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Introductory Chapter: An Outline of Chemical Reagents and Reactions in Inorganic Synthesis

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1. Introduction

A substance that is used in a chemical reaction to detect, measure, examine, or produce other substances is known as chemical reagent. A reagent in chemical science is a “substance or compound that is added to a system in order to bring a chemical reaction or is added to check whether a reaction is occurred or not.” Such a reaction is used to confirm the detection of the presence of another substance. Although the terms “reactant and reagent” are often used interchangeably, a reactant is more specifically a “substance that is consumed in the course of a chemical reaction.” Commonly, the involvement of solvents and catalysts in the course of a chemical reaction is not considered as reactants. Small organic molecules and metal salts/compounds in organic chemistry play a significant role in different organic reactions of laboratory and industrial importance [1].

Grade of chemical reagents remains very important in inorganic synthesis. Purity of a chemical agent can also be expressed in terms of the grade. Chemical reagents in scientific community are commonly found as spectroscopic grade, analytical grade (A.R. grade), synthetic grade, and laboratory grade (L.R. grade). Depending on the nature of uses, specially qualitative or quantitative, one can select the grade of reagents. Whatever the chemical substances we commonly use, a minimum quality must be maintained, and the purity of the chemical substances is determined by few organizations of international standard like ASTM International.

Inorganic chemistry, which is serving as a fundamental branch of chemistry, deals with the synthesis and behavior of inorganic and organometallic compounds. This field covers all chemical compounds consisting of most of the chemical bonding comprising ionic, covalent, coordinate, μ -bonding, δ -bonding, H-bonding, weak interactions, and so on. The important aspects of chemical bonding in inorganic compounds reflect their physicochemical properties

and the materialistic application in industrial catalysis, therapeutic agents, optical materials, magnetic materials, and conducting materials [2]. This chapter describes an overview of chemical regents using inorganic chemical reactions for the synthesis of different compounds including coordination, organometallic, cluster, bioinorganic, solid state compounds, and so on.

2. Inorganic reaction types

A chemical reaction is basically a process in which an integral chemical change of a reactant is taken place during the conversion of reactant to products. In the chemical reactions, electronic motion involves to head a bond making and breaking. Different types of chemical reactions in inorganic chemistry may be classified more than a single way. Only some of the fundamental broad inorganic chemical reactions are categorized in the following.

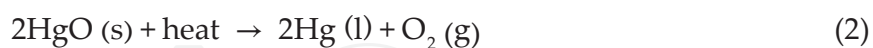
2.1. Combination reactions

This is a type of a chemical reaction in which two or more reactants react themselves to produce a product. Elemental sulfur reacts with molecular oxygen to form sulfur dioxide is an example of combination reaction.



2.2. Decomposition reactions

Often, decomposition reactions are considered as an opposite type of combination reactions. A chemical entity in the decomposition reaction breaks down into two or more substances. Decomposition usually results from electrolysis or heating. An example of a decomposition reaction is the breakdown of mercury(II) oxide into its component elements.



2.3. Displacement reactions

A displacement reaction is occurred during the replacement of an atom or ion with another atom or ion in a compound. An example of a single displacement reaction is the displacement of copper ion in a copper sulfate solution by zinc metal ion to form zinc sulfate.



3. Classification of inorganic compounds

Inorganic chemistry includes different types of compounds comprising coordination compounds, simple metal compounds, organometallic compounds, cluster molecules, biological

compounds, supramolecules, coordination polymers, catalysts, magnetic compounds, and others [3]. A brief discussion on some classes of inorganic compounds is presented.

3.1. Coordination compounds

Coordination compounds cover a wide fundamental area in inorganic chemistry and primarily deal with coordination bonding between a ligand and a metal ion. Classical coordination compounds feature metals bound to “lone pairs” of electrons residing on the main group atoms of ligands such as H_2O , NH_3 , Cl^- , and CN^- . In modern coordination compounds, large numbers of ligands with different coordination motifs are available. Most of the metal complexes contain principally 3D metal ions with different ligand backbones. A. Werner, the founder of coordination chemistry showed different classes of coordination molecules. He also classified the ligands as classical/Werner type and non-classical/non-Werner type ligands. Commonly, in coordination compounds ligands are dominated by the elements belonging to groups 13–17 [4]. On the other hand, Schiff bases, a very common and widely used ligand systems, named after H. Schiff, are the condensation products between primary amines and aldehydes or ketones ($\text{RCH}=\text{NR}'$, where R and R' represent alkyl and aryl substituents), which are used for the preparation of complex compounds [5].

3.2. Transition metal compounds

Compounds with metal ions from group 4 to 11 are considered transition metal compounds. Compounds with a metal from group 3 or 12 are sometimes also incorporated into this group but often classified as main group compounds. Transition metal compounds also show rich coordination geometry, varying from coordination number 3 to 12, although tetrahedral, square planar, square pyramidal/trigonal bipyramid, and octahedral geometry for coordination geometries are commonly seen. A large number of transition elements are primarily responsible for carrying out different bio-functions in living systems like iron in heme proteins, zinc in carbonic anhydrase enzyme in respiration, Fe & Mo in nitrogen fixation, Mn in oxygen evolving complex in photosynthesis, Co in vitamin B_{12} , Cu in hemocyanin and different metallo-enzymes, and so on [6, 7].

3.3. Organometallic compounds

Usually, organometallic compounds are considered as a class of compound which contains at least one M–C bond which is exclusively covalent in character [8]. The metal (M) in these species can either be a main group element or a transition metal. Operationally, the definition of an organometallic compound is also more relaxed to include highly lipophilic complexes such as metal carbonyls, metal nitrosyls, and even metal alkoxides. These compounds have drawn considerable interest mainly as homogeneous and heterogeneous catalysts.

3.4. Cluster compounds

Cluster compounds can be classified as metallic cluster, non-metallic cluster, and metal complex cluster. Clusters can be found in all classes of chemical compounds. According to the

commonly accepted definition, a cluster consists minimally of a triangular set of atoms that are directly bonded to each other, but metal-metal bonded di-/tri-/polymetallic complexes are highly relevant to the area. Clusters occur in “pure” inorganic systems, organometallic chemistry, main group chemistry, and bioinorganic chemistry. The distinction between very large clusters and bulk solids is increasingly blurred. Few examples are $\text{Fe}_3(\text{CO})_{12}$, $\text{B}_{10}\text{H}_{14}$, $[\text{Mo}_6\text{Cl}_{14}]^{2-}$, and $4\text{Fe}-4\text{S}$.

3.5. Bioinorganic compounds

Bioinorganic compounds are one of the most significant classes of a compound in chemical science, which are not only integrally related to the basic processes of nature but also provided significant insights into exploring the chemistry in living world [9]. It is a branch of chemistry, which deals with the interface between chemistry and biology. Starting from DNA to RNA to photosynthesis to oxygen transport and storage to nitrogen fixation, each and every parts of the living world are engrossed with the bioinorganic compounds. Traditional bioinorganic chemistry focuses on electron- and energy-transfer in proteins relevant to respiration. Medicinal inorganic chemistry includes the study of both non-essential and essential elements with applications to diagnosis and therapies.

When bioorganic molecules or drugs are bound to metal ions, there is a drastic change in their biomimetic properties, therapeutic effects, and pharmacological properties. One such remarkable molecule is amino acid, and they form complexes with metal atoms and exhibit significant biological and enzymatic activities. Essential metal ions and their complexes are found to have antitumor, antimicrobial, cytotoxic, and anti-HIV activity [10]. It was, therefore, considered worthwhile to study the complexation and to determine the biological activity of these new complexes.

3.6. Solid state compounds

This important area focuses on structure [11], bonding, and the physical properties [12] of materials. In practice, solid state inorganic chemistry uses techniques such as crystallography to gain an understanding of the structure and structural aspects that result from collective interactions between the subunits of the solid. Metals and their alloys or intermetallic derivatives are included in solid state chemistry. Related fields are condensed matter physics, mineralogy, and materials science. Examples are silicon chips, zeolites, and $\text{YBa}_2\text{Cu}_3\text{O}_7$.

4. Characterization of inorganic compounds

4.1. Solubility test

Solubility is one of the basic parameters that help to understand the properties of a compound. The synthesized compounds are generally tested for their solubility in various polar

solvents like water, ethanol, methanol, dimethyl sulfoxide, and some nonpolar solvents like hexane, ethylacetate, benzene, etc.

4.2. Melting point

Melting point determination for any compound helps to identify the level of reactivity in solid state for any synthesized ligands and metal complexes. It also helps to predict the molecular composition of the complexes.

4.3. CHN analysis

Elemental analysis remains an important method to study the nature of elements (especially C, H, N, and O) exist in compounds. The analysis on the abundance of carbon, nitrogen and hydrogen in a compound also helps to determine the class of compounds in the scientific literature.

5. Structural characterization of metal complexes

Various spectroscopic methods are available in the determination of proposed structures for inorganic complexes. Among these, elemental analyses, FT-IR, UV-Vis, NMR, EPR, mass spectrometry, and thermogravimetric analysis are the most common techniques and widely used globally. But, the most authenticated technique in the determination of detail structural geometry is single crystal X-ray diffraction study. This particular analytical tool helps to locate the atomic position in a 3D structure with an absolute precise manner.

5.1. Electronic spectroscopy

The UV-Vis spectra of all the complexes are recorded in spectrophotometer using different solvents in the wave range of 200–1100 nm.

5.2. Magnetic susceptibility

Magnetic susceptibility measurements of the complexes in the solid state are determined by the Gouy balance at room temperature using metal as the calibrant.

5.3. Molar conductivity

The molar conductivity is measured with a conductivity meter.

5.4. Infrared spectroscopy (IR)

IR spectral analysis is one of the most widely used analytical tools, which is utilized to assign different functional chromophores in the molecule. Incorporation of ligand with metal ions

can be easily detected to draw a simple comparison between IR spectra of free ligand and compound. The principle involved during this spectral analysis is to examine the changes in stretching modes.

5.5. ^1H NMR spectroscopy

Proton nuclear magnetic resonance is probably the best analytical method to determine the geometry and structural backbone for a chemical species. Further, the involvement of the ligand with metal ions in the formation of metal complexes can also be predicted with this technique. The chemical shifts associated with the location of H-atom in the skeletal of ligand remain very important in different applications in material sciences such as proton transfer mechanism and metal organic framework.

5.6. ESR spectroscopy

Electron spin resonance spectral technique is one of the fundamental techniques in detecting the spin state of metal ion or free ligand in inorganic compounds. It actually helps to determine the important aspects of chemical bonding along with significant illumination on structural features for metal complexes. Not only the structural part, but also the generation of ligand centered radical in different organic transformations of laboratory and industrial significance during the investigation of catalytic pathways can also be defined with this particular analytical technique. ESR measurements for the inorganic complexes at different temperatures and in different phases bring additional importance to this technique.

5.7. Thermal analysis

The thermal behavior of the synthesized complexes has been studied to establish different decomposition processes and to confirm the proposed stoichiometry. The thermal behavior of synthesized complexes was characterized on the basis of TGA/DTG and DTA method. Thermal analysis plays an important role in studying the stability, melting point, structure, and decomposition properties of the metal complexes.

5.8. Single crystal X-ray diffraction analysis

The most authenticated way to determine the 3D structure of inorganic complexes is single crystal X-ray diffraction study. It helps to locate the perfect atomic position in a molecule.

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