

246

Topics in Current Chemistry

Editorial Board:

A. de Meijere · K. N. Houk · H. Kessler

J.-M. Lehn · S. V. Ley · S. L. Schreiber · J. Thiem

B. M. Trost · F. Vögtle · H. Yamamoto

Topics in Current Chemistry

Recently Published and Forthcoming Volumes

Anion Sensing

Volume Editor: Stibor, I.
Vol. 255, 2005

Organic Solid State Reactions

Volume Editor: Toda, F.
Vol. 254, 2005

DNA Binders and Related Subjects

Volume Editors: Waring, M.J., Chaires, J.B.
Vol. 253, 2005

Contrast Agents III

Volume Editor: Werner Krause
Vol. 252, 2005

Chalcogenocarboxylic Acid Derivatives

Volume Editor: Kato, S.
Vol. 251, 2005

New Aspects in Phosphorus Chemistry V

Volume Editor: Majoral, J.-P.
Vol. 250, 2005

Templates in Chemistry I

Volume Editors: Schalley, C.A., Vögtle, F.,
Dötz, K.-H.
Vol. 248, 2005

Collagen

Volume Editors: Brinckmann, J.,
Notbohm, H., Müller, P.K.
Vol. 247, 2005

New Techniques in Solid-State NMR

Volume Editor: Klinowski, J.
Vol. 246, 2005

Functional Molecular Nanostructures

Volume Editor: Schlüter, A.D.
Vol. 245, 2004

Natural Product Synthesis II

Volume Editor: Mulzer, J.H.
Vol. 244, 2004

Natural Product Synthesis I

Volume Editor: Mulzer, J.H.
Vol. 243, 2004

Immobilized Catalysts

Volume Editor: Kirschning, A.
Vol. 242, 2004

Transition Metal and Rare Earth Compounds III

Volume Editor: Yersin, H.
Vol. 241, 2004

The Chemistry of Pheromones and Other Semiochemicals II

Volume Editor: Schulz, S.
Vol. 240, 2004

The Chemistry of Pheromones and Other Semiochemicals I

Volume Editor: Schulz, S.
Vol. 239, 2004

Orotidine Monophosphate Decarboxylase

Volume Editors: Lee, J.K., Tantillo, D.J.
Vol. 238, 2004

Long-Range Charge Transfer in DNA II

Volume Editor: Schuster, G.B.
Vol. 237, 2004

Long-Range Charge Transfer in DNA I

Volume Editor: Schuster, G.B.
Vol. 236, 2004

Spin Crossover in Transition Metal Compounds III

Volume Editors: Gütllich, P., Goodwin, H.A.
Vol. 235, 2004

Spin Crossover in Transition Metal Compounds II

Volume Editors: Gütllich, P., Goodwin, H.A.
Vol. 234, 2004

Spin Crossover in Transition Metal Compounds I

Volume Editors: Gütllich, P., Goodwin, H.A.
Vol. 233, 2004

New Techniques in Solid-State NMR

Volume Editor: Jacek Klinowski

With contributions by

O. N. Antzutkin · T. Anupöld · H. Eckert · S. Elbers · J. D. Epping ·
C. Fernandez · I. Heinmaa · J. W. Hennel · A. V. Ivanov · M. Janssen ·
M. Kalwei · S. Kazmierski · J. Klinowski · W. Kolodziejski ·
P. K. Madhu · C. M. Morais · J. Past · M. J. Potrzebowski · A. Reinhold ·
J. Rocha · A. Samoson · W. Strojek · T. Tuherm · E. Vinogradov ·
S. Vega · U. Voigt

The series *Topics in Current Chemistry* presents critical reviews of the present and future trends in modern chemical research. The scope of coverage includes all areas of chemical science including the interfaces with related disciplines such as biology, medicine and materials science. The goal of each thematic volume is to give the nonspecialist reader, whether at the university or in industry, a comprehensive overview of an area where new insights are emerging that are of interest to a larger scientific audience.

As a rule, contributions are specially commissioned. The editors and publishers will, however, always be pleased to receive suggestions and supplementary information. Papers are accepted for *Topics in Current Chemistry* in English.

In references *Topics in Current Chemistry* is abbreviated Top Curr Chem and is cited as a journal.

Visit the TCC content at <http://www.springerlink.com/>

Library of Congress Control Number: 2004109923

ISSN 0340-1022

ISBN 3-540-22168-9

DOI 10.1007/b94544

Springer Berlin Heidelberg New York

This work is subject to copyright. All rights are reserved, whether the whole or part of the material is concerned, specifically the rights of translation, reprinting, reuse of illustrations, recitation, broadcasting, reproduction on microfilms or in any other ways, and storage in data banks. Duplication of this publication or parts thereof is only permitted under the provisions of the German Copyright Law of September 9, 1965, in its current version, and permission for use must always be obtained from Springer-Verlag. Violations are liable to prosecution under the German Copyright Law.

Springer is a part of Springer Science+Business Media
springeronline.com

© Springer-Verlag Berlin Heidelberg 2005
Printed in Germany

The use of general descriptive names, registered names, trademarks, etc. in this publication does not imply, even in the absence of a specific statement, that such names are exempt from the relevant protective laws and regulations and therefore free for general use.

Cover design: KünkelLopka, Heidelberg/design & production GmbH, Heidelberg
Typesetting: Fotosatz-Service Köhler GmbH, Würzburg

Printed on acid-free paper 02/3020 xv - 5 4 3 2 1 0

Volume Editor

Professor
Jacek Klinowski
Department of Chemistry
University of Cambridge
Lensfield Road
Cambridge CB2 1EW, Great Britain
jk18@cam.ac.uk

Editorial Board

Prof. Dr. Armin de Meijere
Institut für Organische Chemie
der Georg-August-Universität
Tammannstraße 2
37077 Göttingen, Germany
ameijer1@uni-goettingen.de

Prof. Dr. Horst Kessler
Institut für Organische Chemie
TU München
Lichtenbergstraße 4
85747 Garching, Germany
kessler@ch.tum.de

Prof. Steven V. Ley
University Chemical Laboratory
Lensfield Road
Cambridge CB2 1EW, Great Britain
svl1000@cus.cam.ac.uk

Prof. Dr. Joachim Thiem
Institut für Organische Chemie
Universität Hamburg
Martin-Luther-King-Platz 6
20146 Hamburg, Germany
thiem@chemie.uni-hamburg.de

Prof. Dr. Fritz Vögtle
Kekulé-Institut für Organische Chemie
und Biochemie der Universität Bonn
Gerhard-Domagk-Straße 1
53121 Bonn, Germany
voegtle@uni-bonn.de

Prof. K.N. Houk
Department of Chemistry and Biochemistry
University of California
405 Hilgard Avenue
Los Angeles, CA 90024-1589, USA
houk@chem.ucla.edu

Prof. Jean-Marie Lehn
Institut de Chimie
Université de Strasbourg
1 rue Blaise Pascal, B.P.Z. 296/R8
67008 Strasbourg Cedex, France
lehn@chimie.u-strasbg.fr

Prof. Stuart L. Schreiber
Chemical Laboratories
Harvard University
12 Oxford Street
Cambridge, MA 02138-2902, USA
sls@slsiris.harvard.edu

Prof. Barry M. Trost
Department of Chemistry
Stanford University
Stanford, CA 94305-5080, USA
bmtrost@leland.stanford.edu

Prof. Hisashi Yamamoto
Arthur Holly Compton Distinguished
Professor
Department of Chemistry
The University of Chicago
5735 South Ellis Avenue
Chicago, IL 60637
773-702-5059, USA
yamamoto@uchicago.edu

Topics in Current Chemistry also Available Electronically

For all customers who have a standing order to Topics in Current Chemistry, we offer the electronic version via SpringerLink free of charge. Please contact your librarian who can receive a password for free access to the full articles by registration at:

springerlink.com

If you do not have a subscription, you can still view the tables of contents of the volumes and the abstract of each article by going to the SpringerLink Homepage, clicking on "Browse by Online Libraries", then "Chemical Sciences", and finally choose Topics in Current Chemistry.

You will find information about the

- Editorial Board
- Aims and Scope
- Instructions for Authors
- Sample Contribution

at <http://www.springeronline.com> using the search function.

Preface

After the discovery of nuclear magnetic resonance, [1, 2] the new spectroscopy was used for the study of ^1H nuclei in liquids, but then the signal from copper in the receiver coil itself, the first observation of NMR in the solid state, was found. “Wide-line NMR”, named thus because of the line-broadening effects of dipolar interaction and chemical shift anisotropy, was not far behind, and soon led to significant advances through the analysis of spectral lineshapes. In this way Richards and Smith [3] demonstrated the presence of H_3O^+ cations in solid hydrates of strong acids, while Andrew and Eades [4] investigated the details of molecular motion in three solid benzenes. Even now, 50 years later, it is difficult to think of a technique which would provide a more convincing demonstration of the reality of these effects.

The development of magic angle spinning (MAS)[5, 6] initiated a new era in the structural study of solids, greatly enhancing our knowledge of a wide range of materials encountered in chemistry, physics, biology, earth sciences and technology. The introduction of Fourier-transform NMR, [7] cross-polarization [8] and high-field superconducting magnets, improved the sensitivity of the spectra. MAS was combined with cross-polarization for ^{13}C NMR of organics, [9] and this approach greatly contributed to the study of ^{29}Si and ^{27}Al in molecular sieve catalysts, minerals and many other materials. High-resolution spectra of solids are now routinely obtained using a combination of MAS and cross-polarization. However, MAS does not eliminate the quadrupolar interaction, and since 74% of all magnetically active naturally occurring isotopes are quadrupolar, the main effort in solid-state NMR is now directed towards methods for the observation of high-resolution spectra of such nuclei.

Springer, the publishers of this volume, originally asked me to edit a book on advances in magic-angle spinning. Aware that, while there have been significant advances in MAS itself, strategies developed for increasing the resolution of NMR spectra almost invariably combine MAS with various radiofrequency pulse trains, I invited several leading modern practitioners of solid-state NMR to describe their recent work. I am grateful to them for their contributions, and I hope that the volume will be of interest to readers in various fields involving the physics and chemistry of solids.

-
- [1] Purcell, E. M.; Torrey, H. C.; Pound, R. V. *Phys. Rev.* **1946**, *69*, 37.
 - [2] Bloch, F.; Hansen, W. W.; Packard, M. *Phys. Rev.* **1946**, *69*, 127.
 - [3] Richards, R. E.; Smith, J. A. S. *Trans. Faraday Soc.* **1951**, *47*, 1261.
 - [4] Andrew, E. R.; Eades, R. G. *Proc. Royal Soc. London* **1953**, *A218*, 537.
 - [5] Andrew, E. R.; Bradbury, A.; Eades, R. G. *Nature* **1959**, *183*, 1802.
 - [6] Lowe, I. J.; Norberg, R. E. *Phys. Rev.* **1957**, *107*, 46.
 - [7] Ernst, R. R.; Anderson, W. A. *Rev. Sci. Inst.* **1966**, *37*, 93.
 - [8] Pines, A.; Gibby, M. G.; Waugh, J. S. J. *Chem. Phys.* **1972**, *56*, 1776.
 - [9] Schaefer, J.; Stejskal, E. O. *J. Am. Chem. Soc.* **1976**, *98*, 1031.

Contents

| | |
|--|-----|
| Magic-Angle Spinning: a Historical Perspective J. W. Hennel · J. Klinowski | 1 |
| New Horizons for Magic-Angle Spinning NMR A. Samoson · T. Tuherm · J. Past · A. Reinhold · T. Anupöld · I. Heinmaa . . . | 15 |
| Strategies for High-Resolution Proton Spectroscopy in Solid-State NMR E. Vinogradov · P. K. Madhu · S. Vega | 33 |
| High-Resolution Solid-State NMR Studies of Inclusion Complexes M. J. Potrzebowski · S. Kazmierski | 91 |
| Progress in Multiple-Quantum Magic-Angle Spinning NMR Spectroscopy J. Rocha · C. M. Morais · C. Fernandez | 141 |
| Dipolar Solid State NMR Approaches Towards Medium-Range Structure in Oxide Glasses H. Eckert · S. Elbers · J. D. Epping · M. Janssen · M. Kalwei · W. Strojek U. Voigt | 195 |
| Solid-State NMR Studies of Bone W. Kolodziejski | 235 |
| Natural Abundance ^{15}N and ^{13}C CP/MAS NMR of Dialkyldithiocarbamate Compounds with Ni(II) and Zn(II) A. V. Ivanov · O. N. Antzutkin | 271 |
| Author Index Volumes 201–246 | 339 |
| Subject Index | 355 |

Contents of volume 225

Modern Mass Spectrometry

Volume Editor: Christoph A. Schalley

ISBN 3-540-00098-4

I Reactivity

Physical Organic Chemistry of the Gas Phase. Reactivity Trends for Organic Cations

E. Uggerud

Mass Spectrometric Approaches to Interstellar Chemistry

S. Petrie · D. K. Bohme

Transient Intermediates of Chemical Reactions by Neutralization-Reionization Mass Spectrometry

F. Tureček

II Metalorganic Chemistry

Diastereoselective Effects in Gas-Phase Ion Chemistry

D. Schröder · H. Schwarz

Metalorganic Chemistry in the Gas Phase: Insight into Catalysis

D. A. Plattner

III Mass Spectrometric Methodology

Gas-Phase Conformations: The Ion Mobility/Ion Chromatography Method

T. Wyttenbach · M. T. Bowers

Threshold Collision-Induced Dissociations for the Determination of Accurate Gas-Phase Binding Energies and Reaction Barriers

P. B. Armentrout

IV Medicinal Chemistry

Investigating Viral Proteins and Intact Viruses with Mass Spectrometry

S. A. Trauger · T. Junker · G. Siuzdak

High-Throughput Mass Spectrometry for Compound Characterization in Drug Discovery

M. Brönstrup