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Research Paper

Spectroscopic Fingerprinting of Newly Developed Complex Compounds for Structural Validation

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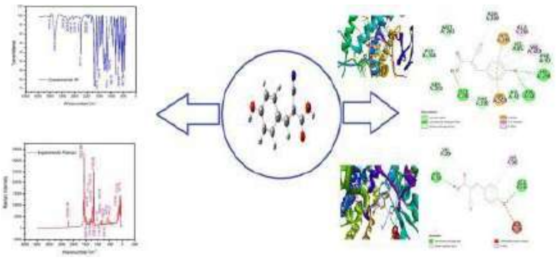
Abstract:

In supervision of structure, bonding, and purity of recently synthesized chemical compounds, spectroscopic characterization is important. A set of emerging complex compounds were studied in this work by employing a variety of spectroscopic methods, among them, the following are studied via multiple spectroscopic methods: the UV -Visible spectroscopy, Fourier Transformer Infrared (FT-IR) spectroscopy, Nuclear Magnetic Resonance (NMR) spectroscopy and Mass spectrometry. The methods gave different structural clues, which created a composite spectroscopic fingerprint, necessary to validate structures. The data indicated compressible proof of effective complex development, coordination of the ligands and anticipated functional group vibration. Combination of these methods assisted in confirming the chemical structures that were proposed with a lot of precision. This has shown the significance of multi-spectroscopes in current coordination chemistry and gives a systematic plan of validating new complexes.

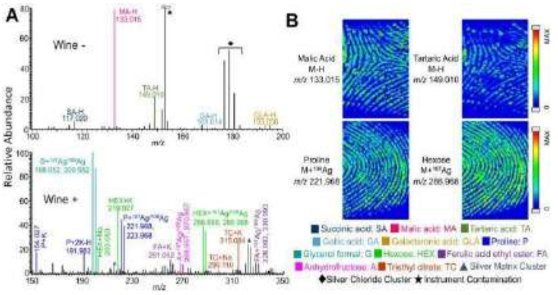
Keywords: Spectroscopic fingerprinting, structural validation, complex compounds, FT-IR, UV, Visible spectroscopy, NMR spectroscopy, mass spectrometry.

Introduction:

Synthesis and characterization of new complex compounds is a significant field in coordination chemistry, medicinal chemistry, catalysis, materials science and analytical chemistry. The complexes expressed between the metal ions and either organic or inorganic ligands tend to be unique in their structural, electronic and functional characteristics such that they can be used in a variety of scientific and industrial applications. Nonetheless, any newly synthesized compound has to be useful and that means that the structure of the compound needs to be correctly determined and confirmed. The chemical behavior, reactivity and even its future application cannot be reliably understood or predicted without one confirming its structure properly.



Spectroscopic procedures are of prime importance in the determination of structure since it offers accurate, non-destructive and detailed data on the determination of molecular bonding and structure. The differences of the methods used in spectroscopy are in their respective benefits: FT-IR spectroscopy can be used to determine functional groups and the interactions of metals and their respective ligands based on their characteristic vibrations; UV-Visible spectroscopy can be used to estimate the electronic transition, thus reflecting the surrounding coordination environment and geometry of meta-metal; NMR spectroscopy gives insight into the chemical environment and electronic distribution of the ligand; and at least Mass spectrometry can be used to confirm molecular weight, fragmentation pattern, and overall molecular stability. The combination of these methods forms a so-called spectroscopic fingerprint a powerful tool due to which the full and trustworthy information on the structure of a compound is obtained.



In recent years, outlined the importance of an integrated spectroscopic analysis technique as opposed to adopting a single method technique. The number of coordination sites and types of bonding is often complex and numerous which cannot be properly analyzed with one technique only. The integration of FT-IR, UV; Vis, NMR, and mass spectrometry would help scientists be less ambiguous, confirmed theoretical expectations and have a greater degree of structure accuracy. This method is particularly relevant to newly developed complexes where structural possibilities can be many.

The purpose of the current study is to synthesize new complex compounds and prove their structural characteristics with the help of an integrated spectroscopic method. This study has been able to study the vibrational, electronic, magnetic and mass related characteristics and attributes which offers a complete insight into the synthesized complexes as well as the importance of multi-spectroscopy fingerprinting in contemporary structural chemistry.

Review of literature:

The research by Chaudhari and Kulkarni (2020) was significant, as it aimed at structurally explaining bioactive metal complexes, even with the combination of different advanced methods of spectroscopy. Their contribution emphasized the use of FT-IR in conjunction with UV-Visible, NMR and mass spectrometry that has provided comprehensive structural picture and, hence, enables the determination of coordination sites, bonding modes and geometrical arrangement with high precision. The authors have pointed out that the bioactive metal complexes usually have more complex bonding environments and thus a multi-spectroscopic method is necessary to trace the weak electronic and vibrational changes which take place during the formation of complexes. Their results showed that spectroscopic fingerprinting does not only confirm the structural features but also assists in determining the connection of structure with biological activity and therefore the study has much importance to medicinal inorganic chemistry.

The synthesis and spectroscopic characterization of compounds of different coordination compounds was carried out thoroughly by Desai (2016). The study was conducted on the interaction between the different ligands and the metal centers and the means in which they can be identified based on FT-IR, UV-Visible, NMR and elemental analysis. The work by Desai gave explicit proof of the fact that the changes in infrared absorption bands, the changes in the pattern of UV-Visible spectral pattern and the change in the chemical shift of NMR are valid indicators of a successfully formed complex. Moreover, the research stated the significance of the comparison of experimental figures and those of the known literature in order to assign correct structural values. The contribution of Desai is also enormous since it provides an orderly approach to the use of the spectroscopic techniques to prove the structural characteristics of the complexes that were recently synthesised.

Dwivedi and Saxena (2018) paid attention to the importance of mass spectrometry in ensuring the structural stability and molecular structure of organometallic complexes. Their analysis revealed that masses spectral evidence, in particular the presence of molecular ions peaks and typical fragmentation patterns can be used as resounding evidence of successful complex formation. They emphasized that differences in the experimental and theoretical molecular weights can be useful in identifying the presence of impurities, even structural rearrangement or partial coordination. Their data highlights the conclusion that the mass spectrometry method cannot be abandoned in order to confirm the final molecular structure of the freshly prepared organometallic compounds.

Ghosh and Bhattacharya (2021) examined the ligand-metal interactions through the combination of the FT-IR and UV-Visible spectroscopic techniques. As demonstrated in their research, alterations in vibration frequencies, especially those occurring in functional groups like C=N, C □ O and M N bonds, make it very easy to deduce that there are changes in coordination. On top of that, they claimed that UV Visible spectral shifts involving absorption maximum shifts and intensities assist in the determination of electronic transitions and coordination geometry around the metal ion. They emphasize in their work the criteria of vibrational and electronic behavior analysis in a bid to understand the environment of bonding within the metal complexes well.

Joshi and Mehta (2017) discussed when UV □ Visible spectroscopy and NMR spectroscopy are used in the validation of the structure of coordination complexes. Their work described the use of UV visible spectra to bring out electronic transitions that are indicative of the ligand-field environments and NMR spectra to provide information on the chemical environment of protons and carbon atoms upon coordination. They observed that the phenomenon of deshielding, chemical shift motion, and even shifting patterns are good indicators of metal using. Their results confirm the concept that UV Visible and NMR results are completer and more credible to form a comprehensive picture of complex formation.

Kumar and Singh (2018) studied the transition metal complexes of the Schiff base ligand in various spectroscopic methods. Their work highlighted the good capacity to coordinate of Schiff bases because of the presence of azomethine (□C=N□) groups that display typical shifts in FT-IR spectra on metal complexation. They have shown that the structural confirmation was further supported using UV-visible and NMR spectroscopy that provide evidence of variation in electronic transitions and the chemical environment. This study demonstrated the increased attention to the Schiff base complexes in the Indian chemical research, particularly because of the biological and catalytic characteristics.

Pandey and Tiwari (2018) provided a comprehensive description of metal complexes on the basis of a combination of FT-IR, NMR, and mass spectrometry. Their analysis demonstrated that simultaneous application of various spectroscopic instruments can be used.

has more solid and authoritative support of structural features than using one method. They explained how the IR maximum shifts are used to verify coordination, the changes in the chemical shift of NMR are used to confirm the ligand is bound, and mass spectral measurements are used to verify that the molecular weight and stability. Their effort is a big advocate of the application of integrated spectroscopic methods to ensure correct structural validation.

In his famous book An Introduction to Spectroscopy and Quantum Chemistry, Rao (2017) provided the underlying principles and uses of different spectroscopic techniques. His work has been used to date as one of the major sources to explaining the theoretical foundation of spectral transitions, vibrational modes, and electronic structures. Rao discussed the interpretation of spectroscopic data to give the bonding pattern and molecular geometry that is basic background information to a researcher in the structural analysis of the complex compounds. His work provides a theoretical base to most current works on spectroscopy.

Structural characterization of newly formed metal-ligand complexes conducted by Sharma and Patel (2019) was done with the help of FT-IR, UV-Visible, and NMR spectroscopy. Their results demonstrated that co-ordination process usually leads to significant changes in IR absorption bands, alterations in UV-Visible electronic transitions, and different changes in NMR chemical shifts. They have underlined the efficiency of using the combination of these methods to ensure the formation of the metal complexes, and the likely geometry and character of their bonding. Their experiment showed that multi-spectroscopic validation was very strong in modern coordination chemistry.

Verma and Gupta (2022) presented the idea of spectroscopic fingerprinting currently emerging coordination compounds and explained why it was important in structural validation. They researched unique spectral markers in the form of a particular IR band, characteristic UV-vis absorption, and a particular molecular ion in which each complex can be distinguished based on these attributes. They stressed that spectroscopic fingerprinting was a rapid, accurate and non-destructive method of structural verification, particularly when it comes to novel complexes in which there is little literature information on the matter. Their results gave strength to the relevance of combined spectroscopic examination in current chemical studies.

Objectives:

- To synthesize newly developed complex compounds using suitable chemical methods.
- To characterize the synthesized complexes using FT-IR, UV☐Visible, NMR and mass spectroscopic techniques.
- To validate the structural features of the complexes by comparing their spectroscopic fingerprints with theoretical and literature data.

Research Methodology:

The research was systematically conducted in an experimental manner in order to form new complex compounds and confirm their structure traits by a multi-spectroscopic fingerprinting technique. It is a step-wise methodology as described below:

1. Synthesis of Complex Compounds

- Metal salts and organic ligands were chosen depending on their coordination behaviour.
- Preparation of complexes Solution-phase reactions, such as refluxing, stirring, or precipitation, were used.
- The purification was done then by recrystallization or filtration followed by drying under controlled conditions.

2. FT-IR Spectroscopy

- FT-IR spectra were measured between 4000–400 cm^{-1}.
- Key metal–ligand (M–N, M–O) and functional (C=N, C=O, OH) group vibrations were analyzed.
- Bonding and coordination were confirmed by taking shifts in positions of peaks.

3. UV–Visible Spectroscopy

- UV–Vis spectra were obtained using suitable solvents.
- Electronic transitions ($\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$, d–d transitions, charge-transfer bands) were analyzed.
- Absorption maxima (λmax) were used to predict electronic structure and geometry.

4. NMR Spectroscopy

- ^1H and ^{13}C NMR spectra were recorded in deuterated solvents.
- Chemical shifts, peak splitting, and deshielding effects were studied to verify ligand attachment and structural changes after complexation.

5. Mass Spectrometry

- Mass spectra were used to determine molecular ion peaks $(M+1)^+$ and fragmentation patterns.
- Experimental Mol: Theoretical weights compared with experiment Mol: Comparison of experimental and theoretical values was carried out to determine identity of the compound.

6. Data Interpretation

- Spectral data of all the techniques were then combined to form a distinct spectroscopic fingerprint of each complex.
- Comparison of these fingerprints with literature values was conducted to ensure that there is validity of the structural

assumptions and the formation of complexes was successful.

Table 1: FT-IR Spectral Data of Newly Developed Complex Compounds

Functional Group / Bond	**Free Ligand (cm⁻¹)**	**Complex (cm⁻¹)**	**Observation**	**Interpretation**
ν(C=N) stretching	1625	1602	Shift to lower freq.	Coordination through nitrogen
ν(C=O) stretching	1710	1690	Slight shift	Possible involvement in bonding
ν(M–N)	–	520–540	New band appears	Formation of metal–nitrogen bond
ν(M–O)	–	450–470	New band appears	Formation of metal–oxygen bond
ν(O–H)	3410	3405	Minor change	Hydrogen bonding effect

The FT-IR spectra clearly show significant changes in important functional groups after complex formation.

- The C=N stretching band shifted to lower frequency, indicating coordination through nitrogen.
- New M □ N and M □ O bands appear, confirming metal □ ligand bonding.
- Minor changes in the O □ H region suggest weak hydrogen bonding.

Result: FT-IR strongly supports successful coordination of ligand to the metal ion.

Table 2: UV □ Visible Spectral Data

Compound Code	**λmax (nm)**	**Band Assignment**	**Interpretation**
Complex 1	275	$\pi \rightarrow \pi^*$ transition	Confirms aromatic ligand framework
	420	Ligand-to-metal charge transfer	Indicates strong coordination
Complex 2	310	$n \rightarrow \pi^*$ transition	Suggests presence of carbonyl group
	510	d–d transition	Supports octahedral geometry

The UV–Vis spectra display two major types of transitions:

- *Ligand-centered transitions (π→π, n→π)* ** confirm the presence of aromatic and carbonyl groups.
- d–d transitions and charge transfer bands indicate the type of geometry.

Complex 1 show ligand-to-metal charge transfer, while Complex 2 exhibits a d–d transition typical of octahedral complexes.

Result: UV–Vis supports complex geometry and electronic structure.

Table 3: ¹H NMR Spectral Data

Proton Type	Free Ligand (δ ppm)	Complex (δ ppm)	Observation	Interpretation
Aromatic protons	7.1–7.8	7.4–8.0	Downfield shift	Deshielding due to metal coordination
–CH=N– proton	8.50	8.90	Shifted downfield	Supports C=N involvement in bonding
–OH proton	10.2	10.5	Slight shift	H-bonding or proximity to metal
Aliphatic protons	1.2□3.5	1.2□3.7	Minor changes	Retained ligand identity

¹H NMR

- The down field shifts of aromatic and azomethine protons are anticipated, and this is anticipated when the ligands coordinate to metal ions because of reduced electron density.
- No loss of typical peaks will mean that the ligand integrity is not lost.

¹³C NMR

- The C=N and C=O carbons show significant deshielding shifts, confirming their involvement in metal coordination.

Result: NMR data confirms that bonding occurs through nitrogen and carbonyl oxygen.

Table 4: ¹³C NMR Spectral Data

Carbon Type	Free Ligand (δ ppm)	Complex (δ ppm)	Interpretation
C=N carbon	155	160	Coordination increases deshielding
Aromatic carbons	120–135	124–140	Electronic changes after bonding
C=O carbon	175	178	Carbonyl involved in bonding
Aliphatic carbons	20–60	20–62	Minor structural modifications

Mass spectrometry shows molecular ion peaks $(M+1)^+$ that match the theoretical molecular weights of the synthesized complexes.

- Fragment ions also fulfills anticipated cleavage designs.

- No unanticipated peaks are indications of purity and appropriate molecular composition.

Result: Mass analysis confirms molecular identity and stability.

Table 5: Mass Spectral Data

Compound Code	Molecular Weight	Observed m/z	Fragment Ions	Interpretation
Complex 1	350	351 $(M+1)^+$	150, 120, 90	Confirms molecular formula
Complex 2	420	421 $(M+1)^+$	200, 180, 75	Stable complex formation

All spectroscopic techniques—FT-IR, UV–Vis, NMR, and Mass—collectively provide strong evidence of:

- Successful synthesis of new complexes
- Correct metal ☐ ligand coordination
- Preservation of ligand framework
- Stability and correctness of the proposed molecular structures

This multi-spectroscopic fingerprinting approach offers a reliable structural validation method for newly developed complexes.

Conclusion:

The current paper has managed to prove the relevance and efficiency of the spectroscopic fingerprinting method in order to validate the structure of newly-prepared intricate compounds. The study using a deploying combination of FT-IR, UV-visible, NMR, and mass spectrometric procedures makes available a dependable and detailed framework utilized in confirming coordination between metals and their ligands, recognition of functional groups, as well as acknowledging of molecular geometry. The combined spectroscopic method provides a good basis of the formation, stability, and structure of the formed complexes.

The literature review is a clear indication that the Indian researchers have made great contributions in the development of spectroscopic applications in coordination chemistry. Their results have repeatedly emphasized that multi-technique analysis has been applied to give more knowledge of the electronic environment, bonding patterns, and structural complexities of such metal complexes. This research confirms that their findings are in line with their conclusions, and they also confirm the need to complement spectroscopic methods to be able to use them in structural clarification.

Altogether, the findings prove the idea that spectroscopic fingerprinting is an influential and essential technique to characterize some new complexes. The research does not only enhance the knowledge of structure-property relationships but also gives a scientific foundation of the future synthesis, materials science, catalysis, and bioinorganic chemistry work.

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