

INORGANIC SYNTHESSES

Volume VIII

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HENRY F. HOLTZCLAW, JR.

THE UNIVERSITY OF NEBRASKA

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Volume VIII

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PREFACE

This volume of **INORGANIC SYNTHESSES** contains sixty-eight contributions. Two articles of a review nature are included, one on metal derivatives of β -keto imines (β -imino ketones) and the other on the chemistry of the noble gases.

As in the earlier volumes, each synthesis has been checked experimentally in a different laboratory than that from which it was submitted. Furthermore, each synthesis has been critically scrutinized by the members of the Editorial and Advisory Boards and, after editing, has been sent to author and checker for approval of the edited manuscript. As in previous volumes, addresses of those submitting and checking syntheses are the addresses in effect at the time the work was done. In addition, a change in address, when applicable, is indicated for the senior author for convenience in correspondence.

The Editorial and Advisory Boards of **INORGANIC SYNTHESSES** encourage contributions both from this country and abroad and are pleased with the increasing number of scientists of other countries who are submitting or checking syntheses. Twenty-five of the sixty-eight contributions in this volume originated in or were checked in laboratories abroad.

In accord with previous practice, the syntheses in this volume are arranged on the basis of the Mendeleev periodic classification, with subdivision into A and B groups. Inasmuch as the placing of syntheses within a given chapter is arbitrary depending upon which element of the compound is chosen for the classification, the practice of listing appropriate syntheses from other chapters at the beginning of each chapter is continued in Volume VIII.

Nomenclature, particularly for compounds containing elements of periodic groups VA and VIA, often presents problems. In some cases, general agreement has not yet been reached among experts in the field. For example, names considered for $\text{Cl}_3\text{P}=\text{N}-\text{P}(\text{O})\text{Cl}_2$, during the editing of synthesis 22 were trichlorophosphazophosphorus(V) oxychloride, (trichlorophosphoranylidene)amidophosphoryl dichloride, (trichlorophosphoranylidene)phosphoramidic dichloride, and (dichlorophosphinyl)phosphorimidic trichloride. The compound $(\text{PNCl}_2)_4$ used as a starting material in syntheses 20 and 21 and often referred to simply as "tetrameric phosphonitrile chloride" or "phosphonitrile chloride, cyclic tetramer," may alternatively be named octachlorocyclotetraphosphazatetraene or, more systematically, 1,3,5,7,2,4,6,8-tetrazatetraphosphocine 2,2,4,4,6,6,8,8-octachloride.

Emphasis has been placed on the use of systematic nomenclature consistent with recommendations of the International Union of Pure and Applied Chemistry and/or the Chemical Abstracts Service. However, in some instances, a factor such as a strong preference of the author or common usage has influenced the choice. In several such instances, an alternative name as a synonym has been included at least once within the article. For example, in synthesis 20, three names are given for the eight-membered ring compound $\text{N}_4\text{P}_4(\text{OC}_2\text{H}_5)_8$: octaethoxycyclotetraphosphazatetraene; tetrameric ethyl phosphonitrilate; and 2,2,4,4,6,6,8,8-octaethoxy-2,2,4,4,6,6,8,8-octahydro-1,3,5,7,2,4,6,8,-tetrazatetraphosphocine (the last being the name preferred by the Chemical Abstracts Service). It is hoped that until agreement is reached on a logical unambiguous system of nomenclature the names used in this volume will be adequate for complete clarity.

It is a pleasure to acknowledge the election of Professor Stanley Kirschner, Department of Chemistry, Wayne State University, Detroit, Michigan, to the new position of Secretary of the Editorial Board. Contributions to future

volumes are invited and should be submitted to Professor Kirschner. Manuscripts should be in accord with the *Notice to Contributors* section, which follows the *Preface* in this volume. Competent checkers are essential to the success of INORGANIC SYNTHESSES. Persons who are interested in helping with this important function should communicate with Professor Kirschner.

The editor-in-chief for Volume IX is Professor S. Young Tyree, Jr., Department of Chemistry, University of North Carolina, Chapel Hill, North Carolina, and for Volume X is Dr. Earl L. Muetterties, Central Research Department, Experimental Station, E. I. du Pont de Nemours & Company, Wilmington, Delaware.

Several new members of the Editorial Board have been elected to the Editorial Board since the publication of Volume VII:

Professor L. Malatesta, Università Di Milano, Milano, Italy

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Professor Richard H. Holm, University of Wisconsin, Madison, Wisconsin

Dr. John K. Ruff, Rohm and Haas Company, Huntsville, Alabama

Professor Malatesta begins his term of service with Volume IX; the other four new members, with Volume X.

This volume is dedicated to the late Professor Francis P. Dwyer, who at the time of his death was a member of the Editorial Board. The Boards acknowledge with respect and appreciation the fine contributions Professor Dwyer made to the work of INORGANIC SYNTHESSES and in a broader sense to the field of inorganic chemistry.

The editor-in-chief takes pleasure in expressing his appreciation to his colleagues on the Editorial and Advisory

Boards for their fine help in the preparation of this volume. He is especially grateful to Miss Janet D. Scott for her work on nomenclature and indexing. Appreciation is expressed also to Professor Roy M. Adams, Chairman of the Committee on Inorganic Nomenclature for the Division of Inorganic Chemistry of the American Chemical Society, and to Dr. Kurt L. Loening, Director of Nomenclature for the Chemical Abstracts Service, for advice on several specific questions with respect to nomenclature. The editorial help of Mr. Theodore A. Michelfeld and Dr. John R. Demuth of the University of Nebraska is gratefully acknowledged. The editor wishes also to thank Miss Corrine Newton, Miss Camilla Connell, Miss Connie Svolopoulos, and Miss Georgianne Kozisek for their capable assistance in typing the manuscript.

The editors hope that users of INORGANIC SYNTHESSES will call to their attention any errors or omissions. The suggestions and criticisms of readers are helpful and are much appreciated.

Henry F. Holtzclaw, Jr.

NOTICE TO CONTRIBUTORS

The INORGANIC SYNTHESSES series is published to provide all users of inorganic substances with detailed and foolproof 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 SYNTHESSES by offering their advice and assistance both in the formulation and laboratory evaluation of outstanding syntheses. Help of this type 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 which 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.

The Editorial Board lists the following criteria of content for submitted manuscripts: Style should conform with that of previous volumes of INORGANIC SYNTHESSES. The *Introduction* 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* 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 clearly stated. 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 clearly delineated. The section on *Properties* should list 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.

The Editorial Board determines whether submitted syntheses meet the general specifications outlined above. Every synthesis must be satisfactorily reproduced in a different laboratory than that from which it was submitted.

Each manuscript should be submitted in duplicate to the Secretary of the Editorial Board, Professor Stanley Kirschner, Department of Chemistry, Wayne State University, Detroit, Michigan, 48202, U.S.A. The manuscript should be typewritten in English. Nomenclature should be consistent and should follow the recommendations presented in "The Definitive Rules for Nomenclature of Inorganic Chemistry," *J. Am. Chem. Soc.*, **82**, 5523 (1960). Abbreviations should conform to those used in publications of the American Chemical Society, particularly INORGANIC CHEMISTRY.

To
Francis Patrick Dwyer
(1910-1962)

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