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Solid-State NMR of Chemical Compounds - a Review

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Abstract: Solid-state Nuclear Magnetic Resonance (NMR) spectroscopy has become an invaluable tool for elucidating the structural, dynamic, and compositional properties of chemical compounds across various fields, from organic and inorganic chemistry to materials science. This review summarizes recent advancements in solid-state NMR techniques, including high-field NMR, magic-angle spinning (MAS), and multidimensional approaches, which have significantly enhanced spectral resolution and sensitivity. The review explores applications in studying crystalline and amorphous compounds, probing atomic-level structure, and investigating molecular dynamics critical to catalysts, polymers, pharmaceuticals, and complex hybrid materials. Additionally, it highlights the synergy between solid-state NMR and other characterization methods, such as X-ray diffraction and electron microscopy, which together provide a comprehensive understanding of material properties. Concluding with an outlook on future developments, this review underscores solid-state NMR's growing impact on molecular and materials characterization.

Keywords: Solid-state Nuclear Magnetic Resonance (NMR) spectroscopy; X-ray diffraction

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1. Introduction

Solid-state Nuclear Magnetic Resonance (NMR) spectroscopy has become an essential analytical tool in the study of chemical compounds, offering unique insights into molecular structure, dynamics, and interactions that are often inaccessible through other techniques. Unlike solution-state NMR, which requires compounds to be dissolved, solid-state NMR can analyze materials in their native solid forms, making it particularly valuable for studying a diverse array of substances, including crystalline solids, amorphous compounds, polymers, pharmaceuticals, and hybrid materials. The introduction of high-resolution techniques, such as magic-angle spinning (MAS) and high-field NMR, has dramatically improved the resolution and sensitivity of solid-state NMR, allowing for detailed structural analysis of complex compounds. Through these advances, solid-state NMR has been widely applied in material sciences for characterizing catalysts, drug formulations, and organic semiconductors, where precise structural information is essential for understanding and optimizing functionality. Additionally, recent technological progress in multidimensional and relaxation-assisted NMR methods has enabled the separation of overlapping signals, thereby enhancing the resolution of complex mixtures. This review explores how these innovations have broadened the scope of solid-state NMR applications across chemistry and material science, making it an indispensable tool for the detailed analysis of both organic and inorganic compounds.

2. NMR Techniques

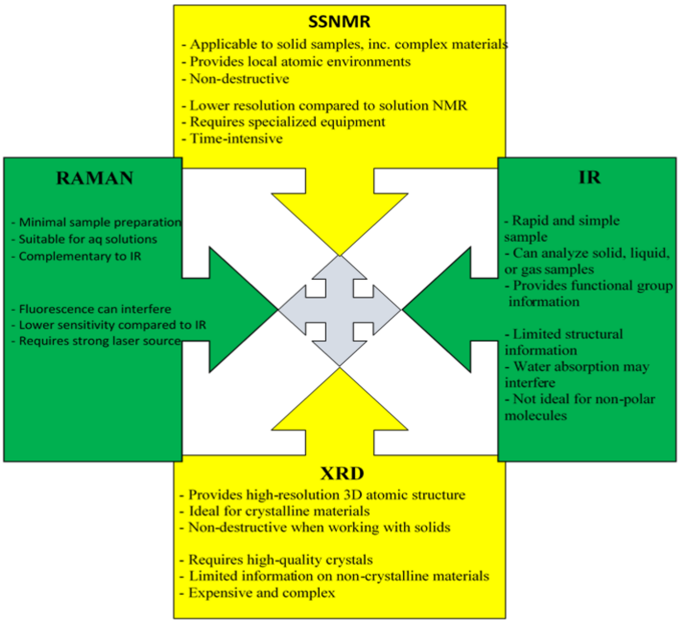
In-Depth Analysis of NMR, Solid-State NMR, IR, Raman, and X-Ray Diffraction Techniques

Nuclear Magnetic Resonance (NMR), Solid-State NMR (SSNMR), Infrared (IR) spectroscopy, Raman spectroscopy, and X-ray diffraction (XRD) are among the most powerful tools in modern analytical chemistry, each providing unique insights into molecular structure, dynamics, and material properties. This paper examines the principles, applications, strengths, and limitations of each technique and compares their capabilities for comprehensive molecular and structural analysis across diverse sample types. By evaluating these techniques in parallel, this paper elucidates how they complement one another in solving complex analytical challenges in chemistry, biology, and materials science.

Structural elucidation and molecular analysis are foundational to advancements in materials science, chemistry, and biology. Each analytical technique offers unique benefits based on the nature of the sample, the level of structural detail required, and the conditions under which the analysis is conducted. Nuclear Magnetic Resonance (NMR) spectroscopy, in both its solution and solid-state forms, provides atomic-level structural information based on the magnetic properties of nuclei. Infrared (IR) and Raman spectroscopy allow for rapid vibrational analysis and functional group identification, while X-ray diffraction (XRD) is fundamental for determining crystalline structures in solid-state materials. This paper reviews the principles and applications of each technique and presents a comparative analysis, highlighting how these techniques are used individually and in concert to analyze molecular structures, crystallinity, and sample properties.

NMR spectroscopy exploits the magnetic properties of nuclei that possess a non-zero nuclear spin, such as ^{1}H, ^{13}C, and ^{15} N. In the presence of an external magnetic field, these nuclei align with or against the field, producing distinct energy levels. When a radiofrequency (RF) pulse is applied, the nuclei absorb energy and transition to higher energy states. As they return to their equilibrium state, they emit RF signals that can be detected and translated into an NMR spectrum. The spectrum provides information about chemical shifts, which reflect the electronic environment around each nucleus, revealing details about molecular structure, bonding, and dynamics. Solution-state NMR is commonly used for characterizing small organic molecules and studying macromolecular structures and dynamics. Advanced techniques, such as 2D NMR (e.g., COSY, HSQC) and NOESY, offer correlation data and spatial information about nuclei within close proximity, which is particularly valuable for complex molecules like proteins. Limitations of NMR include its relatively low sensitivity, which requires large sample concentrations, and the high cost of instrumentation and maintenance due to the need for high-field superconducting magnets and liquid helium cooling [1–10].

SSNMR adapts traditional NMR principles for samples in the solid phase, where molecular motions are restricted, resulting in broadened spectral lines due to dipolar couplings and anisotropic chemical shifts. Techniques such as Magic Angle Spinning (MAS) and Cross Polarization (CP) improve spectral resolution in SSNMR by averaging out these interactions. MAS involves spinning the sample at an angle of 54.74° (the "magic angle") relative to the magnetic field, which reduces line broadening and enhances spectral clarity. Cross polarization transfers polarization from abundant nuclei (such as ^{1}H) to rare nuclei (such as ^{13}C), significantly increasing sensitivity. SSNMR is indispensable for studying complex solid-state materials, including polymers, pharmaceuticals, and ceramics, as well as insoluble biological assemblies like membrane proteins and amyloid fibrils. However, SSNMR requires specialized equipment and extended acquisition times due to the inherently low sensitivity of solid samples. Furthermore, the spectra obtained from SSNMR are often broader and less resolved compared to solution-state NMR, presenting challenges in peak assignment and data interpretation [11–35]. The advantages and disadvantages of four methods described in this review are given in scheme 1.



Scheme 1. The advantages and disadvantages of solid-state NMR, IR, Raman and X-ray methods.

IR Spectroscopy

IR spectroscopy measures the absorption of infrared light by molecular bonds, causing vibrational transitions that are characteristic of functional groups within the molecule. When a molecule absorbs IR radiation at specific frequencies, it excites vibrations such as stretching and bending modes, resulting in an absorption spectrum that serves as a molecular fingerprint. Fourier-transform infrared (FTIR) spectroscopy enhances resolution and acquisition speed, while Attenuated Total Reflectance (ATR) allows for straightforward analysis of solid and aqueous samples without extensive sample preparation. Although IR spectroscopy does not provide atomic-level structural information, it is an efficient and widely used technique for identifying functional groups, analyzing organic and inorganic compounds, and monitoring chemical reactions in real-time. A notable limitation of IR is that it may have difficulty analyzing mixtures with overlapping peaks, and it is less sensitive to non-polar bonds, limiting its applicability for symmetrical or non-polar compounds [36–46].

Raman Spectroscopy

Raman spectroscopy relies on the inelastic scattering of monochromatic light, usually from a laser source. When light interacts with a molecule, most photons scatter elastically (Rayleigh scattering), but a small fraction undergoes inelastic scattering, with a frequency shift that corresponds to molecular vibrational modes. This shift provides a unique vibrational signature of the molecule, similar to IR spectroscopy, but with complementary selection rules: IR is sensitive to polar bonds, while Raman is generally more responsive to non-polar bonds. Enhanced Raman techniques, such as Surface-Enhanced Raman Spectroscopy (SERS) and Resonance Raman, improve sensitivity and allow for trace analysis. Raman spectroscopy is valuable for studying inorganic compounds, nanomaterials, and aqueous biological samples, as water has a weak Raman signal. However, fluorescence can interfere with Raman signals in some samples, and Raman scattering is inherently weak, necessitating longer acquisition times or enhancement techniques. While it is less quantitative than NMR, Raman provides rapid, non-destructive molecular insights that make it suitable for a wide range of applications [47–54].

X-ray Diffraction

X-ray diffraction is a cornerstone technique for determining the crystalline structure of solid materials. When X-rays are directed at a crystal, they diffract in specific directions based on the atomic arrangement within the crystal lattice. By measuring the angles and intensities of these diffracted beams, an electron density map of the crystal can be generated, allowing for the determination of atomic positions. XRD provides detailed structural information at atomic resolution, which is essential for materials science, mineralogy, and the analysis of small-molecule and protein crystals. XRD is particularly effective for highly ordered, crystalline samples; however, it is less effective for amorphous materials, which lack long-range order and therefore do not produce clear diffraction patterns. Single-crystal XRD requires well-formed crystals, while powder XRD is suitable for polycrystalline samples, but the latter generally provides less structural detail [55–66].

NMR and SSNMR provide unparalleled atomic-level structural information, particularly for organic compounds and complex biological molecules in solution or solid state. They excel in determining connectivity, conformation, and dynamic interactions within molecules. However, NMR requires high sample concentrations and is limited by its need for costly, specialized equipment, while SSNMR additionally requires longer acquisition times. IR and Raman spectroscopy, in contrast, offer rapid analysis with minimal sample preparation, excelling in identifying functional groups and molecular vibrations. IR is particularly suitable for analyzing polar bonds, while Raman complements this with sensitivity to non-polar bonds. Both techniques, however, offer limited structural detail compared to NMR and XRD. XRD is the gold standard for analyzing crystal structures, providing atomic-level detail for well-ordered samples. It is crucial for materials science and solid-state chemistry but requires high crystallinity and is less useful for amorphous samples.

3. Results and Discussion

Inorganic and Organic Compounds

The complexation of cyclohexanespiro-5-(2,4-dithiohydantoin), with copper and nickel was studied by means of experimental and theoretical methods [67]. Dimeric structures for the Cu(I) and Ni(II) complexes were proposed in which the ligands were coordinated in N3^S4- and N3^S2-bridging ways, respectively, acting as monoanions. The results demonstrate that the combined experimental (^{13}C CPMAS NMR, IR) and theoretical (DFT) approach can be used to characterize the molecular structure of solid complexes for which crystallographic data are not available. A combined approach of quantum chemical calculations and ^{13}C CPMAS NMR data was used to find the most probable structures of nickel and copper complexes of cycloheptanespiro-5-(2,4-dithiohydantoin and cyclooctanespiro-5-(2,4-dithiohydantoin) [68]. Recently, we describe the synthesis and structure of Pt(II) complexes of two spiro-5-(2,4-dihiohydantoins), namely 3′,4′-dihydro-2*H*,2′*H*,5*H*-spiro[imidazolidine-4,1′-naphthalene]-2,5-dithione and spiro-(fluorene-9,4′-imidazolidine)-2′,5′-dithione [69]. Single-crystal analysis was used for the spiro-(benzocyclohexane-4′-imidazolidine)-2′,5′-dithione. The anticancer activity of all compounds were tested against different tumor cell lines (HL-60, BV-173, and K-562).

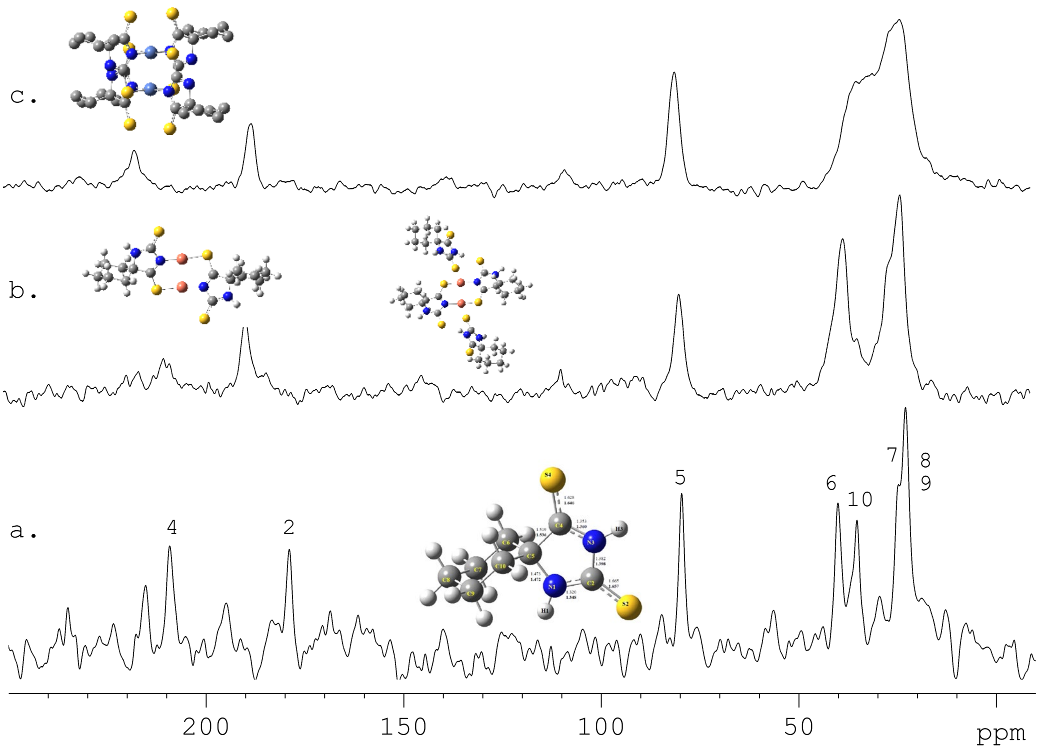


Figure 1. ^{13}C CPMAS NMR spectra of **L** – (a.), **Cu-L** – (b.) and **Ni-L** – (c.) [67].

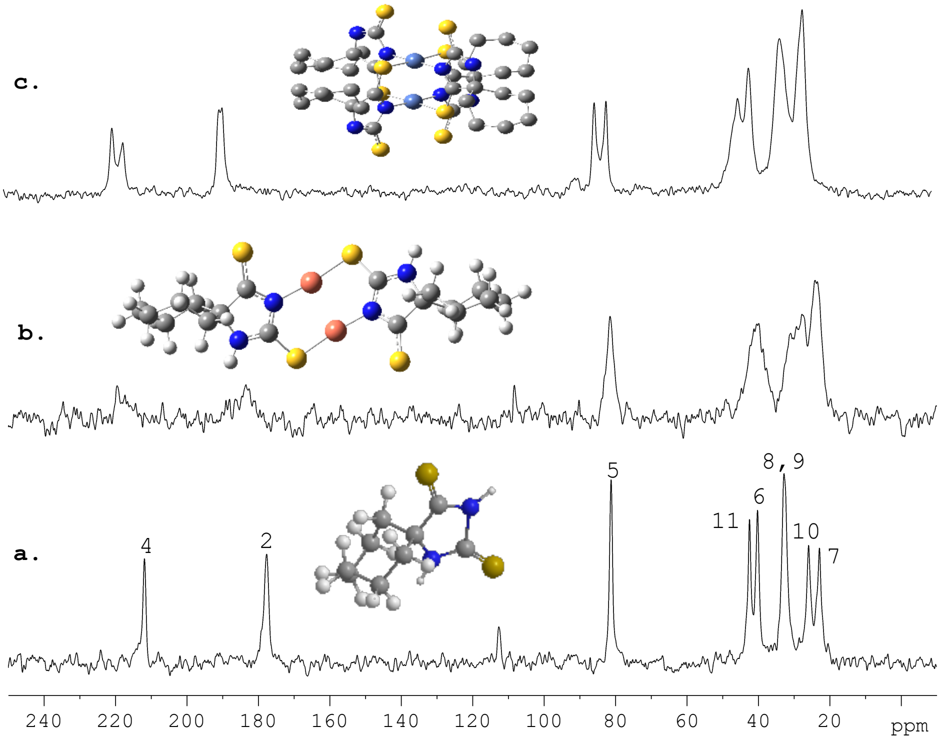


Figure 2. ^{13}C CPMAS NMR spectra of cycloheptanespiro-5-(2,4-dithiohydantoin) – (a), Cu complex – (b) and Ni complex – (c) [68].

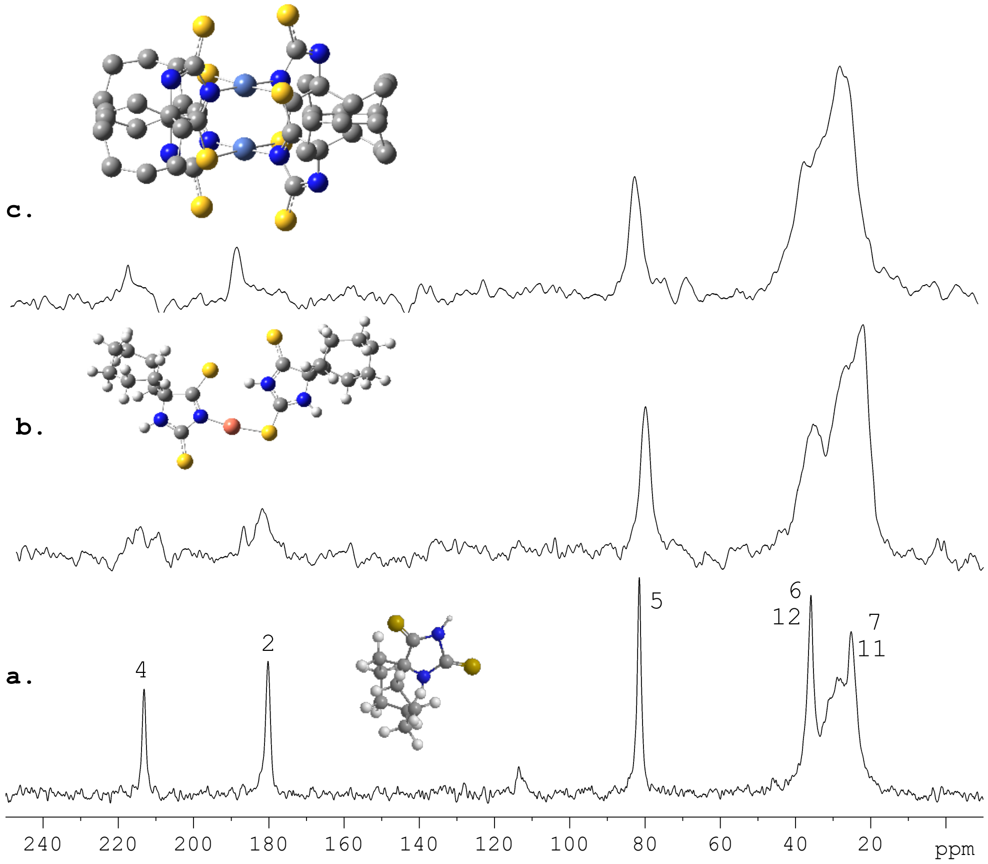


Figure 3. ^{13}C CPMAS NMR spectra of cyclooctanespiro-5-(2,4-dithiohydantoin) – (a), Cu complex – (b) and Ni complex – (c) [68].

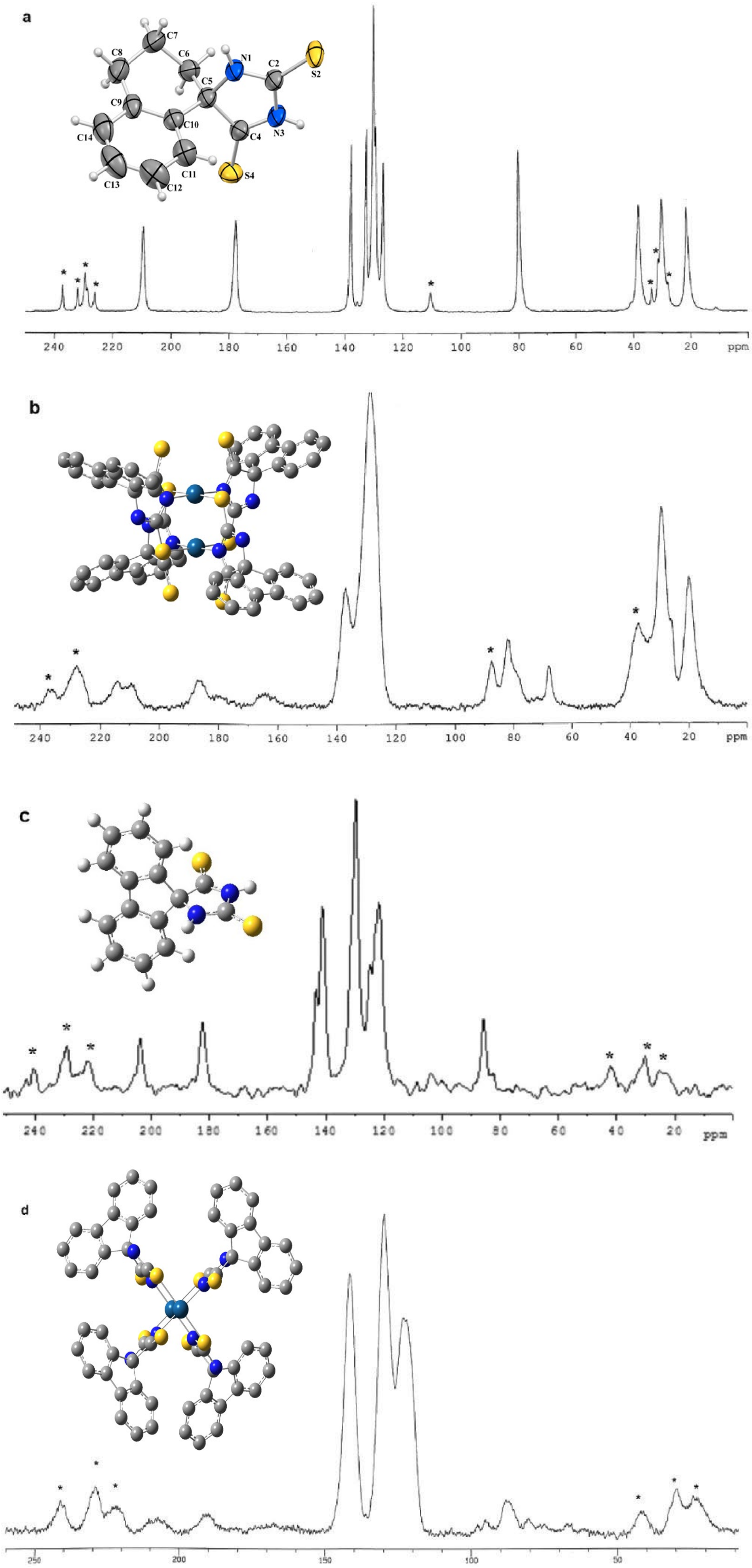


Figure 4. ^{13}C CPMAS NMR spectra of the studied compounds: 3′,4′-dihydro-2H,2′H,5H-spiro[imidazolidine-4,1′-naphthalene]-2,5-dithione (a); Pt complex (b); spiro-(fluorene-9,4′-imidazolidine)-2′,5′-dithione (c); Pt complex (d). Asterisks (*) denote spinning side bands (ssb). [69].

Synthesis and structural characterization of new Cu(II) and Ni(II) complexes of (9′-fluorene)-spiro-5-dithiohydantoin are reported [70]. All compounds were studied by means of experimental (^{13}C CPMAS NMR, EPR, IR) and quantum-chemical (DFT/B3LYP-6-31G(d,p)) methods. Based on the experimental data, supported by theoretical calculation, the most probable structure of the complexes is suggested. The paramagnetic Cu(II) complex has distorted tetrahedral structure with two water and two ligand molecules coordinated to the metal ion. For the Ni(II) complex square planar geometry is suggested with two ligand molecules coordinated in a bidentate fashion.

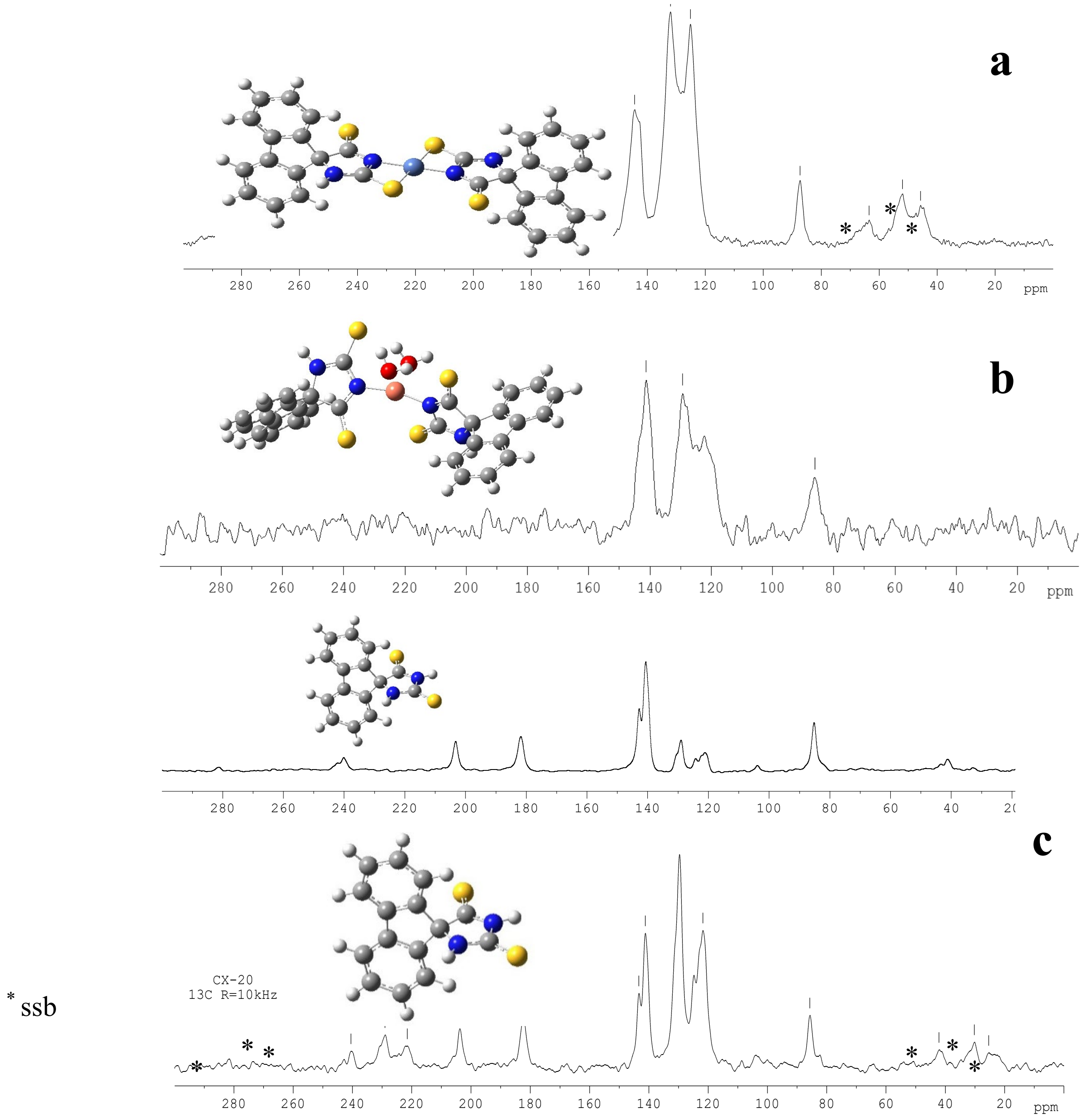


Figure 5. ^{13}C-NMR-CP-MAS spectra of Ni (II) and Cu(II) complexes (**a** and **b**, respectively) of (9'-fluorene)-spiro-5-dithiohydantoin compared with those of the free ligand (**c**). Upper spectrum of (**c**) is recorded with dipolar dephasing pulse sequence (delay time before acquisition of 50 μs) which reduces signals from protonated carbons (at 129.66, 121.75 ppm). [70].

DFT GIAO computations were employed to confirm the experimental ^{13}C CPMAS NMR results and to propose the most suitable model structure for the Al(III) complex [71]. Marinova et al. was obtained new Pd(II) and Cu(II) complexes with 6-methyl-2-thiouracil and 6-propyl-2-thiouracil [72], Au(III) and Cu(II) complexes with 2,4-dithiouracil [73], Au(III) complex with 6-methyl-2-thioxo-2,3-

dihydropyrimidin-4(1*H*)-one [74], Pt(II) complexes with f 5-substituted 2,4-dithiohydantoins [75]. The solid-state NMR spectra of metal complexes [72–74] are given in Figure 6-10.

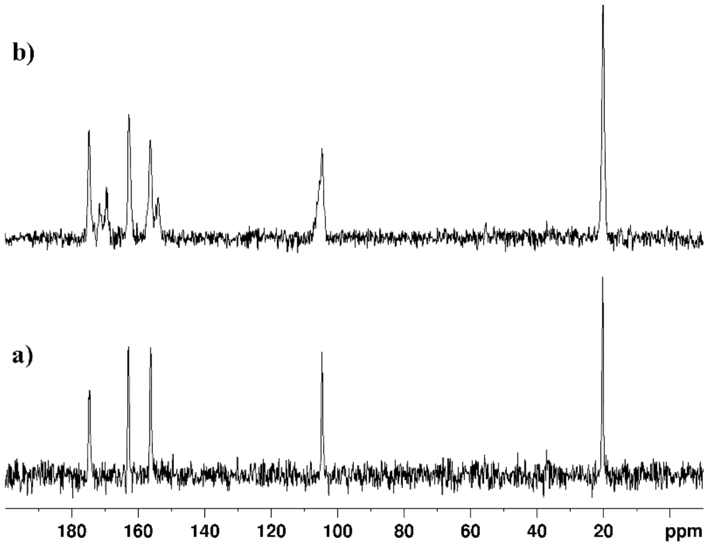


Figure 6. Solid-state NMR of the ligand L1 (a) and its complex with copper (b) [72].

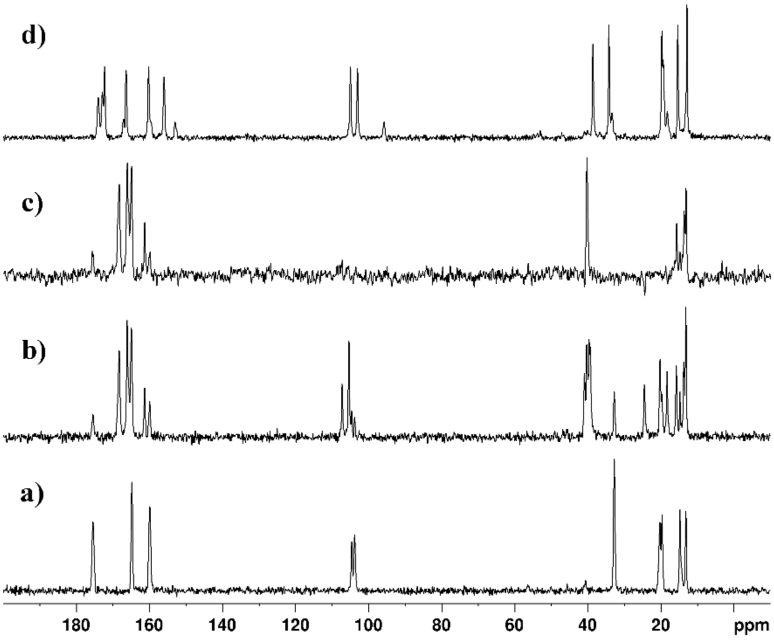


Figure 7. Solid-state NMR of the ligand L2 and its complexes: a) ^{13}C CP spectrum of L2; b) ^{13}C CP spectrum of Cu(II)L2; c) ^{13}C CPPI spectrum of Cu(II)L2; d) ^{13}C CP spectrum of Pd(II)L2 [72].

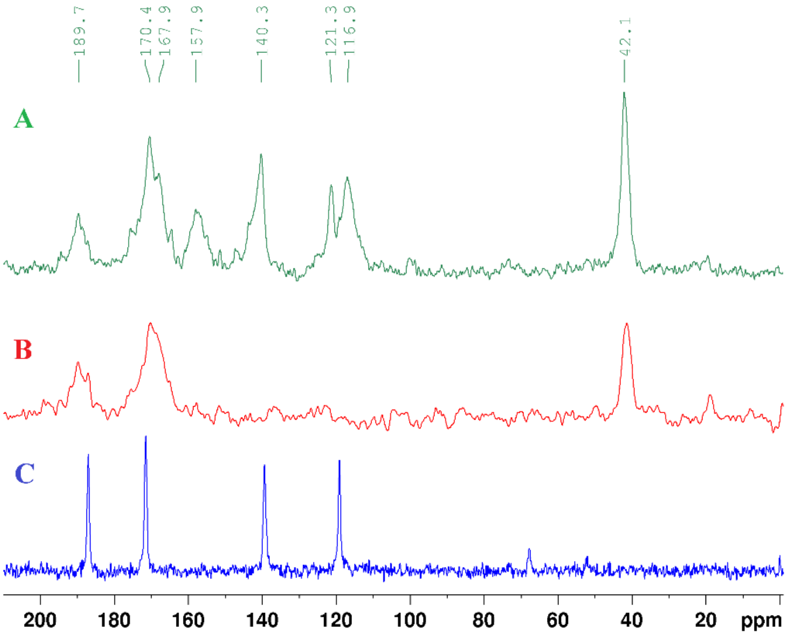


Figure 8. CP MAS NMR spectrum of complex AuL (A); CPPI MAS NMR spectrum of complex AuL (B); CP MAS NMR spectrum of the ligand [73].

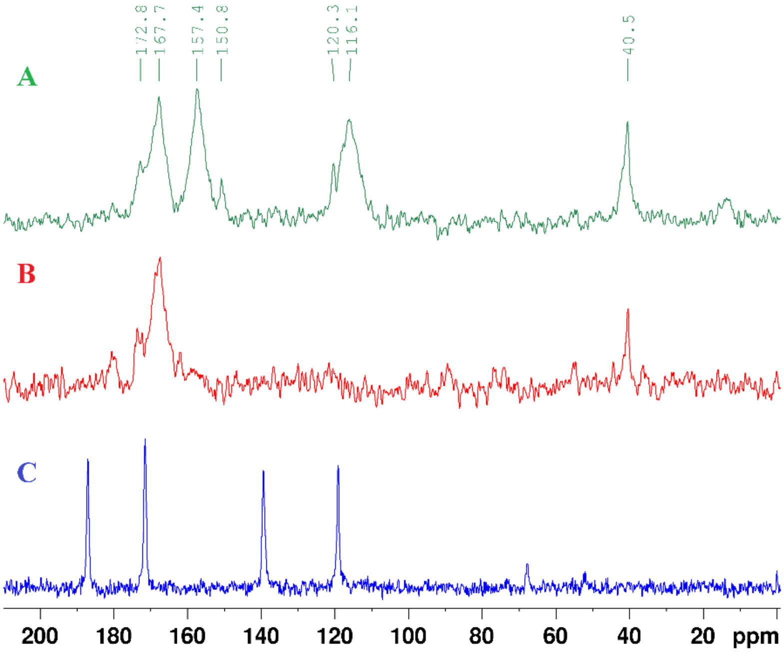


Figure 9. ^{13}C NMR acquired with MAS at 15 kHz. A—CP spectrum of the of complex CuL; B—CPPI spectrum of the complex; C—CP spectrum of the ligand [73].

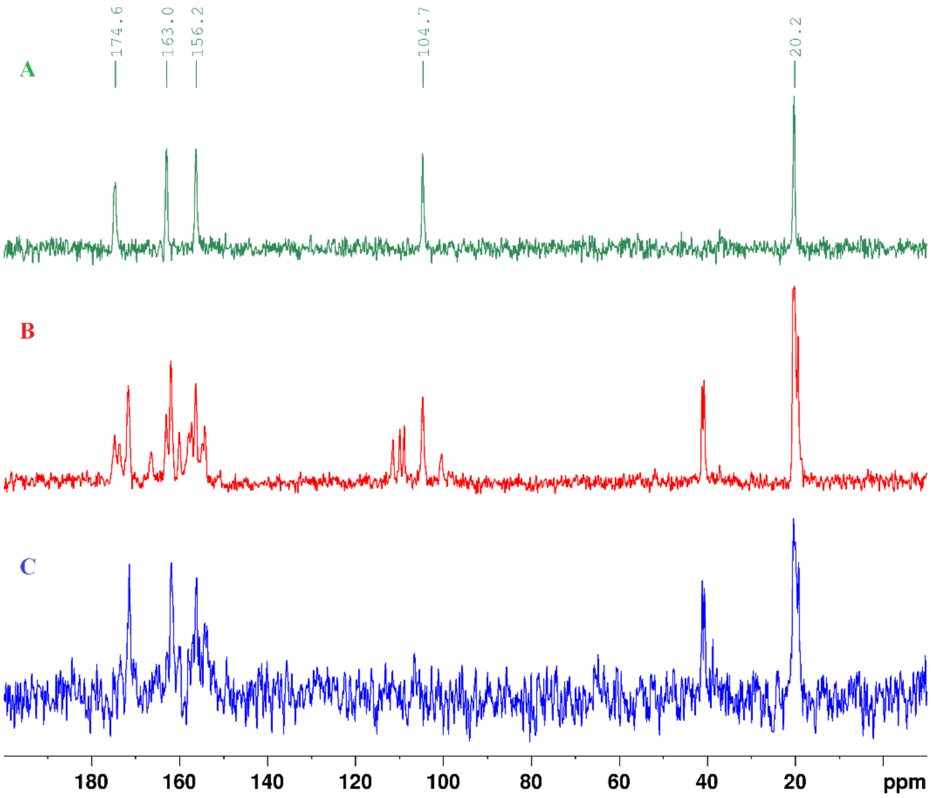


Figure 10. ^{13}C NMR acquired with MAS at 15 kHz. A – CP spectrum of the ligand; B – CP spectrum of the complex; C – CPPI spectrum of the complex [74].

Schiff bases are a widely studied class of organic compounds because of their versatility, ease of synthesis, and remarkable coordination abilities, which make them useful across multiple scientific fields. Named after the German chemist Hugo Schiff, these compounds are generally formed by the condensation of primary amines with carbonyl compounds (usually aldehydes or ketones). The resulting azomethine (-C=N-) functional group is essential to Schiff bases, lending them a unique chemical reactivity that allows for the formation of stable complexes with various metals.

1. Coordination Chemistry and Structural Properties:

Schiff bases are known for their ability to form stable complexes with transition metals, a property leveraged widely in coordination chemistry. Their coordination with metal ions largely depends on the lone electron pair on the azomethine nitrogen, which has been well-documented for its role in stabilizing these complexes [76] New Cu(II) complexes with 2,2′-bis(((E)-2-hydroxybenzylidene)amino)-[1,1′-biphenyl]-4,4′-dicarboxylic acid were synthesized by Bogdanovi'c et al. [77].

2. Applications in Biological Chemistry:

Schiff bases and their metal complexes often exhibit notable biological activities such as antimicrobial and antifungal properties [78]. The presence of the -C=N- group is thought to contribute to these effects due to its interaction with biological macromolecules. The novel dinuclear Ag(I) complex with 2-((E)-(((E)-1-(thiazol-2-yl)ethylidene)hydrazono)methyl)phenol were obtained [79]. The antioxidant, antimicrobial and cytotoxic activities of the free ligand and the complex were discussed also. The new transition metal complexes with pyridoxal-semicarbazone ligand have shown significant biological activity [80]. Recently Sinicropi et al. presents a review witch highlights the latest research on the antimicrobial and cell-growth-inhibiting effects of Schiff base (SBs) metal complexes [81]. Additionally, recent findings on both single-core and dual-core SBs complexes are outlined, covering activities such as antioxidant, antidiabetic, antimalarial, antileishmanial, anti-Alzheimer's, and catecholase functionalities. A study published describes the X-ray structure of nickel(II) complexes coordinated with a Schiff base derived from s-triazine hydrazine. This work discusses the complexes hexa-coordinated geometry and examines its potential antimicrobial properties, offering insights into the coordination behavior and structural stability [82] Another paper by Kargar et al. discusses various Schiff base complexes with metals such as Cu(II) and Zn(II), also characterized by X-ray diffraction. Their findings reveal the octahedral geometry around the Cu(II) ion, highlighting the Schiff base's versatility in forming stable metal-ligand structures. This work also assesses the biological activity of these complexes, making it a valuable reference for both structural and functional insights [83].

New Mo(VI) complexes with (E)-4-amino-N′-(3-ethoxy-2-hydroxybenzylidene)benzohydrazide) was obtained [84]

Additionally, Al-Shboul et al. provide structural analyses using X-ray crystallography for complexes with metals like iron, copper and zinc coordinated to Schiff bases. This research examines both structure and bioactivity, demonstrating the broad utility of Schiff bases in synthesizing diverse metal complexes with unique geometries [85]

3. Optical Properties and Photoluminescence:

Schiff base-metal complexes are promising in the field of optoelectronics, particularly due to their luminescent properties. Their ability to emit light makes them suitable for applications like LEDs and optical sensors [86]. The new Ru(II) complex with dioxygen ligand 3,3-(DPEphos)-3-(η_2-O_2)-*closo*-3,1,2-$RuC_2B_9H_{11}$ was obtained [87]. The electrochemical studies have shown reversible Ru(II)-Ru(III) transition making the complexes suitable for application in catalysis of polymerization. The p-type doping of poly[NiSchiff] materials forms the foundation for most of their applications in electrochemical cells [88]. Electrodes modified with this polymer experience cycles of charging and discharging, during which charge-compensating anions, and sometimes solvent molecules, move in and out of the polymer network to preserve its electrical neutrality. Zinc complexes were studied experimentally and theoretically in view of their possible application as emitters in organic light emitting diodes (OLEDs) [89]. Summary data on the structure of the complexes and the donor atoms involved in the coordination are given in **Table 1**.

Table 1. Summary data on the structure of the complexes and the donor atoms involved in the coordination.

technique	donor atom	metal	structure	references
^{13}C CPMAS NMR, IR and FAB-MS and theoretical DFT studies	N3^S4-bridging coordination	Cu(I) and Ni(II)	dimeric structures	[67]
^{13}C CPMAS NMR and theoretical DFT studies	N3^S2-bridging coordination for L1 with Cu(I); monodentate coordination (N3- and S2-) of two non-equivalent ligand molecules for L2 with Cu(I); N3^S4- bridging way for Ni(II)	Cu(I) and Ni(II)	dimeric structure for Cu(I) with L1; square planar for Ni(II) with L1 and L2	[68]
IR and ^{13}C CPMAS NMR	N and S	Pt(II)	square planar	[69]

and theoretical DFT studies				
^{13}C-NMR-CP-MAS, EPR, IR and quantum-chemical (DFT/B3LYP-6-31G (d,p)) methods	N for Cu(II) and N3 and S2 for Ni(II)	Cu(II) and Ni(II)	distorted tetrahedral for Cu(II) and square planar for Ni(II)	[70]
^{13}C CPMAS NMR and theoretical DFT studies, X-ray	O, Cl	Al(III)	six-membered chelate rings	[71]
melting point analysis, MP-AES for Cu and Pd, UV-Vis, IR, ATR, ^{1}H NMR, ^{13}C NMR and Raman, Solid-state NMR spectroscopy	O,S for L1 and S for L2 with Cu(II); N, S, O with Pd(II)	Cu(II) and Pd(II)	tetrahedral for Cu(II) with L1 and octahedral for L2; chelate for Pd(II) with L1 and L2	[72]
MP-AES for Cu and Au, ICP-OES for S, ATR, solution and solid-state NMR, and Raman spectroscopy	N,S for Au(III) and O,S for Cu(II)	Au(III) and Cu(II)	chelate structure	[73]
UV-Vis, IR, ATR, ^{1}H NMR, HSQC, and Raman, solid-state NMR spectroscopy	O, S	Au(III)	tetrahedral	[74]
IR, FAB-MS, XPS, solid-state NMR spectroscopy and theoretical DFT studies	N, S	Pt(II)	dimer, chelate structure	[75]
X-ray	neutral tridentate NNN-chelate	Ni(II)	distorted octahedral geometry	[82]
X-ray	bis-N,O-bidentate Schiff base ligands	Cu(II)	distorted tetrahedral geometry	[83]

X-ray and FT-IR, ^{1}H NMR, ^{13}C NMR, and elemental analysis	tridentate ONO-donor Schiff base ligand	Mo(VI)	distorted octahedral coordination geometry	[84]
X-ray and ^{1}H-, ^{13}C-NMR, IR and UV-Vis spectroscopy and elemental analysis and theoretical DFT studies	O, N	Cu(II), Fe(II) and Zn(II)	chelate structure	[85]
X-ray	O, N	Ag(I)	dinuclear complex, chelate structure	[79]
X-ray, ESR, MALDI mass-spectrometry, NMR spectroscopy	P, O, P	Ru(II) and Ru(III)	chelate structure	[87]
X-ray crystallographic analysis, FTIR, EPR and UV-VIS spectroscopy theoretical DFT studies	O, N	Ni(II)	distorted octahedral coordination geometry	[80]
electrochemical quartz crystal microgravimetry (EQCM) coupled with cyclic voltammetry (CV).	O, N	Ni(II)	tetrahedral geometry, polymer	[88]
SC-XRD, IR, ^{1}H-, ^{13}C-NMR, TG–MS, X-ray for free ligand	O, N	Cu(II)	coordination number of Cu(II) is five, polymeric chains	[77]
elemental analysis, IR spectroscopy, laser desorption/ionization mass spectrometry (LDI MS), and X-ray diffraction,	O,N	Zn(II)	binuclear complex	[89]

theoretical analysis of electronic absorption spectra by the quantum-chemical TD DFT method		

4. Solar Cells and Photovoltaic Applications:

In renewable energy, Schiff bases have shown potential for photovoltaic applications due to their tunable electronic properties, enhancing light absorption and electron transfer. Recent studies highlight their use in dye-sensitized solar cells, where Schiff bases serve as dyes or sensitizers [90,91].

5. Future Directions and Structure-Property Relationship:

The design of new materials with improved electronic and photonic properties involves a deep understanding of the relationship between structure and function in Schiff base complexes [92].

These references provide a foundation for understanding the chemistry, properties, and wide-ranging applications of Schiff bases and should aid in further exploration of this field. In summary, Schiff bases are an attractive subject in coordination chemistry due to their versatility, multifaceted applications, and potential to advance various technological fields, especially in renewable energy and photonics.

One paper focuses on the analysis of solid-state NMR for catalysts and surface species, particularly leveraging high magnetic field strengths (28.2 T) and fast magic angle spinning (MAS) for improved resolution [93]. This approach is especially useful in studying surfaces such as silica-supported catalysts and alumina. It highlights the benefits of fast-MAS in resolving complex spectral details, which can improve understanding of active sites on catalyst surfaces. Another study examines phase evolution in perovskite materials using solid-state NMR, in combination with powder X-ray diffraction (XRD) and calorimetry [94]. This research explores mechanochemically synthesized dual-cation perovskites and provides insights into cation distribution and crystal phase transitions, which are critical for materials used in electronic and energy applications. A study presented a 3D relaxation-assisted separation technique to improve the resolution of solid-state NMR patterns, especially useful for overlapping signals in complex organic and biological compounds. This technique enhances signal processing through principal component analysis, allowing for better structural insights in compounds like polymers and pharmaceutical materials [95].

Cano et al. used ^{13}C CP/MAS-NMR spectra of organic matter as influenced by climate, vegetation, and soil characteristics in soils from Murcia, Spain [96]. Solid-state cross-Polarization Magic Angle Spinning Carbon-13 Nuclear Magnetic Resonance used to investigate the structure and interactions of cellulose I by Larsson et al. [97]. Solid-state carbon-13 cross-polarization magic-angle spinning nuclear magnetic resonance spectroscopy is commonly used to study starches from plants such as potato, maize and wheat. However, its application to rice starch has been limited. In this study, was combined ^{13}C CP/MAS NMR with deconvolution and subtraction techniques to analyze various rice varieties, including mutants that lack one or more enzymes involved in the biosynthesis of amylose and/or amylopectin. [98]. A series of 2-phenylimidazolecarbaldehydes [99] and 5-spiro-2,4-dithiohydantoins [100] were synthesized and investigated by detailed in the solid-state NMR and ^{1}H and ^{13}C NMR in solution, as well as DFT calculations also used. The summary data of organic compound are given in Table 2.

Table 2. Summary data on the organic compounds.

technique	**compounds**	**reference**
powder XRD, solid state NMR and calorimetric study	$(Cs_x(CH_3NH_3)1\text{-}x)PbX_3$	[94]

^{13}C CP/MAS-NMR spectra	organic matter	[96]
CP/MAS ^{13}C-NMR spectroscopy	cellulose I	[97]
^{13}C CP/MAS NMR	Rice Starch	[98]
^{13}C CPMAS NMR, Cross-polarization/polarization-inversion (CPPI), ^{1}H-^{13}C HETCOR MAS NMR spectra	(2-Phenyl-1H-imidazol-4(5)-yl)methanol; (2-(4-Methoxyphenyl)-1H-imidazol-4(5)-yl)methanol; -(4-(hydroxymethyl)-1H-imidazol-2-yl)benzonitrile; 2-Phenyl-1H-imidazole-4(5)-carbaldehyde; 2-(4-methoxyphenyl)-1H-imidazole-4(5)-carbaldehyde; 4-(4-formyl-1H-imidazol-2-yl)benzonitrile; 2-(4-hydroxyphenyl)-1H-imidazole-4(5)-carbaldehyde	[99]
^{13}C CPMAS NMR and theoretical DFT studies	cyclopentanespiro-5-(2,4-dithiohydantoin); cyclohexanespiro-5-(2,4-dithiohydantoin); cycloheptanespiro-5-(2,4-dithiohydantoin); cyclooctanespiro-5-(2,4-dithiohydantoin); 9’-fluorenespiro-5-(2,4-dithiohydantoin)	[100]

4. Conclusions

A review on solid-state NMR of chemical compounds would likely conclude by summarizing its essential contributions and advancements across chemistry and materials science. Here’s an outline of potential conclusions:

1. **Versatile Tool for Structural Analysis**: Solid-state NMR has emerged as a critical tool for studying the atomic-level structure of organic, inorganic, and hybrid materials. Its capability to provide information on both ordered and disordered phases allows scientists to probe complex structures, including crystalline lattices and amorphous regions that are often inaccessible by other techniques like X-ray diffraction.
2. **Enhanced Understanding of Molecular Dynamics**: Solid-state NMR enables the analysis of molecular motions and interactions within materials. This is particularly valuable for materials with specific applications, such as catalysts, pharmaceuticals, and organic semiconductors, where the dynamics impact material stability and functionality.
3. **Technological Improvements**: Advances like fast magic-angle spinning (MAS), high-field magnets, and multidimensional NMR techniques (such as 2D and 3D relaxation-assisted separation) have significantly improved the resolution and sensitivity of solid-state NMR. These advancements allow for detailed analysis of complex mixtures and overlapping signals, opening up new possibilities in fields like drug development and polymer science.
4. **Complementary Role with Other Analytical Techniques**: Solid-state NMR complements other methods, such as X-ray scattering and electron microscopy, providing insights at different structural levels. Together, these methods form a comprehensive toolkit for characterizing both the chemical and physical properties of materials.

Overall, the conclusions emphasize the value of solid-state NMR as an irreplaceable tool in modern chemical research, providing critical insights into the structure, dynamics, and properties of a vast range of chemical compounds.

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