

Preface

Researchers continue to find new applications for nuclear magnetic resonance (NMR) spectroscopy in the fields of physics, chemistry, material science, geology, biology, and medicine. As an impressive measure of the current scope of NMR research, one can find about 29,000 discrete references in the Web of Science (domains science and technology) in the year 2015 alone. The corresponding reference numbers in 2000 and 1990 are 20,000 and 9500, respectively. The portion of these studies focusing on solid-state NMR has increased, amounting to about 20% of all NMR studies since the year 2000. Presently, about 10% of all solid-state NMR studies, i.e. about 400 papers per year, deal with quadrupolar nuclei. Fewer than one-quarter of these, mainly ^2H studies, deal with integer spins; the remaining three-quarters or more address half-integer spins.

The study of quadrupole effects in the solid-state NMR of nuclei with half-integer spins began with the fundamental paper by R.V. Pound "Nuclear Electric Quadrupole Interactions in Crystals" [1], published in 1950; the early developments in this field are summarized in the 1957 review by M.H. Cohen and F. Reif, "Quadrupole Effects in NMR Studies of Solids" [2]. Jumping to the year 2012, we recommend the review by C. Fernandez and M. Pruski "Probing Quadrupolar Nuclei by Solid-State NMR Spectroscopy: Recent Advances" [3] and the book edited by R.E. Wasylishen *et al.*, "NMR of Quadrupolar Nuclei in Solid Materials" [4], which contains 28 chapters [5-32] written by 40 specialists in this field.

A few years ago we started to update our own review [33], originally published in 1993. It was titled "Quadrupole Effects in Solid-State NMR" and was limited to nuclei with half-integer spins in powder samples. The present review covers the identical topic; we again exclude integer spins. Some parts of the previous review [33] survived. This means that the current review is not free of self-plagiarism. The use of text parts and equations from our previous review [33] is mostly not indicated. We also re-used the basic content of some tables about quadrupole parameters of powder materials and some text from our (D.F.'s) 2000 review [34].

Tables about the ^{17}O , ^{23}Na and ^{27}Al parameters of inorganic powder materials are again supplemented, although it has become more difficult to keep up to date with the accelerating publication of relevant materials. A very useful source is the comprehensive and regularly updated compilation of quadrupole effects and their applications in solid-state NMR, presented by Pascal Man on his internet page www.pascal-man.com.

With the present work, we address the readership of our old review with many thanks for consulting it and with the hope that this new version will merit its recommendation to others. We would much appreciate any advice concerning mistakes or other deficiencies in our presentation, as well as any suggestion for extension. The present review is presented only on the Internet and will be casually updated. Please use, as its reference, D. Freude and J. Haase, www.quad-nmr.de.

References

- [1] R.V. Pound, Nuclear Electric Quadrupole Interactions in Crystals, *Phys. Rev.* 79 (1950) 685-702.
- [2] M.H. Cohen, F. Reif, Quadrupole Effects in NMR Studies of Solids, *Solid State Phys.* 5 (1957) 321-438.
- [3] C. Fernandez, M. Pruski, Probing Quadrupolar Nuclei by Solid-State NMR Spectroscopy: Recent Advances, *Top. Curr. Chem.* 306 (2012) 119-188.
- [4] R.E. Wasylishen, S.E. Ashbrook, S. Wimperis, *NMR of Quadrupolar Nuclei in Solid Materials*, Wiley, Chichester, 2012.

- [5] P.P. Man, Quadrupolar Interactions, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 3-16.
- [6] A.J. Vega, Quadrupolar Nuclei in Solids, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 17-44.
- [7] S.E. Ashbrook, S. Wimperis, Quadrupolar Coupling: An Introduction and Crystallographic Aspects, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 45-62.
- [8] D.L. Bryce, R.E. Wasylishen, Quadrupolar Nuclei in Solids: Influence of Different Interactions on Spectra, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 63-74.
- [9] R.W. Schurko, Acquisition of Wideline Solid-State NMR Spectra of Quadrupolar Nuclei, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 77-94.
- [10] T.T. Nakashima, R.E. Wasylishen, Sensitivity and Resolution Enhancement of Half-integer Quadrupolar Nuclei in Solid-state NMR, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 95-106.
- [11] A.P.M. Kentgens, Quadrupolar Nutation Spectroscopy, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 107-120.
- [12] P.I. Grandinetti, Dynamic Angle Spinning, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 121-132.
- [13] R. Dupree, Double Rotation (DOR) NMR, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 133-142.
- [14] J.-P. Amoureux, M. Pruski, MQMAS NMR: Experimental Strategies, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 143-162.
- [15] S.E. Ashbrook, S. Wimperis, STMAS NMR: Experimental Advances, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 163-178.
- [16] M. Deschamps, D. Massiot, Correlation Experiments Involving Half-integer Quadrupolar Nuclei, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 179-198.
- [17] J.W. Zwanziger, Computing Electric Field Gradient Tensors, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 199-209.
- [18] L.A. O'Dell, C.I. Ratcliffe, Quadrupolar NMR to Investigate Dynamics in Solid Materials, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 213-232.
- [19] G. Wu, Alkali Metal NMR of Biological Molecules, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 233-254.
- [20] L. Duma, Nitrogen-14 NMR Studies of Biological Systems, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 255-272.
- [21] G. Wu, Oxygen-17 NMR Studies of Organic and Biological Molecules, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 273-290.
- [22] S.E. Ashbrook, M.E. Smith, Oxygen-17 NMR of Inorganic Materials, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 291-320.

- [23] D.L. Bryce, C.M. Widdifield, R.P. Chapman, R.J. Attrell, Chlorine, Bromine, and Iodine Solid-State NMR, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 321-348.
- [24] F. Blanc, L. Speneer, G.R. Goward, Quadrupolar NMR of Ionic Conductors, Batteries, and other Energy-Related Materials, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 349-370.
- [25] M. Haouas, C. Martineau, F. Taulelle, Quadrupolar NMR of Nanoporous Materials, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 371-386.
- [26] J.F. Stebbins, Quadrupolar NMR in the Earth Sciences, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 387-400.
- [27] N.J. Curro, Quadrupolar NMR of Superconductors, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 401-416.
- [28] J.P. Yesinowski, Quadrupolar NMR of Semiconductors, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 417-438.
- [29] T. Polenova, A.S. Lipton, P.D. Ellis, Quadrupolar NMR of Metal Nuclides in Biological Materials, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 439-452.
- [30] S. Kroeker, Nuclear Waste Glasses: Insights from Solid-State NMR, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 453-466.
- [31] O.B. Lapina, V.V. Terskikh, Quadrupolar Metal NMR of Oxide Materials Including Catalysts, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 457-494.
- [32] F. Haarmann, Quadrupolar NMR of Intermetallic Compounds, in: R.E. Wasylishen, S.E. Ashbrook, S. Wimperis (Eds.) NMR of Quadrupolar Nuclei in Solid Materials, Wiley, Chichester, 2012, pp. 495-509.
- [33] D. Freude, J. Haase, Quadrupole Effects in Solid-State NMR, NMR Basic Principles and Progress 29 (1993) 1-90.
- [34] D. Freude, Quadrupole Nuclei in Solid-state NMR, in: R.A. Meyers (Ed.) Encyclopedia of Analytical Chemistry, John Wiley & Sons, Chichester, 2000, pp. 12188-12224.