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NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

by

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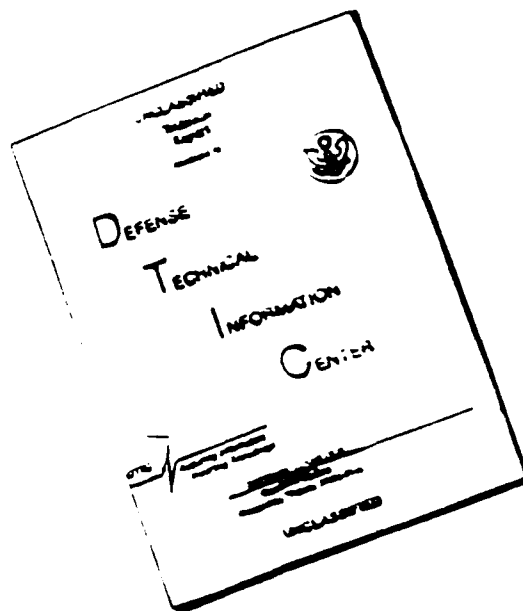
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Nuclear Magnetic Resonance Spectroscopy

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TXT03

INTRODUCTION AND SCOPE

SEN03 1 NMR continues to grow and find new applications. A
SEN06 3 maturing understanding of spin physics has allowed the de-
11 velopment of sophisticated techniques for assigning reso-
17 nances, determining internuclear distances, and averaging or
SEN09 24 exploiting orientation-dependent interactions. The recent and
8 continuing revolution in biotechnology would not be possible
13 without the increasingly elegant NMR methods that are used
22 routinely to characterize the structure and dynamics of pro-
30 teins and nucleic acids and their interactions with other
SEN12 39 biomolecules. NMR imaging has revolutionized diagnostic
37 medicine and is beginning to have an impact on nondestructive
SEN15 17 materials testing. Spatially localized spectroscopy is revealing
7 the chemistry of the life process noninvasively.

PAR06
SEN03 1 The few remaining obstacles to high-resolution NMR
SEN06 9 spectroscopy of solids are falling rapidly. Multidimensional
3 solid-state NMR experiments are revealing the structures of
SEN09 11 catalysts. High-temperature catalytic reactions are being
7 simulated in NMR probes so that reaction mechanisms may
SEN12 16 be elucidated. NMR studies of high- T_c superconductors have
8 been underway in a number of laboratories since shortly after
SEN15 18 the discovery of the prototypical materials. NMR spectroscopy
3 has proven itself to be exceptionally adaptable to new
SEN18 13 problem areas. A good example of this was recently provided
10 by Yannoni who accurately measured the bond distances in
19 C_{60} months before the first crystal structure of a fullerene
29 derivative was determined.

PAR09
SEN03 1 NMR spectroscopy continues to be invaluable as a routine
SEN06 11 technique for the study of structure and dynamics of organic
21 and inorganic compounds. A significant fraction of the articles
8 in the chemical literature mentions data obtained by NMR
17 spectroscopy, if only in passing; and most chemists readily
26 appreciate why Richard Ernst was awarded the Nobel Prize
35 in chemistry for his contributions to NMR spectroscopy.

PAR12
SEN03 1 My predecessor surveyed the literature through March of
SEN06 10 1990. A search of *Chemical Abstracts* revealed 6954 publi-
9 cations on NMR spectroscopy between April 1990 and De-
17 cember 1991 and 7936 publications for the entire biennium.
1 The corresponding totals for 1970 plus 1971 and 1980 plus
SEN09 12 1981 were 3186 and 5808, respectively. NMR spectroscopy
SEN12 12 as a scholarly enterprise is clearly in good health. Ironically,
SEN15 4 the number of NMR articles published in this journal is down
3 from 29 in 1980-1981 to 18 in 1990-1991. It is clearly im-
SEN18 14 possible to cite all 6954 publications, and to do so nonselec-
15 tively would probably not be much of a contribution. I have
SEN21 15 emphasized a chemist's perspective. I have completely ignored
SEN24 4 clinical work and largely ignored in vivo experiments and
SEN27 15 studies of excised tissue. I have tried to give a balanced
9 coverage of the quickly expanding literature on proteins and
SEN30 12 nucleic acids in solutions. The coverage of imaging and
7 spatially localized spectroscopy emphasizes technique de-
12 velopment and materials science applications rather than
SEN33 19 human or animal studies. The coverage of NMR spectroscopy
7 of solids and chemical applications thereof is probably the
16 most complete, reflecting my better knowledge of that liter-
24 ature.

PAR15
SEN03 1 The division of topics is necessarily arbitrary. For example,
SEN06 4 some of the citations in the Multidimensional NMR Spec-
12 troscopy section could have been placed in the Biomolecules
SEN09 21 in Solution section and vice versa. Even the division between
6 topics like Synthetic Polymers in Solution and Solid Polymers
15 seems arbitrary, but division is necessary even if only to reduce
26 the task of writing this review to a series of manageable steps.

TXT06

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PAR18
SEN03 1

BOOKS

SEN03 1 A number of books on NMR spectroscopy have either been
12 published or caught the eye of other reviewers in the past 20
SEN06 24 months. In many cases, it was easier to find a book review
13 in the *Journal of the American Chemical Society*, *Journal*
22 *of Magnetic Resonance*, or some other publication than to get
SEN09 32 access to the book itself. Making a virtue of necessity, I have
SEN12 9 cited both the books and their respective reviews. There were
SEN15 4 quite a few additions to numbered series (A1-A10). Marshall
SEN18 3 published a new book on the Fourier transform (A11). Slichter
7 published a third edition of *Principles of Magnetic Resonance*

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12 (A12), and several other texts were published and reviewed
SEN21 21 (A13-A17). Two books discussed biological applications (A18,
SEN24 8 A19). Others dealt with imaging (A20), polymer micro-
SEN27 8 structure (A21), and ^{31}P (A22). Farrar published a second
6 edition of his text *Introduction to Pulse NMR Spectroscopy*
15 (A23) and the first volume of this treatment on density ma-
25 trices (A24).

TXT09

SEN03 1 CONCEPTS IN MAGNETIC RESONANCE

SEN03 1 A positive development in the NMR literature was the
PAR21 creation of the journal *Concepts in Magnetic Resonance* which
SEN03 1 is subtitled "An Education Quarterly". Each issue publishes
SEN06 20 three or four tutorials on various aspects of NMR spectro-
5 scopy. The level is variable, but is generally well suited to a
SEN09 14 second-year student specializing in NMR spectroscopy.
13

PAR24 1 Some of the articles which caught my eye included treat-
SEN03 1 ments of density matrices (B1, B2), instrumentation (B3-B6),
11 CRAMPS (B7), shaped pulses (B8), imaging (B9), and chem-
19 ical shift anisotropy (B10).
27

TXT12

SEN03 1 INSTRUMENTATION

SEN03 1 The best way to learn of new instrumental developments
11 in NMR spectroscopy is to attend the annual Experimental
20 NMR Conference (ENC) and tour the poster sessions and
SEN06 20 vendor suites. The progress in instrumentation development
7 in this field is understated by the relatively few papers that
SEN09 18 have a primarily instrumental focus. This is due in part to
8 the tendency of spectrometer and probe vendors not to publish
SEN12 18 in the open literature. NMR spectroscopists also have a
7 tendency to downplay the role of instrument development,
15 relegating its description to a subordinate role in papers de-
24 voted to chemical or spectroscopic results or to presentations
33 at the ENC.

PAR30 1 High pressures are sometimes desirable for forcing equi-
SEN03 1 librium or characterizing a variety of physical phenomena.
9
SEN06 1 Two improvements in sample tubes for high-pressure solution
10 NMR spectroscopy have been described (C1, C2).

PAR33 1 One of the most important recent advances in solid-state
SEN03 1 NMR spectroscopy was the development of double rotation
11 (DOR) and dynamic-angle spinning (DAS) for high-resolution
19 studies of noninteger quadrupole nuclei. These techniques
SEN06 26 are extremely demanding on the design of the spinning sys-
4 tems. Pines and co-workers have described designs of DOR
SEN09 13 (C3) and DAS (C4) probes and their applications to selected
10 problems (see sections on solids). Another spinning system
SEN12 20 suitable for DAS studies has also been described (C5). Yan-
SEN15 5 noni and co-workers described the design of a magic-angle-
2 spinning (MAS) probe that works at temperatures down to
19 5 K and used it to study carbocations at near-liquid-helium
SEN18 29 temperatures (C6, C7). Stebbins has reviewed high-temper-
SEN21 5 ature solid-state NMR spectroscopy (C8). Temperatures over
4 1000 K have been achieved with a laser heating system (C9),
15 and more conventional high-temperature probe designs have
SEN24 22 also been reported (C10, C11). Inductive heating has also been
7 proposed as a method for achieving very high temperatures
SEN27 16 (C12). An apparatus for achieving temperature jumps in a
10 short time has also been reported (C13).

PAR36 1 Accurate measurement of sample temperatures in NMR
SEN03 1 spectroscopy can be a vexing problem. Refinements in ratio
SEN06 9 pyrometry have been reported that seem applicable to very
5 high temperatures (C14, C15). Chemical shift thermometry
SEN09 14 for MAS NMR spectroscopy and its application to measuring
5 temperature gradients have received attention (C16-C18).
14 Low-temperature probe designs for dilution refrigerator (C19)
SEN12 1 and matrix isolation experiments were also described (C20).
9

PAR39 1 A spinning speed controller for MAS experiments was de-
SEN03 1 scribed (C21) as were several useful radio-frequency devices
10 (C22-C24). A spectrometer based on a dc SQUID that is
SEN06 18 suitable for NQR and low-frequency NMR spectroscopy has
11 been developed (C25).
19

TXT15

SEN03 1 DATA PROCESSING, CALCULATIONS, AND
6 SIMULATIONS

PAR42 1 The data processing demands of modern NMR experiments
SEN03 1 continue to grow as a result of multidimensional spectroscopy,
10 computationally-intensive alternatives to Fourier transfor-
19 mation, distance-geometry calculations from NOE data, etc.
23 This has been reflected in two trends in data processing
SEN06 1 hardware. Spectrometer vendors are starting to move away
SEN09 12 from in-house data systems to generic interfaces that connect
9 to commercial high-performance workstations. Secondly, data
SEN12 18 processing is increasingly performed off-line. Indeed, several
SEN15 4

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PAR42

4 fairly sophisticated NMR data processing packages are
SEN18 11 available that run on PCs and McIntosh computers. As NMR
4 spectroscopists begin to make use of recent advances in com-
13 putational chemistry, techniques such as molecular mechanics
20 and ab initio calculations will be integrated with experimental
SEN21 29 results. This section reviews recent developments in data
9 processing and other computer-intensive reports including
15 spectral simulation, interpretation, and chemical shift calcu-
21 lations.

PAR45

SEN03 1 NMR data processing was reviewed in detail by Hoffman
SEN06 11 and Levy (D1). Several parts of an introductory review on
9 the Fourier transform have appeared in *Journal of Chemical*
SEN09 18 *Education* (D2-D4). Improvements in multidimensional FT
SEN12 6 data processing have also been discussed (D5, D6). Both linear
4 prediction and maximum entropy methods have been explored
12 as alternatives to Fourier transformation, and these methods
SEN15 20 have been reviewed (D7). Many workers regard the im-
6 provements in resolution and sensitivity provided by maxi-
SEN18 13 mum entropy to be purely cosmetic. Jones and Hore have
SEN21 6 critiqued this method (D8, D9). Several improvements in
5 linear prediction methods have been reported (D10, D11).
SEN24 1 Even standard nonlinear least squares has been considered
SEN27 10 as an alternative to Fourier transformation (D12). If the FT
5 is used (as it still is in almost all cases), the resulting spectrum
18 requires phase correction; and new algorithms for doing this
27 automatically have been reported (D13, D14).

PAR48

SEN03 1 Computer methods are being used for structural elucidation
10 (D15) including the application of neural networks (D16) and
SEN06 10 graph theory (D17, D18). Chemical shifts have been analyzed
7 or predicted using structure-property relationships or other
SEN09 14 methods (D19-D30). First-principles calculation of chemical
6 shifts have been carried out using ab initio or other molecular
17 orbital methods (D31-D38).

PAR51

SEN03 1 Several unrelated computer-intensive studies that also
SEN06 8 deserve citation include the following. A statistical method
5 for correcting for finite spinning speeds in magic-angle-spin-
SEN09 12 ning spectra was reported (D39). A procedure for interactive
SEN12 6 product-operator calculations was described (D40). Ernst has
4 discussed computer-optimized TOSCY experiments (D41).
SEN15 1 Finally, a method to evaluate the octane number of gasolines
12 from ¹H spectra was reported (D42).

TXT18

RELAXATION PHENOMENA

SEN03

PAR54 1 Relaxation is at least an incidental issue in many of the
SEN03 1 papers cited in other sections of this review. Several stand-out
SEN06 13 papers dealing mainly with relaxation are cited here. Anet
SEN09 4 and co-workers have shown that there can be an antisymmetric
3 component of the shielding tensor that can, under special
13 circumstances, result in the surprising observation of $T_2 > T_1$
22 (E1). Interesting relaxation effects in solids were reported
SEN12 32 (E2-E5). Farrar has been exploring differential line broad-
SEN15 9 ening and other relaxation phenomena in solution (E6-E8),
16 and related ideas have been applied to macromolecules (E9)
25 and membrane structure (E10).

PAR57

SEN03 1 Several unrelated relaxation studies also caught my eye.
SEN06 1 Relaxation rate matrix analysis was discussed for interproton
10 distance determination (E11). T_2 processes in porous rocks
18 were studied (E12). T_1 was used to probe surface viscosity
26 (E13), and field-cycling relaxation spectroscopy was used to
SEN09 28 study protein backbone fluctuations (E14). Other contribu-
3 tions relating to relaxation theory included a treatment of
12 relaxation under continuous rf fields (E15) and solutions of
21 the Bloch equations in the linear response approximation
29 (E16).

TXT21

MISCELLANEOUS DEVELOPMENTS IN
SOLUTION NMR SPECTROSCOPY

SEN03

PAR60 1 NMR pulse sequences were once composed nearly exclu-
SEN03 9 sively of rectangular pulses. Advances in rf electronics and
SEN06 9 theoretical methods for obtaining shaped pulses tailored for
15 specific purposes have led to a continuum of designer pulse
SEN09 25 sequences. Shaped pulses can be designed using a variety of
11 methods including average Hamiltonian theory and iterative
SEN12 11 numerical simulations. Gezelter and Freeman have recently
7 reported the use of neural networks simulated on a serial
SEN15 17 computer for the design of shaped pulses (F1). The early
SEN18 4 results are very encouraging. Shaped pulses are commonly
6 used for selective excitation (F2, F3).

PAR63

SEN03 1 Molecular hydrogen exists in two isomers with a total spin
SEN06 12 of 1 (ortho hydrogen) or 0 (para hydrogen). In 1987 Wei-
4 tekamp proposed and later demonstrated that very large
12 enhancements in ¹H signal intensities could be obtained by
21 synthetically incorporating hydrogen enriched in the para

TXT21
PAR63

28 isomer into a product of interest immediately prior to the rf
SEN00 39 pulse. This effect is analogous to CIDNP and was given the
12 name PASADENA (para hydrogen and synthesis allows dy-
SEN12 12 namically enhanced nuclear alignment). Eisenbert has re-
4 viewed his own work with para-hydrogen-induced polarization
11 (F4), and Bargon and co-workers have demonstrated an
19 analogous effect with enriched ortho hydrogen (F5).

PAR66

SEN03 1 Another new twist that is in its infancy is a laser version
SEN06 14 of NMR spectroscopy on optical-phase conjugation. The
3 theory of this radically new form of spectroscopy has been
13 outlined by Evans (F6), and Warren has undertaken exper-
SEN06 21 imental verification. Ultrasound might offer a new way to
SEN12 9 manipulate relaxation times (F7). Very strong magnetic field
6 gradient pulses are being incorporated in a number of ex-
15 periments (F8), and high-resolution spectroscopy in a static
23 magnetic field gradient has been demonstrated (F9, F10).
SEN15 1 Improvements in cross polarization in solution have been
SEN18 10 reported (F11-F13). Bodenhausen has described a graphical
7 approach for understanding NMR experiments that is more
SEN21 15 intuitive than density-matrix methods (F14). Solvent sup-
SEN24 3 pression continues to be an important problem. This area has
5 recently been reviewed (F15).

TXT24

SEN03 1 MULTIDIMENSIONAL NMR SPECTROSCOPY

PAR69

SEN03 1 Multidimensional NMR spectroscopy used to be a fancy
SEN06 10 way of saying 2-D NMR spectroscopy. In the last several years
SEN09 7 it has come to include 3- and 4-D NMR spectroscopy. Just
3 as a 2-D experiment involves an evolution period and a de-
13 tection period, a 3-D experiment involves two evolution periods
SEN12 22 prior to detection. The three time periods correspond (after
SEN15 8 Fourier transformation) to the three frequency axes. The
3 development of higher-dimensional experiments is largely
9 motivated by the application of NMR spectroscopy to in-
17 creasingly more complicated problems in biological chemistry
SEN18 24 involving molecules of ever higher molecular weight. The
3 improved resolution afforded by multidimensional filtering
9 has allowed NMR structures to be determined for proteins
SEN21 18 of 150 residues or greater. Applications to protein structure
6 determination have been covered in two recent reviews (G1,
SEN24 15 G2). The use of 2-D NMR spectroscopy to study chemical-
SEN27 10 exchange kinetics has also been reviewed (G3). There were
4 a number of reports of applying 3-D (G4-G11) and 4-D
14 (G12-G15) NMR spectroscopy to proteins as well as appli-
22 cations of multidimensional NMR spectroscopy to nucleic
SEN30 29 acids (G16-G19). 2-D NMR spectroscopy was applied to
8 paramagnetic molecules (G20) and at high pressure to study
SEN33 17 phospholipid vesicles (G21). An improved method for 2-D
7 data collection was also proposed (G22).

TXT27

SEN03 1 BIOMOLECULES IN SOLUTION

PAR72

SEN03 1 There was a large number of applications of NMR to
SEN06 12 proteins, nucleic acids, and other biomolecules in solution. In
3 a review article such as this, one cannot hope to do justice to
SEN09 16 this large and diverse field. I have attempted to cite repre-
7 sentative work to highlight trends rather than provide a
16 comprehensive listing of citations.

PAR75

SEN03 1 NMR studies of proteins in solution were reviewed (H1-
SEN06 10 H3). The combination of NMR spectroscopy and molecular
9 mechanics or molecular dynamics has become common (H4-
SEN09 16 H8). NMR spectroscopy has been used to study protein
SEN12 10 folding (H9-H12), and metal binding (H13-H17). Studies of
4 binding to cytochrome c (H18, H19) and nucleic acids (H20-
SEN18 13 H22) have also been reported. Paramagnetic effects continue
SEN18 5 to be exploited in protein studies (H23-H26). Triple-reso-
2 nance studies have probed protein structure (H27, H28).
SEN21 1 Loops in nucleic acids were studied (H29, H30). Carbohy-
SEN24 2 drates were the subject of other investigations (H31, H32).

TXT30

SEN03 1 SYNTHETIC POLYMERS IN SOLUTION

PAR78

SEN03 1 This field has reached a high level of maturity. ¹³C NMR
SEN06 4 analysis of polymers was recently reviewed (I1). ¹⁹F analysis
SEN12 4 has been applied to Nafion (I2). The chemistry of poly-
5 phosphazenes and related materials has been studied (I3-I5).
SEN15 1 Many studies have dealt with microstructures (I6-I10) or
SEN18 10 conformation (I11, I12). 2-D NMR spectroscopy was used to
SEN21 8 probe polymer-polymer interactions (I13). A ¹³C study of a
7 soluble polydiacetylene was reported (I14).

TXT33

SEN03 1 INORGANIC COMPOUNDS IN SOLUTION

PAR81

SEN03 1 One of the more interesting applications of NMR spec-
10 troscopy in recent years was the proposal by Hamilton and
20 Crabtree that ¹H T₁ measurements could be used to distin-
29 guish between classical and nonclassical transition-metal
SEN06 35 hydrides. Halpern and co-workers recently published a de-

in a...
- based

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PAR81

8 tailed assessment of this method (J1) which also serves as a
19 good overview of the relevant literature. Olah has published
5 NMR studies of inorganic ions (J2, J3). Bell has studied
5 aluminosilicate and borosilicate solutions (J4), while Oster-
11 young has characterized molten salts (J5).

PAR84

1 Variable-temperature and/or variable-pressure measure-
5 ments have been used to characterize dynamics (J6-J10). ¹⁷O
3 analysis has been applied to polyoxo complexes (J11, J12).
1 SEN09 1 ¹⁹⁵Pt NMR spectroscopy has been used to study the chemistry
12 of anticancer agents (J13-J16). NMR analysis of various other
7 metal nuclei has been applied to a range of chemical problems;
18 a representative sampling is cited (J17-J26).

TXT36**SOLIDS IN GENERAL**

1
1 SEN03
1 PAR87

1 Some recent developments in NMR analysis of solids have
11 been reviewed by Chmelka and Pines (K1). One of the more
6 interesting new experiments has been Tyko's development
13 of zero-field NMR spectroscopy in high field (K2, K3). Several
3 approaches for the study of dynamics by multidimensional
11 spectroscopy or line shape analysis have been reported (K4-
19 K7). An anomalous effect of MAS speed on chemical shifts
11 of cuprous halides has been interpreted as due to the Lorentz
22 force (K8). Thankfully, this effect is restricted to ionic con-
9 ductors.

PAR90

1 **Proton and Fluorine Spectroscopy.** The spectroscopy
1 of abundant spins in solids (usually ¹H or ¹⁹F) is often dom-
1 SEN06 4 inated by strong homonuclear dipolar couplings. Progress
15 continues to be made in coherent averaging by multiple pulse
3 sequences and/or high speed MAS. Some of the most im-
13 portant applications of ¹H NMR analysis to solids are in the
6 area of catalysis. Protons are typically chemically dilute on
17 oxide surfaces, and MAS at modest speeds usually suffices
1 SEN15 8 for high resolution. Thus, ¹H studies of solid catalysts are
17 reviewed in the Adsorption Phenomena and Catalysis section.
9

PAR93

1 New developments in the spectroscopy of abundant species
10 in solids include pulse sequences for homonuclear dipolar
18 decoupling (K9, K10), elimination of probe ringing in mul-
26 tiple-pulse spectroscopy (K11), and a treatment of residual
1 SEN06 34 line widths under MAS (K12, K13). Applications of ¹⁹F NMR
15 spectroscopy of solids have been reviewed (K14). An im-
3 provement in ¹⁹F CRAMPS has been described (K15), and
12 fast MAS ¹⁹F analysis has been applied to materials related
22 to dental enamel (K16). Chemical applications of ¹H
6 CRAMPS have also been published (K17, K18).

PAR96

1 **Distance Measurements.** There has been a renaissance
1 SEN03 1 in the use of dipolar couplings to determine internuclear
7 distances. These couplings are inversely related to the third
16 power of the separation. Although nonspinning techniques
12 continue to be useful (K19), there have been several break-
14 throughs that have allowed information about dipolar cou-
21 plings to be preserved in high-resolution spectra obtained with
30 MAS. Tyko has described the DRAMA sequence for "dipolar
1 SEN18 10 recovery at the magic-angle" (K20). Schaefer and co-workers
5 have developed a rapidly growing family of rotational-echo,
13 double-resonance (REDOR) experiments for measuring het-
18 eronuclear dipolar couplings (K21-K27). Griffin and co-
4 workers have exploited the rotational resonance condition to
12 measure homonuclear couplings between spin pairs (K28).
1 SEN24 1 Cross polarization between isolated spin pairs has also been
11 proposed for heteronuclear distance determinations (K29).
1 SEN27 1 Several applications of the above techniques to distance de-
10 termination in biomolecules and C₆₀ are cited in the sections
20 which follow.

PAR99

1 **Spectral Assignment.** The development of techniques
1 SEN03 1 for spectral editing and heteronuclear correlation is proceeding
6 vigorously. Burum has described the WIMSE technique for
14 distinguishing among CH, CH₂, and CH₃ groups in ¹³C CP/
9 MAS spectroscopy (K30). Sethi has described spectral editing
18 in a one-dimensional separated local field approach (K31).
7 Two- (K32) and three-dimensional (K33) heteronuclear cor-
1 SEN15 1 relation experiments in solids have been performed. Dou-
8 ble-quantum filtering has been used to select resonances from
2 pairs of dipolar-coupled spins (K34). An alternative approach
11 to spectral editing via "cross depolarization" has also been
5 proposed (K35, K36).
14

PAR102

1 **Polarization.** Several papers have dealt with the polari-
1 SEN03 1 zation of the nuclear spins in solids. Very high polarizations
8 have been achieved from a photoexcited triplet state and
5 dynamic nuclear polarization (K37) or alternatively by optical
14 pumping (K38). The Overhauser effect has been exploited
22 in MAS spectra (K39). Hartmann-Hahn cross polarization
1 SEN14 8 has been reexamined (K40-K42).
5

PAR105

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PAR105

SEN03 1 **Chemical Shift Anisotropy.** There is a lot of interest in
SEN06 9 further developing methods for recovering CSA data from high
SEN09 18 resolution MAS spectra. A variety of strategies for doing this
9 were proposed or refined during the period of the review
SEN12 19 (K43-K50). Chemical shift tensors were measured using
8 single-crystal methods (K51) and in the presence of dipolar
SEN15 17 couplings (K52). Refinements in the TOSS experiments for
8 sideband suppression were also reported (K53-K55).

PAR108

SEN03 1 **Coupling and Decoupling.** Scalar coupling is usually less
SEN06 7 important in solid-state vs solution-state NMR as a result of
17 the lower resolution, and in the case of scalar coupling to ^1H ,
SEN09 29 the need to remove dipolar couplings. J couplings can be
6 important in ^{31}P MAS spectra and for many inorganic com-
SEN12 15 pounds. In the case of highly mobile systems such as weak-
11 ly-adsorbed molecules on catalysts, plastic crystals, inclusion
18 complexes, or elastomers, the NMR properties may be in-
28 termediate between solidlike and solution-like, and even J
SEN15 34 couplings to ^1H may be observed. Several articles focused on
SEN18 6 J coupling during the period of the review (K56-K62). Im-
2 proved strategies for ^1H dipolar decoupling in ^{13}C MAS NMR
12 were also proposed (K63-K65).

PAR111

SEN03 1 **Quadrupoles.** The development of DOR and DAS was
SEN09 9 described above. Several applications of DOR are cited in the
SEN12 10 section on Adsorption Phenomena and Catalysis. Pines has
SEN15 4 described pure adsorption phase DAS (K66). Most experi-
3 ments on quadrupoles used MAS or variable-angle spinning
SEN18 11 (VAS) (K67-K75). Several developments in ^2H spectroscopy
SEN21 7 were reported (K76-K79). The effects of dipolar coupling to
8 $I = 1$ nuclei on the spectrum of spin- $1/2$ nuclei were discussed
SEN24 20 (K80-K82). NQR with a dc SQUID was developed (K83).

TXT39

SEN03 1 ADSORPTION PHENOMENA AND CATALYSIS

PAR114
SEN03 1 The application of NMR methods to the study of catalysts
SEN06 12 and adsorbed species continues to grow. At first glance, NMR
6 spectroscopy might seem inappropriate to the study of surfaces
15 due to its low inherent sensitivity relative to traditional forms
SEN09 25 of surface spectroscopy. Indeed, NMR studies of two-di-
6 mensional surfaces have been few in number, and there has
16 been little effort to connect NMR studies to the single-crys-
SEN12 25 tal-face world of UHV surface science. The virtue of NMR
6 spectroscopy is that it is frequently applicable to the actual
SEN15 16 working catalyst with remarkable sensitivity. Amorphous
3 oxide and zeolite catalysts typically have surface areas mea-
SEN18 11 sured in tens or hundreds of square meters per gram. Since
3 radio waves readily penetrate through such catalysts, the
11 NMR experiment integrates over a considerable surface area.
SEN21 1 NMR spectroscopy is now being used in an in situ mode to
14 investigate the reaction mechanisms of various catalytic
21 processes.

PAR117

SEN03 1 NMR analysis has also made remarkable contributions to
10 the understanding of framework structures of zeolites and
SEN06 18 aluminum phosphate molecular sieves. Although most such
6 studies involve MAS, DOR was quickly applied to sharpen
14 the resonances of quadrupolar nuclei in catalysts.

PAR120

SEN03 1 This section also reviews NMR studies of surface-bound
10 alkylsilanes as well as clathrates and inclusion complexes.

PAR123

SEN03 1 **Reviews.** Slichter and co-workers have reviewed NMR
SEN06 8 techniques for the study of supported transition-metal cata-
SEN09 15 lysts (L1). Fyfe and co-workers reviewed the use of NMR
10 spectroscopy to determine zeolite lattice structure (L2).
SEN12 1 Klinowski reviewed molecular sieve catalysts (L3, L4). ^1H
SEN15 3 MAS studies were reviewed by Mastikhin and co-workers (L5).

PAR126

SEN03 1 ^1H Studies. Magic-angle spinning alone generally suffices
SEN06 7 for reasonable resolution in studies of catalysts, and the re-
SEN09 16 sidual line width may simply reflect heterogeneity (L6). Other
3 ^1H studies dealt with acid sites and acidity (L7-L11), silanol
SEN12 13 groups (L12), and adsorbates (L13, L14). In a similar vein,
6 ^{19}F MAS NMR spectroscopy has also been applied to catalysts
16 (L15).

PAR129

SEN03 1 **Zeolite Frameworks.** The application of standard ^{29}Si
SEN06 7 and/or ^{27}Al MAS NMR spectroscopy to the study of frame-
SEN09 16 works is a routine characterization method (L16-L24). The
3 combination of NMR spectroscopy and X-ray powder dif-
SEN12 10 fraction can be fruitful (L25-L27). Fyfe and co-workers have
6 been determining bonding connectivities in zeolites by 2-D
SEN15 14 solid-state experiments (L28-L33). Gallium is sometimes
5 substituted for aluminum in zeolite synthesis, and $^{69/71}\text{Ga}$ MAS
14 NMR spectroscopy has been explored in their study (L34,
23 L35).

PAR132

SEN03 1 **Organic Adsorbates.** ^{13}C MAS NMR spectroscopy has
SEN06 7 been used to characterize reaction products on catalysts.

TXT39
PAR132

SEN09 15 sometimes after progressive off-line heating (L36-L41). Haw
SEN12 3 and co-workers have studied catalytic reaction mechanisms
SEN12 10 with an in situ variable-temperature approach. Processes
SEN12 3 studied include reactions of unsaturated hydrocarbons
SEN15 9 (L42-L44), cracking reactions (L45), and methanol-to-gasoline
SEN15 8 chemistry (L46, L47). MAS NMR spectroscopy has also been
SEN18 17 used to study templates (L48-L52) and the effects of adsor-
SEN18 4 bates on ^{29}Si spectra of zeolites (L53). Several studies ad-
dressed coke formation on catalysts (L54-L58).

PAR135

SEN03 1 **Inorganic Adsorbates.** Inorganic clusters and organo-
SEN03 5 metallic clusters on catalyst supports are of interest for their
SEN09 15 roles as catalysts, catalyst precursors, or other materials such
SEN09 24 as semiconductors. NMR spectroscopy was used to charac-
SEN12 7 terize a number of such materials (L59-L65). Molybdenum
SEN12 3 and vanadium oxides on alumina and silica were studied by
SEN15 13 ^{95}Mo and ^{51}V MAS NMR spectroscopy (L66-L71). ^{31}P MAS
SEN15 4 was used to characterize zeolite HZSM-5 modified with or-
thophosphoric acid (L72).

PAR138

SEN03 1 **Metal Surfaces.** The NMR observation of species ad-
SEN06 7 sorbed on metal surfaces can be complicated by susceptibility
SEN09 16 problems, Knight shifts, and site heterogeneity. Several
SEN12 3 studies addressed CO adsorption on colloidal Pt or Pd
SEN12 12 (L73-L75). The observation of CO on a Pt electrode by NMR
SEN15 12 spectroscopy has also been reported (L76). CO and other
SEN15 5 adsorbates have been characterized on supported-metal cat-
SEN18 11 alysts (L77-L80). CO and other adsorbates have been char-
SEN21 8 acterized on supported-metal catalysts (L77-L80). NMR
SEN21 3 analysis of adsorbed hydrogen has been used to probe chem-
SEN24 12 isorption (L81-L86). Promotor effects on supported metals
SEN24 7 have also been studied (L87, L88).

PAR141

SEN03 1 **Aluminophosphate Molecular Sieves.** The growing
SEN06 4 family of AlPO_4 frameworks and their derivatives prepared
SEN09 12 by substituting other elements for Al are well suited for NMR
SEN09 23 study. Pines double rotation (DOR) technique has been
SEN12 9 applied to obtain high-resolution ^{27}Al spectra of these materials
SEN12 18 (L89, L90). ^{31}P and ^{27}Al MAS NMR spectroscopy has also
SEN15 10 been applied to characterize AlPO_4 frameworks (L91-L96).
SEN15 11 Adsorption of water and other species has been studied
SEN15 20 (L97-L99), and ^{129}Xe NMR analysis has been applied (L100,
L101).

PAR144

SEN03 1 **Other Applications.** Cations in zeolites have been studied
SEN09 8 by ^{133}Ca , ^{23}Na , and other techniques (L102-L106). ^{129}Xe NMR
SEN09 13 spectroscopy has been applied as a probe by Dybowski,
Fraissard, and others to address a number of problems in
SEN12 23 adsorption phenomena (L107-L126). In conjunction with
SEN12 5 multiple-quantum NMR spectroscopy, ^{129}Xe analysis has been
SEN12 12 applied to determine adsorbate heterogeneity in zeolite sam-
SEN15 19 ples (L127). Amorphous carbons have been studied (L128,
SEN18 8 L129). Diffusion in catalysts has been studied by a pulsed
SEN18 11 field gradient (L130, L131) and tracer-exchange spectroscopy
SEN21 18 (L132). Acidity has been investigated by NMR analysis of
SEN24 10 probe molecules (L133-L139). The structure and dynamics
SEN24 6 of alkylsilyl-modified silica have been studied by various
SEN24 14 methods (L140-L143).

PAR147

SEN03 1 **Clathrates and Inclusion Compounds.** NMR studies
SEN06 4 of a variety of organic (L144-L153) and inorganic (L154-L158)
SEN09 13 guest molecules have been reported. ^{129}Xe analysis has been
SEN09 6 applied to the study of clathrates (L159, L160).

TXT42

PAR150

SOLID BIOLOGICAL SYSTEMS

PAR150

SEN03 1 **Solid Biomolecules.** The resolution available in NMR
SEN06 7 studies of solid proteins and nucleic acids cannot hope to
SEN09 17 approach those of analogous studies in solutions. One reason
SEN09 4 for this is the lack of conformational averaging in typical
SEN12 14 powder samples. Bryant has shown that the resolution ob-
SEN12 8 tained in ^{13}C CP/MAS spectra of lyophilized lysozyme can
SEN15 17 be improved by partial hydration (M1). Solid-state studies
SEN15 4 of biomolecules are motivated by reasons similar to those that
SEN15 14 motivate other such investigations, low solubility, confor-
SEN18 20 mational change upon dissolution, etc. The resolution problem
SEN18 5 is frequently dealt with by labeling, or sometimes double
SEN21 14 labeling, the structural feature of interest. The development
SEN21 4 of MAS experiments that preserve dipolar couplings has af-
SEN21 12 forded the opportunity for doing distance determinations that
SEN21 20 cannot conveniently be done crystallographically for a lack
SEN24 28 of suitable crystals. One such method is rotational resonance,
SEN24 8 which is applicable for measuring internuclear distances from
SEN27 16 homonuclear dipolar couplings. Griffin and co-workers have
SEN27 6 used rotational resonance to identify the configuration of
SEN30 14 retinal in bacteriorhodopsin (M2). Another very promising
SEN30 5 high-resolution technique for distance determinations is the
SEN33 12 REDOR experiment. Schaefer and co-workers technique for
SEN36 7 distance determinations is the REDOR experiment. Schaefer
SEN36 3 and co-workers have used that technique to detect cross-links

TXT42
PAR150

12 in insect cuticle (M3).

PAR153

SEN03 1 Solid-state NMR spectroscopy has also been applied to
10 study dynamics in biomolecules including DNA duplex oli-
17 gomers (M4, M5), a DNA intercalation complex (M6), proteins
26 and peptides (M7-M9), and model cell membranes (M10).
SEN06 1 Principal components of chemical shift tensors in biomolecules
10 have been measured in an effort to obtain more structural
20 information than that afforded by isotropic shifts alone (M11,
29 M12).

PAR156

SEN03 1 Other work in solid-state NMR spectroscopy of biomolec-
9 ules has been covered in reviews on DNA (M13) and proteins
20 (M14).

TXT45**SOLID POLYMERS****SEN03 1****PAR159**

SEN03 1 Solid state NMR techniques such as MAS, wide-line ^2H ,
11 and relaxation studies are widely applied to characterize the
SEN06 2 morphology and dynamics of polymers. The structure of the
6 polymer is typically known, but this can be the main issue
17 in studies of resins and cross-linking.

PAR162

SEN03 1 Studies of local motions in polymers have been reviewed
SEN06 11 (N1). Harris has observed nuclear Overhauser enhancements
SEN09 11 in polymer films (N2). Tonelli has studied polymer chains
SEN12 7 confined to channels in clathrates (N3, N4). Spiess has ad-
4 vanced the use of 2-D NMR spectroscopy to study ultraslow
14 chain motion (N5).

PAR165

SEN03 1 Selected applications of NMR spectroscopy to solid poly-
9 mers are probably most usefully cited on the basis of class of
SEN06 21 material. Several studies by Maciel looked at the chemistry
SEN09 10 of resins (N6-N9). Diverse polymers of biological origin have
SEN12 8 been studied (N10-N14). Synthetic polymer classes examined
6 by NMR spectroscopy included polycarbonates (N15-N17),
12 polystyrenes (N18-N22), Nylons (N23, N24), PEEK (N25),
19 liquid crystalline polymers (N26, N27), and polyethylene
SEN15 26 (N28-N30). Reports were published on the study of con-
9 ductive polymers (N31-N33) and polyacetylenes (N34, N35).
SEN18 1 Urethanes have been investigated (N36, N37) as have com-
SEN21 3 posites (N38, N39) and a number of blends (N40-N47). The
3 effects of small molecules in polymers have been characterized
12 (N48-N50), and the ubiquitous technique of ^{129}Xe NMR
20 spectroscopy has been applied to polymers (N51, N52).

TXT48**SOLID INORGANIC COMPOUNDS****SEN03 1****PAR168**

SEN03 1 The application of solid state NMR spectroscopy to prob-
SEN06 10 lems in inorganic chemistry continues to expand. The di-
3 versity of elements and problems of interest in that field would
14 seem to present a challenge to the organization of the relevant
SEN09 25 NMR literature. Much of it, however, can be grouped into
SEN12 10 one of several categories. There were a number of studies
8 which related solid-state NMR and X-ray crystallographic
SEN15 15 data (O1-O9). Studies of stereochemical nonrigidity in solid
8 inorganic compounds have become almost as common as
SEN18 16 analogous studies in solutions (O10-O16). A number of studies
6 have been directed at measuring chemical shift tensors and/or
15 indirect spin-spin coupling tensors in inorganic compounds
SEN21 22 (O17-O23). Variable-temperature studies have probed mag-
6 netic properties (O24, O25), and an improvement in the res-
15 olution of spectra of paramagnetic lanthanide complexes has
SEN24 23 been reported (O26). Phosphorus multiple bonds have been
SEN27 7 characterized (O27, O28). NMR analysis continues to be
17 applied to the study of phase transitions of various sorts
(O29-O31).

PAR171

SEN03 1 Several other studies of interest did not fit into any easily
SEN06 13 contrived categories. The ^{127}I MAS spectrum of βAgI is very
10 sensitive to Ag^+ diffusion over a wide temperature range (O32).
SEN09 10 A single-crystal ^{23}Na study of the sodide $\text{Na}^+(\text{cryptand}$
SEN12 10 $[2.2.2])\cdot\text{Na}$ has been reported (O33). ^{199}Hg MAS NMR
5 spectroscopy has been used to probe coordination number in
14 Hg(II) complexes (O34), and ^{27}Al MAS has characterized
22 $\text{AlCl}_3\cdot\text{THF}$ complexes (O35).

TXT51**INORGANIC MATERIALS****SEN03 1****PAR174**

SEN03 1 Some of the more creative applications of NMR spectrosc-
10 opy to inorganic materials included Eckert's studies of
18 non-oxide glasses and related materials (P1-P10), Stebbins'
25 studies of silicates at extremes of temperature and pressure
34 (P11-P13), and the application of DAS and DOR to ^{17}O
SEN06 44 analysis of solid silicates (P14). MAS NMR analysis was also
SEN09 7 applied to silicates and aluminates (P15-P20). Other classes
4 of materials investigated included semiconductors (P21-P23),
SEN12 10 ceramics (P24-P28), and inorganic polymers (P29-P33). Clays
3 and other layered materials were also studied (P34-P43).
SEN15 1 High-resolution ^{27}Al spectra of small particles of AlF_3 were

TXT51
PAR174

11 obtained with line narrowing by Brownian motion in a liquid
21 phase (P44).

TXT54

SEN03 1
PAR177

FULLERENES

SEN03 1 The discovery and isolation of C_{60} , C_{70} , and a growing
12 number of other fullerenes is probably the most interesting
21 development in chemistry in recent memory, certainly since
SEN06 29 the discovery of high- T_c superconductors (see below). Early
3 success in fullerene research was controlled to a large extent
13 by access to materials, so it is not surprising that the first
25 NMR papers on C_{60} came from groups at the IBM Almaden
36 Research Center (Q1) and AT&T Bell Laboratories (Q2).
SEN09 1 Solid C_{60} undergoes rapid isotropic motion at room temper-
10 ature that averages the ^{13}C chemical shift anisotropy (Q1, Q2).
SEN12 1 This motion is quenched at 77 K, and it was possible to
14 measure principal components of the ^{13}C chemical shift tensor
SEN16 23 of 220, 186, and 40 ppm (Q1). These values compare favorably
SEN18 6 to a theoretical calculation (Q3). As mentioned in the In-
8 troduction and Scope, Yannoni and co-workers used the
14 Carr-Purcell sequence to measure bond lengths in ^{13}C -enriched
SEN21 22 C_{60} at 77 K (Q4). These values were 1.45 ± 0.015 and 1.40
10 ± 0.015 Å.

PAR180

SEN03 1 Fullerenes have proven to be far more reactive than was
12 originally believed, and a systematic chemistry is beginning
SEN06 20 to emerge. NMR studies of osmylated C_{60} (Q5) and poly-
SEN09 9 methylated C_{60} (Q6) have recently been reported. ESR studies
17 of C_{60}^- trapped in a zeolite (Q7) and the radical anion of C_{60}
(Q8) have also appeared.

TXT57

SEN03 1
PAR183HIGH- T_c SUPERCONDUCTORS

SEN03 1 NMR experiments are contributing to the understanding
9 of magnetic properties of high transition temperature ceramic
SEN06 17 superconductors. Studies of the $YBa_2Cu_3O_{6+x}$ series were
SEN09 8 reviewed by Walstedt and Warren (R1). A number of su-
5 perconductor materials have been investigated by ^{63}Cu and/or
13 ^{65}Cu (R2-R7), ^{17}O (R8-R11), ^{205}Tl (R12, R13), or ^{89}Y (R14-R16)
SEN12 23 spectroscopy. Relationships between theories of supercon-
6 ductivity and NMR experiments have also been explored
14 (R17-R19).

TXT60

SEN03 1
PAR186

GAS-PHASE NMR SPECTROSCOPY

SEN03 1 NMR studies of gases are generally geared toward under-
SEN06 10 standing the energetics of collisions. Gas-phase NMR studies
5 published during the period covered by this review included
14 studies related to conformational processes (S1, S2), nuclear
22 shielding (S3-S5), and relaxation phenomena (S6-S8).

TXT63

SEN03 1
PAR189

IMAGING, MICROSCOPY, AND DIFFUSION

SEN03 1 NMR imaging has had a profound effect on clinical med-
11 icine, and it is having an effect on chemical problems involving
SEN06 22 spatial inhomogeneities. One of the most intriguing examples
8 of this to catch my eye was the detection of chemical waves
20 in the Mn^{2+} -catalyzed Belousov-Zhabotinsky reaction (T1).
SEN09 1 Clinical aspects of imaging and spatially-localized spectroscopy
9 are outside the scope of this review, but the interested reader
SEN12 20 is referred to a good introductory article (T2). Some of the
5 most common applications of NMR imaging in chemistry and
14 engineering include diffusion of solvents in swollen polymers
22 (T3-T5) and oil and/or water in porous rock (T6-T8).

PAR192

SEN03 1

12 In the limit of smaller sample sizes and high spatial reso-
SEN06 21 lution, NMR imaging is frequently referred to as NMR mi-
8 croscopy (T9-T11). Since microscopy can be performed in
20 standard wide-bore (89-mm) magnets, it is likely to grow in
SEN09 18 popularity among chemists. Volume-localized spectroscopy
4 is important for slice selection in in vivo spectroscopy or other
15 problems requiring selective excitation of a spatial region near
SEN12 24 the coil (T12-T14). This can be possible even for solids
SEN15 9 (T15-T17). Solid-state NMR imaging has been reviewed
8 (T18), and several improvements or applications of the
SEN18 16 technique have been reported (T19-T26). In spite of the
15 apparent difficulty of imaging a rotating solid, the advantages
SEN21 15 of line narrowing have motivated study (T27-T29). NQR
3 imaging has also been reported (T30).

PAR195

SEN03 1

12 Field gradients are also used in NMR studies of diffusion,
SEN06 10 dating from the work of Stejskal and Tanner in the mid-60s.
SEN09 1 The availability of pulsed-field gradient coils has extended
6 the range of this method. Several applications of diffusion
SEN12 15 methods were reported (T31-T36) and others were cited in
4 the section on Adsorption Phenomena and Catalysis. The role
14 of diffusion in imaging studies should not be neglected (T37,
T38).

TXT66
PAR198

TXT66
PAR198

SEN00 1

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9 of papers on the structure, dynamics, and morphology of synthetic and natu-
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