ($C_6H_{13}NO_2$) (M.W.131.17 $g\cdot mol^{-1}$) is an α -amino acid. It is an essential amino acid, which means that humans cannot synthesize it. With a hydrocarbon side chain, it is classified as a hydrophobic amino acid. It helps to preserve lean muscle tissue; it supplies the body with energy when under stress. It maintains nitrogen balance, and it enhances thinking abilities that can decline as physical activities become more intense. Medically, L-Leucine has several uses.

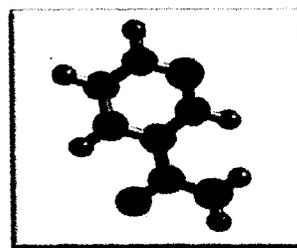


Source: Loba Chemie, India.

Purification: It was purified by re-crystallizing from methanol-water mixture and dried at 373.15 K for 12 h in vacuum desiccator over P_2O_5 before use.

Nicotinamide

Nicotinamide ($C_6H_6N_2O$) (M.W.122.12 g mol⁻¹), also known as niacinamide and nicotinic acid amide, is the amide of nicotinic acid (vitamin B₃ / niacin). Nicotinamide is a water-soluble vitamin and is part of the vitamin B group. Nicotinic acid, also known as niacin, is converted to nicotinamide *in vivo*, and, though the two are identical in their vitamin functions, nicotinamide does not have the same pharmacologic and toxic effects of niacin, which occur incidental to niacin's conversion. Thus nicotinamide does not reduce cholesterol or cause flushing, although nicotinamide may be toxic to the liver at doses exceeding 3 g/day for adults. In cells, niacin is incorporated into nicotinamide adenine dinucleotide (NAD) and nicotinamide adenine dinucleotide phosphate (NADP), although the pathways for nicotinamide and nicotinic acid are very similar. NAD⁺ and NADP⁺ are coenzymes in a wide variety of enzymatic oxidation-reduction reactions.

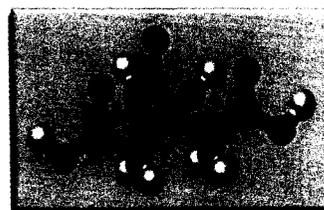


Source: Acros Organics Company.

Purification: Its mass purity as supplied is 98 %. The reagent was always placed in the desiccator over P_2O_5 to keep them in dry atmosphere before use.

Citric acid Monohydrate

Citric acid Monohydrate (2-Hydroxy-1,2,3-propanetricarboxylic acid, in IUPAC naming) ($C_6H_8O_7 \cdot H_2O$) (M.W.122.12 g mol⁻¹), is a colourless crystalline organic compound belong to carboxylic acid family. It exists in all plants (especially in lemons and limes) and in many animal tissues and fluids. In biochemistry, it is involved in important metabolism of almost all living things; the Krebs cycle (also called citric



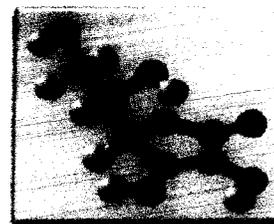
acid cycle or tricarboxylic acid cycle), a part of the process by which animals convert food to energy. Citric acid works as a preservative (or as an antioxidant) and cleaning agent in nature. It is commercially obtained by fermentation process of glucose with the aid of the mold *Aspergillus niger* and can be obtained synthetically from acetone or glycerol. It can be used as a sour taste enhancer in foods and soft drinks. The three carboxy groups lose protons in solution; resulting in the excellent pH control as a buffer in acidic solutions. It is used as a flavouring, stabilizing agent and acidulant (to control acidity) in food industry, in metal-cleaning compositions as it chelates metals. Citric acid is available in forms of anhydrous primarily and in monohydrate, the crystallized form from water. The hydrated form will be converted to the anhydrous form above 74°C. Citrate is a salt or ester of citric acid. Citrates are formed by replacing the acidic one, two, or all three of the carboxylic hydrogens in citric acid by metals or organic radicals to produce an extensive series of salts, esters, and mixed (double) salts. Citrates are used in food, cosmetics, pharmaceutical and medicine industries as well as in plastic industry; nutrient or food additives having functions of acidity regulator, sequestering and stabilizing agent, antioxidants synergist, firming agent; anticoagulant for stored whole blood and red cells and also for blood specimens as citrates chelate metal ions and saline cathartics, effervescent medicines; high boiling solvent, plasticizer and resin for food contact plastics.

Source: Himedia, India.

Purification: Its mass purity as supplied is 99%. The reagent was always placed in the desiccator over P_2O_5 to keep them in dry atmosphere before use.

Ascorbic acid

Ascorbic acid ($C_6H_8O_6$) (M.W.176.12 g mol⁻¹), (5R)-[[[(1S)-1,2-dihydroxyethyl]-3,4-dihydroxyfuran-2-(5H)-one]] is a sugar acid with antioxidant properties. Its appearance is white to light-yellow crystals or powder, and it is water-soluble. One form of ascorbic acid is commonly known



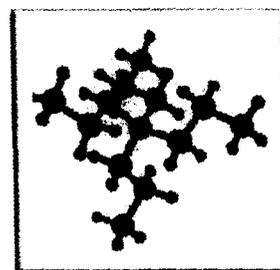
as vitamin C. The name is derived from *a-* (meaning "no") and *scorbutus* (scurvy), the disease caused by a deficiency of vitamin C. In 1937 the Nobel prize for chemistry was awarded to Walter Haworth for his work in determining the structure of ascorbic acid (shared with Paul Karrer, who received his award for work on vitamins), and the prize for Physiology or Medicine that year went to Albert Szent-Györgyi for his studies of the biological functions of L-ascorbic acid.

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

Purification: Its mass purity as supplied is 98%. Ascorbic acid was recrystallised twice from aqueous ethanol solution and dried under vacuum at $T = 348$ K for 6 hr. Thereafter, it was stored over P_2O_5 in a desiccator before use [25].

Tetraalkylammonium salts

Quaternary ammonium compounds 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 and one acid radical. The organic radicals 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 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 introduces continuity between the two different phase, they have properties of disrupting micro-organisms' cell processes. These compounds are used as: (i) Surface-active agents; (ii) Solvents; (iii) Detergent Sanitisers; (iv) Phase Transfer Catalyst; (v) Emulsifying Agents, etc.



Source: Tetraalkylammonium bromides, viz., tetra n-butylammonium bromide, tetra n-pentylammonium bromide, tetra n-hexylammonium bromide, tetra n-heptylammonium bromide; tetraalkylammonium iodides, viz., tetra n-

butylammonium iodide, tetra n-pentylammonium iodide, tetra n-hexylammonium iodide, tetra n-heptylammonium iodide were of Fluka's purum or puriss grade.

Purification: These were purified by dissolving in mixed alcohol medium and recrystallised from solvent ether medium. After filtration, the salts were dried in an oven for few hours. The crystallised salts were dried in vacuum. The salts were stored in glass bottles in darkened desiccator over fused CaCl_2 [9,26-28].

The purity of the solutes finally obtained was better than 99.0 % as checked by melting point determination.

Stock solutions of different salts/solutes in different mixed solvents and in pure solvents were prepared by mass and the working solutions were prepared by mass dilution. The conversion of molality into molarity was accomplished using the experimental density values. Great care was taken in minimizing evaporation losses and preventing moisture pick-up. All solutions were prepared afresh before use.

III.A.3. Mixed Solvents

The research work has been carried out with binary or ternary solvent systems with tetrahydrofuran, N,N-dimethylformamide, acrylonitrile, dimethylsulfoxide, benzene, dichloromethane, some aromatic aldehydes viz. cinnamaldehyde, anisaldehyde and benzaldehyde, some monoalkanols viz. methanol, ethanol, 1-propanol, 2-propanol, 1-butanol, 2-butanol, 1-pentanol and iso-amyl alcohol as primary solvents with some polar, weakly polar and non-polar solvents as well as with some solutes.

For the preparation of mixed binary and ternary mixtures, 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 binaries and

ternaries 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.

The following different binary and ternary solvent mixtures have been prepared and used for my research studies:

Binary Systems Studied:

1. Acrylonitrile (1) + Cinnamaldehyde (2).
2. Acrylonitrile (1) + Anisaldehyde (2).
3. Acrylonitrile (1) + Benzaldehyde (2).

Ternary Systems Studied:

1. Tetrahydrofuran (1) + Dimethylsulphoxide (2) + Methanol (3).
2. Tetrahydrofuran (1) + Dimethylsulphoxide (2) + Ethanol (3).
3. Tetrahydrofuran (1) + Dimethylsulphoxide (2) + 1-Propanol (3).
4. Tetrahydrofuran (1) + Dimethylsulphoxide (2) + 2-Propanol (3).
5. Tetrahydrofuran (1) + Dimethylsulphoxide (2) + 1-Butanol (3).
6. Tetrahydrofuran (1) + Dimethylsulphoxide (2) + 2-Butanol (3).
7. Tetrahydrofuran (1) + Dimethylsulphoxide (2) + Amyl alcohol (3).
8. Tetrahydrofuran (1) + Dimethylsulphoxide (2) + *iso*-Amyl alcohol (3).

Solute-solute / ion-ion and solute-solvent / ion-solvent interactions studied in the following mixed solutions:

1. Resorcinol (1) + Water (2) + Glycine (3).
2. Resorcinol (1) + Water (2) + L-Alanine (3).
3. Resorcinol (1) + Water (2) + L-Valine (3).
4. Resorcinol (1) + Water (2) + L-Leucine (3).

5. Tetra n-butylammonium bromide (1) + Water (2) + Ascorbic acid (3).
6. Nicotinamide (1) + Water (2) + Citric acid monohydrate.
7. Dichloromethane (1) + N,N-dimethylformamide (2) + Tetra n-butylammonium iodide (3).
8. Dichloromethane (1) + N,N-dimethylformamide (2) + Tetra n-pentylammonium iodide (3).
9. Dichloromethane (1) + N,N-dimethylformamide (2) + Tetra n-hexylammonium iodide (3).
10. Dichloromethane (1) + N,N-dimethylformamide (2) + Tetra n-heptylammonium iodide (3).

