Editor-in-Chief

JOHN R. SHAPLEY

University of Illinois, Urbana-Champaign

INORGANIC SYNTHESES

Volume 34

WILEY-INTERSCIENCE

JOHN WILEY & SONS, INC. PUBLICATION

INORGANIC SYNTHESES

Volume 34

●●●●●●●

Board of Directors

Future Volumes

35 ALFRED P. SATTELBERGER *Los Alamos National Lab*
36 THOMAS B. RAUCHFUSS *University of Illinois, Urbana-Champaign*
37 TOBIN J. MARKS *Northwestern University*

International Associates

Editor-in-Chief

JOHN R. SHAPLEY

University of Illinois, Urbana-Champaign

INORGANIC SYNTHESES

Volume 34

WILEY-INTERSCIENCE

JOHN WILEY & SONS, INC. PUBLICATION

Published by John Wiley & Sons, Inc., Hoboken, New Jersey.
Published simultaneously in Canada.

Library of Congress Catalog Number: 39:23015

ISBN 0-471-64750-0

Printed in the United States of America

10 9 8 7 6 5 4 3 2 1

PREFACE

The present volume of *Inorganic Syntheses* continues the pattern of the last three volumes in the series, namely, specific thematic chapters along with other contributions that together reflect the diversity of inorganic synthetic activities in modern research.

The five chapters in this volume are arranged in a rough order of increasing complexity for the compounds described. Chapter 1 is a collection of syntheses for main group compounds, some interesting in their own right and others primarily for their use in metal complexes. Chapter 2 details procedures for largely mononuclear organometallic and coordination complexes, with central elements ranging across the periodic table and with a wide variety of ligand types. In contrast, Chapter 3 has a specific focus on transition metal compounds containing carbonyl ligands. Chapter 4 illustrates an explosively developing research theme in which the cyanide ligand is used as a linking agent in the assembly of polynuclear metal complexes with the purpose, for example, of achieving unique magnetic properties. Finally, procedures for other types of polynuclear and cluster compounds are displayed in Chapter 5. The articles in this volume will provide tested syntheses of compounds targeted for ongoing research. However, I trust, as in my own experience with *Inorganic Syntheses*, that they also will stimulate new research ideas, the results of which will serve to nurture future volumes in the series.

A volume of this sort does not happen without the contributions of many people, first and foremost, of course, the submitters and the checkers of the individual articles. I appreciate their patience when progress appeared to be slow and their quick responses when urgency was requested. Several individuals deserve explicit acknowledgment for their critical support of this project: Heinrich Vahrenkamp for his insight and effort in soliciting the cyanide-related syntheses in Chapter 4; Herb Kaesz for his interest in involving me with *Inorganic Syntheses*, initially with Volume 26, and for his sharp editorial eye regarding many articles that appear here; Marcetta Darensbourg for her experienced advice and counsel throughout the process of planning and assembling this volume; Stan Ching for his prompt and efficient handling of the manuscripts as they were submitted; and Julie Sides and Maureen Buxton for their truly invaluable secretarial assistance. I thank also the members of the Editorial Board for their many useful comments and suggestions regarding the submitted manuscripts.

Finally, I dedicate this volume to the memory of John A. Osborn (1940–2000), who first showed me the fun to be had in exploring inorganic syntheses.

JOHN R. SHAPLEY
Urbana, Illinois

NOTICE TO CONTRIBUTORS AND CHECKERS

The *Inorganic Syntheses* series is published to provide all users of inorganic substances with detailed and reliable procedures for the preparation of important and timely compounds. Thus the series is the concern of the entire scientific community. The Editorial Board hopes that all chemists will share in the responsibility of producing *Inorganic Syntheses* by offering their advice and assistance in both the formulation and the laboratory evaluation of outstanding syntheses. Help of this kind will be invaluable in achieving excellence and pertinence to current scientific interests.

There is no rigid definition of what constitutes a suitable synthesis. The major criterion by which syntheses are judged is the potential value to the scientific community. An ideal synthesis is one that presents a new or revised experimental procedure applicable to a variety of related compounds, at least one of which is critically important in current research. However, syntheses of individual compounds that are of interest or importance are also acceptable. Syntheses of compounds that are readily available commercially at reasonable prices are not acceptable. Corrections and improvements of syntheses already appearing in *Inorganic Syntheses* are suitable for inclusion.

The Editorial Board lists the following criteria of content for submitted manuscripts. Style should conform with that of previous volumes of *Inorganic Syntheses*. The introductory section should include a concise and critical summary of the available procedures for synthesis of the product in question. It should also include an estimate of the time required for the synthesis, an indication of the importance and utility of the product, and an admonition if any potential hazards are associated with the procedure. The Procedure section should present detailed and unambiguous laboratory directions and be written so that it anticipates possible mistakes and misunderstandings on the part of the person who attempts to duplicate the procedure. Any unusual equipment or procedure should be clearly described. Line drawings should be included when they can be helpful. All safety measures should be stated clearly. Sources of unusual starting materials must be given, and, if possible, minimal standards of purity of reagents and solvents should be stated. The scale should be reasonable for normal laboratory operation, and any problems involved in scaling the procedure either up or down should be discussed. The criteria for judging the purity of the final product should be delineated clearly. The Properties section should supply and discuss those physical and chemical characteristics that are relevant to judging the purity of the product

and to permitting its handling and use in an intelligent manner. Under References, all pertinent literature citations should be listed in order. A style sheet is available from the Secretary of the Editorial Board.

The Editorial Board determines whether submitted syntheses meet the general specifications outlined above. Every procedure will be checked in an independent laboratory, and publication is contingent on satisfactory duplication of the syntheses. For online access to information and requirements, see: *www.inorgsynth.com.*

Each manuscript should be submitted in duplicate to the Secretary of the Editorial Board, Professor Stanton Ching, Department of Chemistry, Connecticut College, New London, CT 06320. The manuscript should be typewritten in English. Nomenclature should be consistent and should follow the recommendations presented in *Nomenclature of Inorganic Chemistry*, 2nd ed., Butterworths & Co, London, 1970 and in *Pure and Applied Chemistry*, Volume 28, No. 1 (1971). Abbreviations should conform to those used in publications of the American Chemical Society, particularly *Inorganic Chemistry.*

Chemists willing to check syntheses should contact the editor of a future volume or make this information known to Professor Ching.

TOXIC SUBSTANCES AND LABORATORY HAZARDS

Chemicals and chemistry are by their very nature hazardous. Chemical reactivity implies that reagents have the ability to combine. This process can be sufficiently vigorous as to cause flame, an explosion, or, often less immediately obvious, a toxic reaction.

The obvious hazards in the syntheses reported in this volume are delineated, where appropriate, in the experimental procedure. It is impossible, however, to foresee every eventuality, such as a new biological effect of a common laboratory reagent. As a consequence, *all* chemicals used and *all* reactions described in this volume should be viewed as potentially hazardous. Care should be taken to avoid inhalation or other physical contact with all reagents and solvents used in this volume. In addition, particular attention should be paid to avoiding sparks, open flames, or other potential sources that could set fire to combustible vapors or gases.

A list of 400 toxic substances may be found in the *Federal Register*, Volume 40, No. 23072, May 28, 1975. An abbreviated list may be obtained from *Inorganic Syntheses*, Vol. 18, p. xv, 1978. A current assessment of the hazards associated with a particular chemical is available in the most recent edition of *Threshold Limit Values for Chemical Substances* and *Physical Agents in the Workroom Environment* published by the American Conference of Governmental Industrial Hygienists.

The drying of impure ethers can produce a violent explosion. Further information about this hazard may be found in *Inorganic Syntheses*, Volume 12, p. 317.

CONTENTS