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Experimental Methodology

2.1 Characterization Techniques

Due to the broad spectrum of elements and the varied properties exhibited by the metal complexes, inorganic chemistry is intricately linked with numerous analytical techniques. A multitude of characterization approaches employs diverse principles to elucidate the chemical composition, crystal structure, composition variation, and photoelectric characteristics of materials. Some spectroscopic methods offer the advantage of studying materials in the solid phase, such as elemental analysis, FTIR, and Single-crystal XRD, etc. while others are suitable for analysis in the solution phase, including UV-vis spectroscopy, Mass spectrometry, and NMR spectroscopy, etc. The characterization techniques used for characterizing various metal complexes are given below.

2.1.1 Single-Crystal X-Ray Diffraction (SC-XRD)

Single crystal X-ray diffraction is a highly effective analytical technique to ascertain crystalline materials' atomic-level three-dimensional structure. In this technique, a single crystal of the material under investigation is bombarded with X-rays, which are diffracted by the crystal lattice. The resulting diffraction pattern provides information about the arrangement of atoms within the crystal. This includes the determination of unit cell dimensions, bond lengths, bond angles, and specific details pertaining to site-ordering. Single crystals of the synthesized metal complexes were grown in appropriate organic solvents by using slow evaporation and vapor diffusion techniques. The Bruker AXS Smart Apex CCD diffractometer was further utilized to load X-ray-appropriate crystals to conduct single-crystal X-ray diffraction measurements. The structures were determined by direct methods utilizing SHELXS-97 software and subsequently refined using full-matrix least-squares on F^2 with SHELXL-97. The hydrogen atoms were incorporated into idealized places and subsequently calibrated based on the riding model. Anisotropic displacement parameters were employed to refine non-hydrogen atoms.

2.1.2 Nuclear Magnetic Resonance (NMR) Spectroscopy

Nuclear magnetic resonance (NMR) spectroscopy, also known as magnetic resonance spectroscopy (MRS), is a spectroscopic method that relies on the reorientation of atomic nuclei possessing non-zero nuclear spins when subjected to an external magnetic field. The phenomenon of re-orientation takes place when electromagnetic radiation is absorbed within the radio frequency range of approximately 4 to 900 MHz. This re-orientation is contingent upon the isotopic composition of the nucleus and is directly proportional to the strength of the external magnetic field. The resonance frequency of each nucleus that is NMR active is influenced by its chemical surroundings. Consequently, NMR spectra offer insights into the specific functional groups within a given sample, as well as the interrelationships among adjacent nuclei within the same molecule. NMR spectroscopy is a crucial technique for identifying chemical structures, especially organic compounds, due to the distinctive and highly characteristic nature of NMR spectra for individual compounds and functional groups. The NMR spectra of the newly synthesized compounds were obtained using deuterated solvents, including $CDCl_3$ and DMSO. The spectrometer used for this ^{1}H, ^{13}C, and ^{119}Sn analysis was the Bruker Ascend 500MHz WB NMR spectrometer, which operated at frequencies of 500, 125, and 186.5 MHz, respectively.

2.1.3 UV-Visible spectroscopy

UV–VIS spectrophotometry is a technique that involves measuring the absorption or reflectance of light in the ultraviolet and visible parts of the electromagnetic spectrum. This methodology is extensively utilized in various applied and basic applications due to its affordability and ease of implementation. UV-Vis spectroscopy is a commonly employed technique in the field of chemistry for the investigation of many compounds, including transition metal complexes containing highly conjugated organic compounds. The absorption spectra of the newly synthesized compounds were measured using the Varian spectrophotometer Cary 4000. The measurements were conducted across a path length of 1 cm, covering the wavelength range of 200-800 nm.

2.1.4 Fourier Transform Infrared (FTIR) Spectroscopy

Fourier-transform infrared spectroscopy (FTIR) is a widely employed method for acquiring an infrared spectrum that represents the absorption or emission characteristics of a solid, liquid, or gas. FTIR spectra exhibit absorption bands that correspond to distinct molecular vibrations. Characteristic absorption bands in specific parts of the spectrum are observed for functional groups such as C-H, O-H, C=O, and N-H, etc. The information regarding the chemical structure and composition of the sample can be derived from the positions and intensities of these bands. The FT-IR spectra of the prepared ligands and the metal complexes were obtained by using the KBr pellets on a Bruker vertex 70V+PMA50 Infrared Fourier vacuum spectrometer.

2.1.5 High-Resolution Mass Spectrometry (HRMS)

High-resolution mass spectrometry (HRMS) is an analytical methodology employed for the determination of the molecular masses of organic and inorganic compounds. HRMS is a widely employed technique across several domains owing to its high level of precision and capability to detect the presence of numerous chemical species, even at exceedingly low concentrations. HRMS analysis initiates with the introduction of a sample into the spectrometer, where it undergoes ionization. This approach has the ability to disassemble the complex into smaller ionic fragments, which can subsequently be analyzed according to their m/z values. The prepared ligands and their metal complexes were analyzed using a 6545 LC/Q-TOF HRMS instrument. The analysis is performed using positive ionization mode in either methanol or acetonitrile solvent to determine the exact masses.

2.1.6 Elemental Analysis

Elemental analysis is used to assess the elemental composition of a compound sample. It encompasses both qualitative and quantitative approaches, with qualitative involving the identification of elements and quantitative involving the determination of their respective quantities. The predominant method employed for elemental analysis, known as CHNS analysis, involves the utilization of combustion analysis. The process of combustion of the compounds in the presence of oxygen results in the generation of CO_2, H_2O, NO_2, and SO_2 gases, which are introduced into a linked GC chamber to determine the precise percentage of elements present. The bulk purity of the prepared metal complexes in the solid state was determined by using element analysis performed on ThermoFisher Scientific FLASH Smart V CHNS/O elemental analyzer.

2.1.7 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis, often known as thermal gravimetric analysis (TGA), is a thermal analysis technique that involves measuring the mass of a sample as the temperature varies over time. TGA can be employed to assess the thermal stability of the material. Thermogravimetric Analyses of the synthesized metal complexes were performed on a TGA-6000 thermal analyzer (Perkin Elmer) in the temperature range of 30 to 700˚C. The thermal stability of the complexes was analyzed under the nitrogen atmosphere.

2.1.8 X-Ray Photoelectron Spectroscopy (XPS)

X-ray photoelectron spectroscopy is a surface-sensitive quantitative spectroscopic technique based on the photoelectric effect. It enables the identification of elements within a material, whether in its elemental composition or on its surface, while also determining their chemical states. Furthermore, XPS provides insights into the electronic structure and density of electronic states across the material. The oxidation state of the metals present in all the freshly synthesized metal complexes was determined using XPS analysis on Thermo Fisher Scientific ECSALAB Xi+ equipment.

2.1.9 Electron Paramagnetic Resonance (EPR) Spectroscopy

Electron paramagnetic resonance (EPR) or electron spin resonance (ESR) spectroscopy is utilized to analyze materials possessing unpaired electrons. The fundamental principles underlying EPR have a resemblance to those of NMR, albeit with the distinction that the excited spins pertain to electrons rather than atomic nuclei. EPR spectroscopy is a valuable technique for investigating metal complexes and organic radicals. The newly synthesized metal complexes were verified for their paramagnetic properties by the utilization of EPR spectroscopy, employing a JEOL FA200ESR spectrometer.

2.1.10 Cyclic Voltammetry (CV) Technique

Cyclic voltammetry (CV) is a widely adopted electrochemical method known for its effectiveness in studying the reduction and oxidation processes of molecular species. In this technique, the current is measured while the potential is systematically varied back and forth, oscillating between chosen limits from positive to negative and vice versa. The data acquired from CV can be utilized to get insights into the electrochemical characteristics of the material. The metal complexes that were newly synthesized were subjected to characterization utilizing the CV technique on a CHI6154E potentiostat. This analysis was conducted in a three-electrode cell consisting of an Ag/AgCl electrode (reference electrode), a glassy carbon electrode (working electrode), and a Pt wire (counter electrode) using tetra-n-butylammonium hexafluorophosphate electrolyte