Solid-State Nuclear Magnetic Resonance

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Review Contents

SCOPE

This review focuses on developments and applications in the field of nuclear magnetic resonance of solids appearing in the literature between October 1999 and October 2001. The review emphasizes uses of solid-state NMR spectroscopy to answer questions about problems in certain areas of chemistry and materials science. As time passes, the use of solid-state techniques to address problems in a wide variety of fields appears more frequently, so producing a review that spans solid-state NMR is difficult. Choosing topics to emphasize and what not to include necessarily reflects the authors' bias. We do not try to review areas exhaustively. In particular, we may refer to one or a few articles on a system as representative of a particular problem addressed with NMR spectroscopy, and other similar work may be found in the literature. We hope to point the interested reader to areas of research, rather than enumerate all articles on a particular substance or area of endeavor. Further literature research will be necessary to delve into a particular area or into uses of NMR on a particular system.

REVIEWS

Reviews in ongoing series are an important source of information on the NMR spectroscopy of solids. We urge readers to seek information in long-standing review series such as the *Specialist Periodical Reports, Progress in NMR Spectroscopy, NMR: Basic Principles and Progress, Magnetic Resonance Reviews, Advances in Magnetic Resonance,* and *Chemical Reviews.* In addition, from time to time, reviews in journals that specialize in topics such as polymer chemistry or ionic conductors publish reviews on uses of NMR spectroscopy, which are helpful to the reader interested in those specific fields. We have found the judicious use of Internet searches helpful in discovering work in various laboratories around the world.

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SOLIDS

"Solid" is a word representing many different things. In the paragraphs below, we describe NMR experiments on certain kinds of solids such as zeolites or polymers because studies of these materials are manifold. In this section, we describe examples of experiments on specific solid materials that do not fit into the chosen categories but that are worthy of mention.

Although the common independent variable in NMR measurements is temperature (*1, 2*), the NMR parameters can, and do, depend on pressure. The pressure variation of frequency shifts and relaxation times of trichloro- and dichloroanisoles has been reported and interpreted to indicate that relaxation in these compounds is mainly due to torsion motions (*3*).

Exciton dynamics in incommensurate phases of $RbZnBr_4$ and $RbZnCl_4$ have been investigated with [87]Rb NMR to determine critical exponents and the universality class (*4*). Phase transitions in $LiRbSO_4$ have been monitored with [87]Rb NMR (*5*) and in BH_3-NH_3 with proton and deuteron NMR (*6*). Rubidium NMR was also used to study dynamics in the $2Ca(NO_3)_2$–$3RbNO_3$ system (*7*). Lithium NMR was used to monitor transitions to magnetically ordered states in lithium zinc vanadates and lithium zinc vanadium titanates (*8*). Order and disorder in 1,2-dichloro-3,4,5,6-tetramethylbenzene have been investigated with deuterium NMR (*9*). The dynamics in a tetrahydrofuran clathrate have been measured with NMR spectroscopy (*10*).

The effects of proton tunneling can be seen in NMR parameters. Examples include the four protons in calix4arene (*11*) and the deuterons in a *trans*-$(Ru(D_2)Cl(PPh_2CH_2CH_2PPh_2)_2)PF_6$ complex (*12*).

The first report of determination of a [73]Ge relaxation time has appeared (*13*). The materials investigated were organogermanes and the metal. Fluorinated graphite has been investigated with relaxation methods, with the conclusion that paramagnetic dangling bonds behave like isolated spins (*14*). Relaxation in complexes of the structure $A_x(Pt_2I(P_2O_5H_2)_4)\cdot nH_2O$, where A is Cs, Rb, Na, or Li, is interpreted in terms of spin jumps (*15*). Changes in the ^{13}C relaxation of C_{60} at the phase transition at 262 K have been followed in an attempt to determine the changes that happen at the transition (*16*). Relaxation of [45]Sc in scandium hydride (deuteride) showed deviations from the Korringa relation, which were dependent on whether the hydride or the deuteride was being studied (*17*).

ZEOLITES

Zeolites have been actively studied with solid-state NMR spectroscopy for many years. Zeolites are not only important materials from the structural standpoint, with new structures being reported, they are also important as catalysts in a wide variety of

processes. Thus, one may divide the uses of NMR in zeolites into roughly two categories: (1) studies of zeolite structure and changes of it with changes in environmental parameters and (2) studies of materials sorbed in zeolites.

Structural characterization with NMR continues to be important in zeolite analysis. For example, a SAPO-37 structure was analyzed with ^{29}Si NMR to demonstrate that phase segregation occurred upon dehydration [18]. Dehydration of offretite could easily be followed by similar NMR techniques [19]. An important contribution is the demonstration that, at sufficiently high field, ^{27}Al NMR detects all the aluminum in a zeolite material [20]. Reactions of Y zeolite with fluorine or fluoride solutions cause degradation of the material that can be detected with NMR spectroscopy [21, 22]. Studies with NMR allowed one group to discern the position of the fluoride ion in a siliceous material including tetrapropylammonium fluoride [23]. Attempts to use NMR to characterize the structure of Y zeolite exchanged with rare-earth materials failed to yield a signal, with the exception of materials exchanged with samarium [24]. However, in a phosphate-based catalyst, all of the phosphorus could be detected by a spin−echo mapping technique, even in the presence of paramagnetic materials [25]. In merlinoite, two distinct tetrahedral sites were detected with ^{29}Si and ^{27}Al NMR [26]. ^{11}B NMR has been used to probe interactions of boron in pentasil zeolites upon hydration [27]. The siting of ions in chabazite has been addressed with NMR spectroscopy of the ions [28]. Rubidium ions in exchanged zeolites are easily detected with NMR [29]. Similarly, ^{113}Cd and ^{207}Pb NMR have been used to probe the siting of these ions in zeolites and the effects of treatment on this siting [30]. Other alkali ions such as cesium [31] and lithium [32] are also amenable to study with NMR.

The investigation of materials in zeolites is as diverse as the possible sorbed materials are. Many of the studies focus on the state of sorbed reactants. Since these are often organic phases, one investigates them with ^{13}C NMR as well as ^{29}Si or ^{27}Al NMR. Molecules as simple as chlorform and trifluoromethane have been sorbed into zeolites, the NMR spectrum has been determined to specify structural features of the zeolite [33, 34], and materials as subtle as chemical warfare agents in contact with surfaces have been analyzed with NMR spectroscopy [35]. The analysis of materials such as acetone oxime provides information on the state of materials that are considered intermediates in reaction mechanisms [36]. In some cases, materials that are detectors of state, such as acidity, are measured with NMR. For example, in a study of the alkylation of phenol, the authors detected the ^{31}P NMR signal of trimethylphosphine oxide to determine acidity [37]. Phosphorus NMR of trimethylphosphine in HY zeolite shows that it exists as an ion pair by reaction at the Brønsted site [38]. The nitration of toluene over zeolite catalysts showed that the active species was acetyl nitrate [39]. The NMR studies of water and CO over ZSM-5 produced a formate species that disappeared when an alkane was cosorbed [40]. The interaction of CO with isobutane over ZSM-5 shows that D−H exchange is inhibited by the presence of CO [41]. Carbocations created in zeolitic materials are detectable with NMR spectroscopy [42]. The reactions of chlorofluorocarbons over zeolites were monitored with both ^{13}C and ^{19}F NMR spectroscopy [43], and the state of various hydrochlorofluorocarbons in zeolites has been probed with NMR [44].

One active area is photocatalysis, and NMR spectroscopy is ideally suited as a detector [45]. For example, NMR detection during the photochemical oxidation of cyclohexane over zeolite Y has been reported [46]. One difficulty of using NMR spectroscopy to monitor reactions is the fact that reactions must often be done in batch processes because of the needs of the spectroscopic experiment. However, a recent report [47] shows that MAS NMR may be done in flow, with the effluent being analyzed by gas chromatography.

Aside from monitoring the existence of particular phases, NMR is useful in delineating the dynamics in these constrained environments, and that is the focus of a number of reports. For example, temperature-dependent broadening of the NMR spectroscopy of p-nitroaniline in the pentasil ZSM-5 is indicative of mobility of the ring [48]. Similarly, studies of the hydrogen-bonded acetonitrile showed free rotation of the methyl group and librations of the bound material [49]. The molecular dynamics of benzene in a Ag-zeolite were probed with temperature-dependent ^{13}C and ^{2}H NMR [50]. The dynamics of isobutyl alcohol in H-ZSM-5 were studied with deuterium NMR spectroscopy of selectively enriched materials [51]. An example of the use of deuterium NMR for a material other than a zeolite comes as a study of the mobility of water in clays [52].

SOLID IONIC CONDUCTORS

An interesting area to which NMR spectroscopy is applied is the area of solid ionic conductors. Given the sensitivity of the NMR spectrum to dynamics, study with NMR spectroscopy gives a great deal of information on the nature of ion movement in these materials. For example, the measurement of diffusion by the pulsed-gradient method allows a determination of mobility of moieties such as the imidazolium cation in imidazolilum salts [53], of lithium salts in polymer matrixes [54, 55], or of conductive species in sodium hydroxide [56].

In some cases, the line shapes of nuclei in the matrix or in the conducting moiety can be analyzed to provide information on mobility. Thus, in an NMR study of N,N-dimethylpyrrolidinium hydroxide, the deuteron line width indicated liquidlike mobility [57]. In the study of lithium-doped poly(propylene glycol), both ^{13}C and ^{7}Li NMR gave information about the dynamic state [58]. Similar uses of lithium NMR were made in a study of the lithium incorporated in a polyether−polyurethane material [59] and in poly(ethylene glycol)−silica infused with LiClO$_4$ [60]. NMR may, in some instances, indicate multiple sites for ions [61]. In some cases, the lack of NMR signal is an indication of strong interactions that suppress the NMR signal, as in the loss of a lithium signal [62] or the failure to detect a ^{127}I signal [53].

An interesting use of NMR is a demonstration of correlation of motion through a heteronuclear NMR experiment between ^{107}Ag and ^{109}Ag in the "superionic" conductor Ag$_9$GaSe$_6$ [63]. In the case of imidazole, use of a two-dimensional exchange experiment between ^{15}N sites shows that conduction there does not involve a flip of the imidazole ring [6]. In yet another interesting use of NMR techniques, the time dependence of the lithium spin-alignment order was used to monitor slow translational modes in Li$_3$Sc$_2$(PO$_4$)$_3$ [65].

In some cases, the measurement of relaxation times gives information on the dynamic processes in these kinds of systems.

For example, T_1 of ^{31}P was measured over a range up to 1300 °C in a Mg^{2+} conductor, which gives a measure of the fluctuating field at the phosphorus site in these complex systems (*66*). The relaxation times of lithium poly(ethylene oxide)−salt preparations with or without added alumina are dominated by quadrupolar interactions that are modified by the addition of these fillers (*67, 68*). Lithium T_1s in lithium-doped poly(ethylene oxide−propylene oxide) also exhibit sensitivity to the ion hopping (*55*). T_1 measurements of the sodium resonance in sodium orthophosphate and solids solutions of it with added sulfate are interpreted in terms of correlated anion reorientation and cation diffusion (*69*). Relaxation of ^{63}Cu and ^{127}I centers in ionic and superionic conductors appears to be due to thermally generated defects, whose energy of activation for diffusion and enthalpy of formation were determined from the NMR properties (*70*).

From these examples and from others found in the literature, NMR spectroscopy is easily seen to be able to indicate the types of sites for ions, the dynamics of ions, and the cooperativity of motion that may occur in these ionic conducting systems. In addition, NMR spectroscopy readily allows comparison of materials of different composition or materials made in different manners.

SEMICONDUCTING MATERIALS

Semiconducting materials have been studied by a variety of NMR techniques, frequently focusing on metal−semiconducting transitions, as monitored by nuclei at particular points in the material. For example, a variety of materials have been examined with silicon, cadmium, tin, and phosphorus NMR of these spin-$^1/_2$ nuclei (*71*).

The effects of coupling to electrons on spin−lattice relaxation provide a means of measuring interactions of nuclei with the electrons in materials such as CdF_2 doped with Ga (*72*), NaV_6O_{11} (*73*), Fe_2VGa (*74*), $Fe_{2+x}V_{1-x}Al$ (*75*), CsRhSb and CsNiSn (*76*), ϵ-FeSi (*77*), $BaNbS_{2.96}$ and $BaNbS_{3.03}$ (*78*), YB_{12} (*7*), $CdCr_2Se_4$ (*80*), CdTe doped with In and Ga (*81*), Tl_2Te_3 (*82*), carbon in nanotubes (*83*), and amorphous silicon impregnated with hydrogen (*84*). In almost every case, the signature temperature dependences of the relaxation rate of the various nuclei indicate that electron−nuclear interactions dominate the nuclear relaxation behavior.

SYNTHETIC POLYMERS

Synthetic polymers have been studied with solid-state NMR techniques since the very early days of the methods. Polymeric materials are often solid or semisolid, and as such have many of the NMR properties of crystalline solids of low-molecular-weight materials. However, because there are often distributions of properties of nominally chemically identical sites, the spectroscopy is often quite complex. In addition, with the possibility of motion of some polymeric materials in the range of frequencies sampled by NMR, the interpretation of the NMR spectra is often complicated and far from simple. Even in solids composed of small molecules, the NMR dynamics can be very complex (*85*). Thus, the applications are many and varied.

Reactions of polymers can be monitored with NMR spectroscopy. The NMR study of polyacrylonitrile indicated the structural changes during thermal degradation (*86*). The weathering of kerogen was monitored with ^{13}C NMR spectroscopy to determine structural changes (*87*), as has polymer coating used as paints

(*88*). Curing and aging of elastomers (*89*) and vulcanized rubber (*90*) are also shown to be amenable to study by solid-state NMR spectroscopy, as is hydration of films of materials such as poly-(vinyl alcohol) (PVA) (*91*). Copolymers of ethylene with vinyl alcohol have also been analyzed with solid-state NMR techniques (*92*). The structure and dynamics of acrylate−polymer films on glass were examined with NMR (*93*). The binding of liquid crystalline polymers to chromatographic surfaces was clearly demonstrated in a study of the reactions that form these stationary phases using ^{29}Si and ^{13}C NMR spectroscopy (*94*). The mobility and structure of polycarbonate polymers grafted to silica particles have also been studied with NMR spectroscopy (*95*), as have composites of rubber with silica (*96*). A similar study showed that one could identify the linkage of urethane resins to wood with NMR spectroscopy (*97*), and a further study showed how these techniques can monitor the interaction of nylon-6 with clay particles (*98*). The interaction of drugs such as salicylic acid incorporated into polymer matrixes can be monitored to discover the types of interactions between the drug and the polymer (*99*).

Many polymer materials are of interest because of their mechanical or physical properties, particularly in complex formulations. Studies of mechanical properties with NMR spectroscopy are important analytical tools. Studies of cold-drawn high-density polyethylene showed that the "intermediate" component is a primarily all-trans configuration that has higher disorder than the crystalline component (*100*). Similar studies of the dynamics of isotactic poly(1-butene) have been reported (*101*). The dynamics of nylon-6 fibers spun at several speeds were examined with NMR, and the NMR properties were shown to change with the spinning rate (*102*). The effects of compositing nylon-6 with clay were investigated with relaxation measurements (*103*). Deuterium NMR was used to investigate the effects of uniaxial extension on partially deuterated poly(dimethysiloxane) (*104*). Composites of carbon black with poly(ethylene oxide) suggest the dynamics of the polymer chain are affected by the amount of carbon black (*105*). Studies of the ^{13}C NMR of epoxies were used to determine behavioral changes as a function of hardener content (*106*). The effects of inclusion of melamine in urethane foams was investigated with NMR; no discernible difference was measured with ^{13}C NMR, but ^1H provided evidence that melamine affects the char characteristics (*107*). Plasticization of polymers−affects the NMR spectra, as seen for poly(vinyl chloride) (*108*). A study of the dynamics of a poly(methyl methacrylate)−silicone network with relaxation studies showed that the material was not homogeneous (*109*). Aging studies of a polymer binder for propellants was monitored with relaxation measurements, in which a strong correlation between the cross-polarization time constant and the tensile elongation at break was noted (*110*).

Dynamics of polymer systems can be very complex, and NMR provides the means to study these from their effects on line shapes, relaxation times, and other parameters, as in polyethylene (*111*). In some cases, the combination of NMR studies with quantum ab initio calculations allows one to unravel complex dynamics, as in a study of siloxane networks (*112*). Relaxation studies of polyHIPE (high internal-phase emulsion) revealed variations in dynamics along the chain (*113*). Relaxation measurements on drawn bisphenol A−polycarbonates have been analyzed to determine the effects of stretching of the chains on dynamics

(*114*). The temperature-dependent deuterium NMR relaxation of deuterated poly(α-methylstyrene) has been used to discern mobility (*115*), as it has also been used to study dynamics in dendrimers (*116*). The temperature dependence of NMR line widths in ^{13}C MAS NMR spectra of six amorphous polymer materials was used, in the WLF limit, to predict the free volume of the polymer materials (*117*). In a study of poly(ferrocenyl-silanes), measurement of the cross-polarization behavior of a polymer containing butyl side chains indicated disorder of the chains (*118*). Similarly the effects of pressure on dynamics of chains in atactic polypropylene as monitored by the effects on NMR spectra yielded an average correlation time described by a Vogel−Fulcher function (*119*). The dynamics of copolymers of PVA with ethylene were studied to determine different motional components (*120*). NMR relaxation in copolymers of methyl acrylate and sodium methacrylate is interpreted in terms of interchain interactions (*121*). Cross-polarization dynamics between ^{19}F and ^{1}H in Viton fluoroelastomers are discussed in terms of the mobility in these polymers (*122*). The dynamical effects of addition of DMSO to a reverse-osmosis membrane were monitored by measurement of $T_{1\rho}$, which showed changes in the dynamics (*123*). One interesting NMR technique was the 2D correlation of proton NMR spectra of annealed polypropylene to identify relaxing regions of the polymer (*124*). A study of relaxation in isotactic polypropylene by NMR was interpreted in terms of defect concentrations (*125*).

Blends of several different polymers yield to NMR study because NMR can detect two species independently. In a study of blends of poly(acrylic acid) and poly(vinylpyrrolidone), NMR demonstrated that the two components hydrogen bond strongly through the observed changes in chemical shifts (*126*). The measurement of $T_{1\rho}$ also allowed the authors to specify the homogeneity scale of the blend. A similar relaxation result is reported for poly(vinylphenol-*co*-methyl methacrylate) with poly-(ethylene oxide) (*127*). Blends of poly(vinyl chloride) with other materials have been studied with ^{13}C NMR (*128*), as have blends of tri-*p*-tolylamine with bisphenol A−polycarbonate (*129*), phenolic resins with the bis(hydroxy ether) of bisphenol A (*130*), and polystyrene blends (*131*).

An unusual attempt at analytical use of solid-state NMR of polymers is its recent application to the study of condoms (*132*). Unfortunately, solid-state NMR could not provide a distinction among condoms due to the commonality of the latex of which most are made, so the authors developed a fingerprinting technique with solution-state NMR of extractates.

BIOLOGICAL SOLIDS

A wide variety of biologically derived materials can be probed with NMR spectroscopy. Good examples are estradiol (*133*), α-crystallin—the protein in the eye that keeps the lens transparent—in the dry and hydrated states (*134*), and tunneling-assisted relaxation in solid riboflavin (*135*). Analysis of deuterium NMR line shapes and relaxation times of model carbohydrates and molecular modeling showed that a two-site model for the glycosidic linkage fits the NMR data (*136*). The study of methyl group dynamics of aspirin and aspirin complexed with β-cyclodextrin has been reported (*137*). The study of a diacylglycerol, important

in cell signaling, showed polymorphic behavior distinguishable by NMR spectroscopy (*138*).

The analysis of humic substances is an area of ongoing study with NMR spectroscopy (*139*). A more unusual application of NMR is the degradation of wheat straw-amended feces in a composting experiment. The material was monitored with ^{13}C NMR spectroscopy to determine the types and varieties of organic constituents at various stages (*140*). Similarly, the degradation of wheat straw during the growth of mushrooms allowed quantification of various components (*141*). In some sense, the reverse of this process is the analysis of whole plants and parts of plants grown in controlled conditions to determine where ^{13}C enrichment occurs (*142*) or the examination of the genetically modified trees to determine chemical changes that can be detected with NMR spectroscopy (*143*). In one interesting twist, composites of sugarcane bagasse with thermoplastics were analyzed with NMR spectroscopy (*144*).

The study of wood and cellulose is an important area in which NMR spectroscopy makes a contribution. Applications to wood pulps (*145−147*) and to celluloses from a variety of materials (*148*) have been reported. Degradation in air has been monitored by NMR experiments (*149*). These experiments sometimes involve sophisticated data analyses (*150, 151*). In some experiments, the functionalization or chemical treatment of cellulose is monitored with NMR spectroscopy to provide information on the structures formed (*152−155*). In one report, the presence of ^{15}N in an isocyanate linked to wood allowed discrimination in the NMR spectroscopy (*156*).

Water plays an important part in celluloses, so the dynamics and structure of water in cellulose have also been investigated with NMR spectroscopy (*157, 158*). The types of organic materials removed from aquatic waters by a resin were determined by ^{13}C NMR spectroscopy as a way to evaluate differences in the sorptive properties of the resins (*159*). The study of relaxation times of water in frozen emulsions with oil allowed the authors to determine the dynamic state of the water as primarily a solid (*160*). Relaxation analysis can also discriminate the constituents of semisolid milk fats (*161*).

An interesting use of NMR spectroscopy of biological materials is given in a study of the organic matter found at Neolithic pits in Bavaria (*162*). The ^{13}C NMR spectroscopy of these archaeological samples allowed a measurement of the aromatic content of many of the materials. Yet another unique use of NMR spectroscopy is the study of cosmetic emulsions (*163*).

CONCLUSIONS

One is continually amazed at the wide variety of applications of NMR spectroscopy to problems of interest to chemists, physicists, geologists, biologists, engineers, and other scientists. The focus of an experiment may be the structure of a particular site in a biological material, the products of a reaction, the solid-state structures of compounds, the dynamics of molecules in restricted environments, or the connection of microscopic properties to technologically important uses of materials. NMR spectroscopic analyses give information readily used by the chemist to answer fundamental questions about each of these environments. In addition, even though it was first discovered over half a century ago, intellectual challenges in designing and using experiments

to separate or correlate the components of spectra still occupy the thoughts and efforts of many spectroscopists. The examples above attest to the continual development of this technique.

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