Aqueous-Phase Organometallic Catalysis

Concepts and Applications

Edited by
Boy Cornils and Wolfgang A. Herrmann

Second, Completely Revised and Enlarged Edition

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Editors

Prof. Dr. Boy Cornils
Kirschgartenstraße 6
65719 Hofheim
Germany

Prof. Dr. Dr. h.c. mult. Wolfgang A. Herrmann
Anorganisch-Chemisches Institut
der Technischen Universität München
Lichtenbergstraße 4
85747 Garching
Germany

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Preface to the Second Edition

Very recently, some colleagues noticed a statement in one of the monthly columns about the state-of-the-art that "in organic chemistry reactions employing the solvent water are still rare [1]" – which is true and untrue at the same time: certainly, water-based conversions are scarce compared to those in the great majority of other solvents. But whoever was sensitive enough to recognize the tremendous possibilities of aqueous-phase catalysis would never ignore the increasing number of publications concerning this field of activity, the progress which has taken place during recent years, and the breakthroughs which have been brought about following these activities.

So it is a great pleasure for us to announce the second edition of our book *Aqueous-Phase Organometallic Catalysis* – in such close proximity to the first edition in 1998. Responsible for this are on the one hand the dramatic successes of industrial realization: production figures are now close to 1 MM tons per year in various applications (with hydroformylations, at approximately 800 000 tpy, in a senior position). On the other hand, the long overdue in-depth occupation with the scientific basis, the exploratory work with the various possibilities of this "immobilization with the liquid support water", and the exploitation of the immense variability of the method in chemical respects (regarding central atoms and ligands) create an atmosphere of overwhelming interest in this technique.

Thus the sections of this revised edition have been enlarged to different extents. For instance, in respect of the scientific fundamentals and taking into account that the role of water in organometallic conversions is not only purely as a solvent but as a strongly coordinative polar reagent. It contributes considerably to the formation of polar or ionic intermediates or to oxidative additions to lower-valent transition metal complexes (thus explaining the strong pH dependence of many aqueous-phase catalyzed reactions). The work on different central atoms of catalytically active complexes and the search for alternative, highly specialized ligands – including chiral ones – has extended considerably the scope of aqueous-phase organometallic catalysis together with knowledge about coordination catalysis.

The same is true for the application of water-soluble catalysts for quite a bunch of basic organometal-derived chemical reactions ranging from hydrogenations or hydroformylations to more "exotic" applications such as water-based enantioselective Diels–Alder reactions or kinetic resolutions. It also includes improvements and alternative answers for the chemical reaction engineering of aqueous-phase catalyzed conversions.

Last but not least, the success of aqueous-phase catalysis has drawn the interest of the homogeneous-catalysis community to other biphasic possibilities such as organic/organic separations, fluorous phases, nonaqueous ionic liquids, supercritical solvents, amphiphilic compounds, or water-soluble, polymer-bound catalysts. As in the field of aqueous-phase catalysis, the first textbooks on these developments have been published, not to mention Joó's book on *Aqueous Organometallic Catalysis* which followed three years after our own publication and which put the spotlight on Joó's special merits as one of the pioneers in aqueous biphasic catalysis. Up to now, most of the alternatives mentioned are only in a state of intensive development (except for one industrial realization: that of Swan/Chematur for hydrogenations in $scCO_2$ [2]) but other pilot plant adaptations and even technical operations may be expected in the near future.

This second edition is based mainly on the state-of-the-art as described in the published literature up to the year 2003. To make things easier and to avoid errors, parts of the second edition are revised and updated, rather than rewritten. Thus, in some cases the order of the references is unchanged and new references are added without renumbering the existing ones (or substitute existing refs. by new items). The numbering of structures, equations, etc., was changed if necessary.

Once more we have to express our thanks not only to the authors and coauthors of the volume but also to the team at Wiley-VCH at Weinheim, especially Mrs. Claudia Grössl, for the production and their endless patience, and Dr. Elke Maase, the publishing editor. As with all our books, Mrs. Diana Boatman from Redhill, Surrey (UK), served as freelance copy-editor and was an invaluable help during the difficult process of completion. The Munich research group, especially PD Dr. F. E. Kühn, is acknowledged for scientific and technical assistance.

Hofheim and München
January 2004

Boy Cornils
Wolfgang A. Herrmann

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[1] *Nachr. Chem.* **2003**, *51*(5), 516.
[2] B. Cornils, W. A. Herrmann, R. Schlögl, C.-H. Wong, *Catalysis from A to Z*, 2nd Edition, Wiley-VCH, Weinheim, **2003**, p. 746.

Preface to the First Edition

This book describes homogeneously catalyzed reactions under two major boundary conditions: the catalysts employed are *organometallic* complexes that are used in the *aqueous phase*. In this respect the book is restricted to one area of homogeneous catalysis and therefore – though substantially expanded and more detailed – to one special area of our previous book, *Applied Homogeneous Catalysis with Organometallic Complexes* (VCH, Weinheim, Germany, 1996).

The subject of the book is the use of water-soluble organometallic catalysts for chemical reactions. These catalysts are so far the sole successful means of implementing the idea of *heterogenization* of homogeneous catalysts by *immobilizing* them with the aid of liquid supports. They thus solve the cardinal problem of homogeneous catalysis, which lies in the expensive separation of catalyst as well as product that is inherent in the system: the catalyst used in the homogeneous phase is separated by simply decanting the aqueous catalyst phase from the organic phase of the substrates and reaction products. Since all attempts to heterogenize homogeneous catalysts by immobilizing them on solid supports ("anchoring") have to varying degrees been unsuccessful, only the use of homogeneous catalysts in aqueous solution and thus on liquid supports ("biphase operation") leads to a neat, inexpensive solution to the problem that conserves resources and is therefore environmentally friendly.

This book is restricted essentially to *aqueous*-phase catalyses and thus to one area of the more comprehensively defined two-phase catalyses. This restriction to the most recent and successful development of homogeneous catalysis takes account of the rapid technical advances in the process concept first described by Manassen et al. in 1973, which was followed in rapid succession in the 1970s by hesitant basic work and in 1984 by the first commercial implementation. This unusual sequence – industrial implementation in a 100 000 tonnes per year oxo plant for the hydroformylation of propylene **before** years of time-consuming basic research to determine mechanistic, kinetic and other data – demonstrates clearly the great leap forward that this process development represented in the field of homogeneous catalysis and in solving the central problem mentioned earlier. Since then other processes employing homogeneous catalysis have been converted to an aqueous two-phase procedure.

The development work intensified worldwide in various research groups in the years following the first commercial implementation at Ruhrchemie AG in Oberhausen. The obvious course of action was to let colleagues and specialists themselves report on their developments. This led to the formation of the international circle of contributors from the USA, France, the United Kingdom, China, Italy, Japan, India, Hungary and Germany which gives first-hand reports on its work.

One focus of the book is the hydroformylation process, the process involved in the first commercial implementation of aqueous-phase catalysis with its detailed descriptions of fundamental laws, special process features, and the present state of the art. Further focal points of the book are basic research on the complex catalysts (central atoms, ligands) and on the influence of the reaction conditions, solvents, and co-solvents, and a survey of other aqueous two-phase concepts and of proposed applications, with experimental examples and details. Environmental aspects are also considered.

We are sure that the outline chosen and the wide range of contributions from the authors give a multifaced and informative picture of the present state of developments in the field of aqueous two-phase catalysis, which presents not only the principles and accounts of the latest applications but also many aspects of spin-offs and alternative processes.

This description of ideas and process developments appears to us to be highly important for an appreciation of the potential of aqueous biphase catalysis. The familiar assessment of the most important aspects of heterogeneous and homogeneous catalysis demonstrates that only in a solution of the problem of continuous separation of catalyst and product, such as becomes possible with the processes involving aqueous immobilized catalysts, in the key to further progress found. Only *homogeneous* catalysts that can be handled without problems will give us scientists and developers confidence that the clear and sure mechanistic understanding of their mode of action and the possibility of easy variability of steric and/or electronic properties can be transferred to other immobilized, and thus easy-to-handle, catalysts. More optimistically, it is hoped that this will apply especially to those heterogenized catalysts that basically are derived from tailor-made homogeneous catalysts.

The sharp line of demarcation between homogeneous and heterogeneous catalysis would thus be blurred and the possibility opened up of combining in one species the advantages of homogeneous catalysts and none of the disadvantages of heterogeneous catalysts: heterogenized homogeneous catalysts would lead to equally advantageous results as homogenized heterogeneous catalysts – the long-awaited dream of catalysis research would be fulfilled!

We thank the team at WILEY-VCH, especially Mrs. Diana Boatman, Dr. Anette Eckerle, and Mrs. Claudia Grössl for their cooperation during preparation of this book and for helpful technical assistance.

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Frankfurt-Höchst and München — Boy Cornils
Spring, 1998 — Wolfgang A. Herrmann

Contents

Contributors

Prof. Dr. Jacques Augé
Université de Cergy-Pontoise
5, mail Gay-Lussac
Neuville-sur-Oise
F-95031 Cergy-Pontoise/France
Tel: + 33/1 342 57051
Fax: + 33/1 342 57067
E-mail: jacques.auge@chim.u-cergy.fr

Prof. Dr. Manfred Baerns
Institut für Angewandte Chemie
Berlin-Adlershof e. V.
Rudower Chaussee 5
D-12484 Berlin/Germany
Tel: + 49/30 6392 4444
Fax: + 49/30 6392 4454
E-mail: mbaerns@aol.com

Dr. Helmut Bahrmann
Celanese GmbH/Werk Ruhrchemie
Postfach 13 01 60
D-46128 Oberhausen/Germany
Tel: + 49/208 693 2201
Fax: + 49/208 693 2291

Prof. Dr. Arno Behr
Universität Dortmund
Fachbereich Chemietechnik
Lehrstuhl für Technische Chemie A
Emil-Figge-Str. 66
D-44227 Dortmund/Germany
Tel: + 49/231 755 2310
Fax: + 49/231 755 2311
E-mail: arno.behr@bci.uni-dortmund.de

Prof. Dr. Matthias Beller
Leibnitz-Institut für Organische Katalyse
an der Universität Rostock
Buchbinderstr. 5–6
D-18055 Rostock/Germany
Tel: +49/381 466 9313
Fax: +49/381 466 9324
E-mail: Matthias.Beller@ifok.uni-rostock.de

Dr. Bhalchandra M. Bhanage
Division of Materials Science and Engineering
Hokkaido University
Kita 13 Nishi 8, Kita-ku
Sapporo 060-8628/Japan
Tel: +81/11706 6597
Fax: +81/11706 6594
E-Mail: bhanage@proc-ms.eng.hokudai.ac.jp

Dr. Claudio Bianchini
Istituto per lo Studio della Stereochimica
ed Energetica dei Composti
CNR di Coordinazione
Via J. Nardi, 39
I-50132 Firenze/Italy
Tel: + 39/55 243 990/24 5990
Fax: + 39/55 247 8366
E-mail: bianchini@fi.cnr.it

Dr. Sandra Bogdanovic
Booz Allen Hamilton
Lenbachplatz 3
D-80333 München/Germany
Tel.: +49/89 545 250
Fax.: +49/89 545 25 500

Prof. Dr. Armin Börner
Universität Rostock
Fachbereich Chemie
Albert-Einstein-Str. 3a
D-18059 Rostock/Germany
Tel.: +49/381 466 6931/350
Fax: +49/381 466 9324
E-mail: armin.boerner@ifok.uni-rostock.de

Prof. Dr. Mario Bressan
Universitá G. D'Annunzio
Dipartimento di Scienze
Viale Pindaro 42
I-65127 Pescara/Italy
Tel: + 39/85 453 7548
Fax: + 39/85 453 7545
E-mail: bressan@sci.unich.it

Dr. Henry R. Bryndza
DuPont Nylon
Experimental Station, Bldg. 302
P.O. Box 80328
Wilmington, Delaware 19880-0302/USA
Tel: + 1/302 695 3761
Fax: + 1/302 695 9084
E-mail: Bryndza@esvax.enet.duPont.com

Prof. Dr. Raghunath V. Chaudhari
National Chemical Laboratory
Pune 411 008/India
Tel: + 91/2025 893 163
Fax: + 91/2025 893 260
E-mail: rvc@ems.ncl.res.in

Prof. Dr. Peter Claus
Technische Universität Darmstadt
Institut für Technische Chemie und
Makromolekulare Chemie
Petersenstr. 20
D-64287 Darmstadt/Germany
Tel: +49/6151 16 5369
Fax: +49/6151 16 4788
E-mail: claus@ct.chemie.tu-darmstadt.de

Prof. Dr. Boy Cornils
Kirschgartenstr. 6
D-65719 Hofheim/Germany
Tel: +49/6192 23502
Fax: +49/6192 23502
E-mail: boy.cornils@t-online.de

Prof. Donald Darensbourg
Texas A&M University
Department of Chemistry
PO Box 30012
College Station, Texas 77892-3012/USA
Tel: +1/979 845 5417
Fax: +1/979 845 0158
E-mail: djdarens@mail.chem.tamu.edu

Prof. Dr. Mark E. Davis
Chemical Engineering
California Institute of Technology
Pasadena, California 91125/USA
Tel: + 1/626 395 4251
Fax: + 1/626 568 8143
E-mail: mdavis@cheme.caltech.edu

Prof. Dr. Eckehard V. Dehmlow
Universität Bielefeld
Fakultät für Chemie
Universitätsstr. 25
D-33615 Bielefeld/Germany
Tel: + 49/521 106 2051
Fax: + 49/521 106 6146
E-mail: Dehmlow@post.uni-bielefeld.de

Dipl.-Chem. Robert W. Eckl
morphochem AG
Gmundener Str. 37–37a
D-81379 München/Germany
E-mail: robert.eckl@morphochem.de

Dr. Carl D. Frohning
Celanese GmbH/Werk Ruhrchemie
Postfach 13 01 60
D-46128 Oberhausen/Germany
Tel: + 49/208 693 2419
Fax: + 49/208 693 2291
E-mail: Frohning@cityweb.de

Prof. Dr. Robert H. Grubbs
Division of Chemistry
and Chemical Engineering
California Institute of Technology
Pasadena, California 91125/USA
Tel: + 1/626 395 6003
Fax: + 1/626 564 9297
E-mail: rhg@its.caltech.edu

Prof. Dr. Brian E. Hanson
Virginia Polytechnic Institute
and State University
Department of Chemistry
College of Arts and Sciences
Blacksburg, Virginia 24061-0212/USA
Tel: + 1/540 231 7206
Fax: + 1/540 231 3255
E-mail: hanson@vt.edu

Dr. John A. Harrelson, Jr.
DuPont Nylon
Experimental Station, Bldg. 302
P.O. Box 80328
Wilmington, Delaware 19880-0302/USA
Tel: + 1/302 695 3761
Fax: + 1/302 695 9084

PD Dr. Peter Härter
Technische Universität München
Anorganisch-chemisches Institut
Lichtenbergstr. 4
D-85747 Garching/Germany
Tel: +49/89 2891 3099
Fax: +49/89 2891 3473
E-mail: peter.haerter@ch.tum.de

Prof. Dr. Wolfgang A. Herrmann
President of the
Technische Universität München
Arcisstr. 21
D-80333 München/Germany
Tel: + 49/89 2892 2200
Fax: + 49/89 2892 3399
E-mail: sekretariat.ac@ch.tum.de

Dr. Dietmar Hoff
Rheinchemie
Paul-Ehrlich-Str. 10
D-67122 Altrip/Germany
E-mail: dietmar.hoff@rheinchemie.com

Prof. Dr. István T. Horváth
Eötvös University
Department of Chemical Technoloy
and Environmental Chemistry
Pázmány Péter sétány 1/A
H-1117 Budapest/Hungary
Tel: +31/1 2090590
Fax: +31/1 2090607
E-mail: istvan.t.horvath@hit-team.net

Prof. Dr. Zilin Jin
State Key Laboratory of Fine Chemicals
Dalian University of Technology
116012 Dalian/China
Tel: + 86/411 467 1511
Fax: + 86/411 363 3080
E-mail: hpcuo@mail.dlptt.ln.cn

Prof. Dr. Ferenc Joó
University of Debrecen and
Hungarian Academy of Sciences
P.O. Box 7
H-4010 Debrecen/Hungary
Tel: +36/52 512 900
Fax: +36/52 512 915
E-mail: jooferenc@tigris.klte.hu

Prof. Dr. Philippe Kalck
Ecole Nationale Supérieure
des Ingénieurs en Arts Chimiques
et Technologique (ENSIACET)
118, Route de Narbonne
F-31077 Toulouse Cedex 04/France
Tel: +33/562 885 690
Fax: +33/562 885 600
E-mail: Philippe.Kalck@ensiacet.fr

Dr. Paul C. J. Kamer
University of Amsterdam
Institute for Molecular Chemistry
Inorganic Chemistry & Homogeneous
Catalysis
Nieuwe Achtergracht 166
NL-1018 WV Amsterdam
The Netherlands
Tel: + 31/20 525 6495/6454
Fax: + 31/20 525 6456

Dr. Agnes Kathó
University of Debrecen and
Hungarian Academy of Sciences
P.O. Box 7
H-4010 Debrecen/Hungary
Tel: +36/52 512 900
Fax: +36/52 512 915
E-mail: katho@tigris.klte.hu

Prof. Dr. Shu Kobayashi
Graduate School of Pharmaceutical
Sciences
The University of Tokyo
Hongo, Bunkyo-ku
Tokyo 113-0033/Japan
E-Mail: skobayas@mol.f-u-tokyo.ac.jp

Dr. Christian W. Kohlpaintner
Chemische Fabrik Budenheim
Rheinstr. 27
D-55257 Budenheim/Germany
Tel: +49/6139 89495
Fax: +49/6139 89464
E-mail: ckohlpaintner@budenheim-cfb.com

Dr. Jürgen G.E. Krauter
Degussa AG
Rodenbacher Chaussee 4
D-63457 Hanau-Wolfgang/Germany
E-mail: juergen.krauter@degussa.com

PD Dr. Fritz E. Kühn
Technische Universität München
Anorganisch-chemisches Institut
Lichtenbergstr. 4
D-85747 Garching/Germany
Tel: + 49/89 2891 3105
Fax: + 49/89 2891 3473
E-mail: fritz.kuehn@chem.tum.de

Dr. Emile G. Kuntz
CPE-LYON – Laboratoire
d'Electrochimie Analytique
43, Bd du 11 Novembre 1918
F-69616 Villeurbanne/France
Tel: + 33/472 448 478
Fax: + 33/472 448 479

Prof. Dr. Gábor Laurenczy
Institut de Chimie Minerale et
Analytique
Batiment de Chimie (BCH)
CH-1015 Dorigny Lausanne/Suisse
Tel: +41/21693 9858
Fax: +41/21693 9865
E-mail: gabor.laurenczy@epfl.ch

Prof. Dr. Piet W. N. M. van Leeuwen
University of Amsterdam
Institute for Molecular Chemistry
Inorganic Chemistry & Homogeneous
Catalysis
Nieuwe Achtergracht 166
NL-1018 WV Amsterdam
The Netherlands
Tel: + 31/20 525 5419
Fax: +31/20 525 6422
E-mail: pwnm@anorg.chem.uva.nl

Prof. Dr. Walter Leitner
Lehrstuhl für Technische Chemie
und Petrolchemie
RWTH-Aachen
Worringer Weg 1
D-52056 Aachen/Germany
Tel: +49/241 802 6480
Fax: +49/241 802 2177
E-mail: leitner@itmc.rwth-aachen.de

Prof. Dr. André Lubineau
Laboratoire de Chimie Organique
Multifonctionnelle
Université de Paris-Sud
Bat. 420
F-91405 Orsay/France
Tel: + 33/169 157 233
Fax: + 33/169 154 715
E-mail: lubin@icmo.u-psud.fr

Dr. David M. Lynn
Laboratories of Chemistry
California Institute of Technology
Pasadena, California 91125/USA
Tel: + 1/626 395 6003
Fax: + 1/626 564 9297

PD Dr. Stefan Mecking
Albert-Ludwigs-Universität
Institut für Makromolekulare Chemie
Stefan-Meier-Str. 31
D-79104 Freiburg/Germany
Tel: +49/761 203 6304
Fax: +49/761 203 6319
E-mail: stefan.mecking@makro.uni-freiburg.de

Dr. Andrea Meli
Istituto per lo Studio della Stereochimica
ed Energetica dei Composti
CNR di Coordinazione
Via J. Nardi, 39
I-50132 Firenze/Italy
Tel: + 39/55 243 990 245 990
Fax: + 39/55 247 8366

Prof. Dr. Eric Monflier
Université d'Artois
Faculté des Sciences J. Perrin/
Laboratoire de Physicochimie des
Interfaces – CRUAL
Rue Jean Souvraz – Sac postal 18
F-62307 Lens/France
Tel: + 33/321 791 715
Fax: + 33/321 791 717
E-mail: monflier@univ-artois.fr

Prof. Dr. André Mortreux
Université des Sciences et Technologies
de Lille
Ecole Nationale Supérieure de Chimie
de Lille
B.P. 108
F-59652 Villeneuve d'Ascq Cédex/France
Tel: + 33/320 434 993
Fax: + 33/320 436 585
E-Mail: andre.mortreux@enac-lille.fr

Prof. Dr. Antonino Morvillo
Università di Padova
Dipartimento di Chimica Inorganica
Via Marzolo 1
I-35100 Padova/Italy
Tel: + 39/49 827 5156
Fax: + 39/49 827 5161

Prof. Dr. Günther Oehme
Institut für Organische Katalyseforschung
an der Universität Rostock e.V.
Buchbinderstr. 5–6
D-18055 Rostock/Germany
Tel: + 49/381 466 930
Fax: + 49/381 466 9324
E-mail: guenther.oehme@ifok.uni-rostock.de

Prof. Dr. Tamon Okano
Department of Materials Science
Faculty of Engineering
Tottori University
Tottori 680/Japan
Tel: + 81/857 31 5260
Fax: + 81/857 31 0881
E-Mail: okano@che.tottori-u.ac.jp

Prof. Dr. Hélène Olivier-Bourbigou
Institut Français du Pétrole
1–4 Avenue de bois Préau
F-92852 Rueil-Malmaison Cédex
France
Tel: + 33/1 475 26779
Fax: + 33/1 475 26055
E-mail: helene.olivier-bourbigou@ifp.fr

Dr. Yücel Önal
Technische Universität Darmstadt
Institut für Technische Chemie und
Makromolekulare Chemie
Petersenstr. 20
D-64287 Darmstadt/Germany
Tel: +49/6151 16 3766
Fax: +49/6151 16 4788
E-mail: oenal@ct.chemie.tu-darmstadt.de

Dr. Cesar G. Ortiz
Texas A&M University
Department of Chemistry
PO Box 30012
College Station, Texas 77892-3012
USA
Tel: +1/979 845 5417
Fax: +1/979 845 0158

Prof. Dr. Georgios Papadogianakis
University of Athens
Department of Chemistry
Industrial Chemistry Laboratory
Panepistimiopolis – Zografou
GR-15771 Athens/Greece
Tel: + 30/1 728 4235
Fax: + 30/1 724 9103
E-mail: papadogianakis@chem.uoa.gr

Prof. Dr. Peter J. Quinn
King's College London
Section of Biochemistry
Campden Hill
GB-London W8 7AH/Great Britain
Tel: + 44/171 333 4408
Fax: + 44/171 333 4500
E-mail: p.quinn@kcl.ac.uk

Dr. J.N.H. Reek
University of Amsterdam
Institute of Molecular Chemistry
Homogeneous Catalysis
Nieuwe Achtergracht 166
NL-1018 WV Amsterdam
The Netherlands
Tel: +31/20 525 6437
Fax: +31/20 525 6422
E-mail: reek@science.uva.nl

Dr. Claus-Peter Reisinger
Business Development
Exatec LLC
31220 Oak Creek Drive
Wixom, Michigan 48393/USA
E-Mail: www.exatec.biz

Dr. Stefan Rossenbach
Bergische Universität
Gauss-Str. 20
D-42047 Wuppertal

Dr. Ana M. Santos
Technische Universität München
Anorganisch-chemisches Institut
Lichtenbergstr. 4
D-85747 Garching/Germany
Tel: +49/89 2891 3102
Fax: +49/89 2891 3473
E-mail: ana.kuehn@ch.tum.de

Dr. Marie-Christine Scherrmann
Laboratoire de Chimie Organique
Multifonctionelle
Université de Paris-Sud, Bat 420
F-91405 Orsay/France
Tel: +33/1691 54719
Fax: +33/1691 54715
E-mail: mcscherr@icmo.u-psud.fr