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Vol. 30

Yearbook

1976

Synthetic Methods of Organic Chemistry



Vol. 30

Yearbook

1976

Synthetic Methods of Organic Chemistry

(内部文庫)

With Reaction Titles and Cumulative Index
of Volumes 26–30

Synthetische Methoden
der Organischen Chemie

Jahrbuch mit deutschem Registerschlüssel
Mit Titeln und Generalregister der Bände 26–30

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Preface

This is the final volume of the sixth series. It contains a cumulative subject index and all reaction titles for Volumes 26-30, including recent supplementary references. This arrangement again reduces a five-volume search to a search through a single volume. Most of the references in this volume are to papers published between 1973 and 1975. The recommendations for high-coverage searches have been revised (s. page IX).

To prevent an unwieldy expansion of this volume, repetitive parts have been held to a minimum. However, the omitted parts can be found in Vol. 29, and the respective page numbers are listed on the contents page of this volume. Supplementary references to preceding series will be included in Vol. 31.

I again wish to thank my collaborators listed on the title page for their valuable advice and assistance, and other members of Hoffmann-La Roche, Inc., Nutley, for their kind cooperation.

Nutley, New Jersey, U.S.A., May 1976.

W. Th.

From the Prefaces to the Preceding Volumes

New methods for the synthesis of organic compounds and improvements of known methods are being recorded continuously in this series.

Reactions are classified on a simple though purely formal basis by symbols, which can be arranged systematically. Thus searches can be performed without knowledge of the current trivial or author names (e.g., "Oxidation" and "Friedel-Crafts reaction").

Users accustomed to the common notations will find these in the subject index. By consulting this index, use of the classification system may be avoided. It is thought that the volumes should be kept close at hand. The books should provide a quick survey, and obviate the immediate need for an elaborate library search. Syntheses are therefore recorded in the index by starting materials and end products, along with the systematic arrangement for the methods. This makes possible a sub-classification within the reaction symbols by reagents, a further

methodical criterion. Complex compounds are indexed with cross reference under the related simpler compounds. General terms, such as synthesis, replacement, heterocyclics, may also be brought to the attention of the reader.

A table that indicates the sequence of the reagents may help the reader to locate reactions in the body of the text. This table also contains such frequently used reagents as NaOH and HCl, not included in the subject index.

A brief review, *Trends in Synthetic Organic Chemistry*, stresses highlights of general interest and calls attention to developments too recent to be included in the body of the text.

The abstracts are limited to the information needed for an appraisal of the applicability of a desired synthesis. In order to carry out a particular synthesis it is therefore advisable to have recourse to the original papers or, at least, to an abstract journal. In order to avoid repetition, selections are made on the basis of most detailed description and best yields, whenever the same method is used in similar cases. Continuations of papers already included will not be abstracted, unless they contain essentially new information. They may, however, be quoted at the place corresponding to the abstracted papers. These supplementary references (see page 750) make it possible to keep abstracts of previous volumes up-to-date.

Syntheses that are divided into their various steps and recorded in different places can be followed with the help of the notations *startg. m. f.* (starting material for the preparation of . . .) and *prepn. s.* (preparation, see).

Method of Classification

The following directions serve to explain the system of Classification.

1. Reaction Symbols

The first part of the symbol refers to the chemical bonds formed during the reaction. These bonds appear in the reaction symbols as the symbols for the two elements that have been linked together (e.g., the bond between hydrogen and nitrogen, as HN). The order of the elements is as follows: H, O, N, Hal (Halogen), S, and Rem (the remaining elements). C is always placed last.

The "principle of the latest position" is used whenever possible.

The methods of obtaining a particular chemical bond are subdivided according to types of formation. Four types are distinguished: addition (Ψ), rearrangement (\cap), exchange (\updownarrow), and elimination (\uparrow). The last part of the symbol refers to the bonds which are destroyed in the reaction or to a characteristic element which is eliminated.

The following simplifying stipulations facilitate the use of the reaction symbols: (1) The chemical bond is rigidly classified according to the structure formula without taking the reaction mechanism into consideration. (2) Double or triple bonds are treated as being equivalent to two or three single bonds, respectively. (3) Generally speaking, only stable organic compounds are taken into consideration. Intermediary compounds, such as Grignard compounds and sodiomalonic esters, and inorganic reactants, such as nitric acid, are therefore not expressed in the reaction symbols.

Examples: see volume II, page VIII.

Systematic Survey: see page 747.

2. *Reagents*

- A further subdivision, not included in the reaction symbols, is made on the basis of the reagents characteristic of the reaction. A table indicating the sequence of the reagents may be found on page 544 of vol. 29.

3. The material between the listings of the reagents is arranged with the simple examples first and the more complicated ones following.

4. When changes in more than one chemical bond occur during a reaction, as, for example, in the formation of a new ring, or if the reaction can be carried out in different ways, these reactions are introduced in several places when necessary. The main entry in such cases is placed usually according to the "principle of the latest position"; the other entries are cross-referenced back to it.

High-Coverage Searches

A search through Synthetic Methods provides a selection of key references from the journal literature. For greater coverage, as for bibliographies, a supplementary search through the following publications is suggested.

*Chemical Reactions Documentation Service*¹

which also includes abstracts from patents and provides the data coded on magnetic tape and punched cards as additional retrieval tools.

*Science Citation Index*²

for which Synthetic Methods serves as a source of starting references.

*Chemical Abstract Service*³

References may not be included in Synthetic Methods

- 1) to reactions which are routinely performed by well known procedures,
- 2) to subjects which can be easily located in handbooks and indexes of abstract journals, such as the ring system of heterocyclics or the metal in case of organometallic compounds, and
- 3) to inadequately described procedures, especially where yields are not indicated.

References to less accessible publications such as those in the Russian or Japanese language are, as a rule, only included if the method in question is not described elsewhere.

¹ Derwent Publications Ltd., 128 Theobalds Road, London WC1X 8RP, England.

² Institute for Scientific Information, Philadelphia, Pa., USA.

³ Chemical Abstracts Service, Columbus, Ohio, USA.

Trends in Synthetic Organic Chemistry 1976

In contrast to reactions at high temperature those at very low temperature are more difficult to perform and have therefore been much less investigated. Recently, the utility of cryochemical processes has been demonstrated by the smooth preparation of cyclopropanone from ketene and diazomethane at -145°C .¹

Epoxides and episulfides can be deoxygenated and desulfurated respectively with retention of stereochemistry by a new reagent, 3-methylbenzothiazole-2-selone². Dimethylphenylsilyllithium has been used for the *trans*-stereospecific deoxygenation of epoxides³.

Bridgehead functionalization can be conveniently achieved with lead tetracetate⁴. Regio- and stereo-specific vicinal oxyamination of olefins by alkyl imido osmium compounds⁵ has been reported as a novel reaction. A new method for the oxidation of alcohols with peracids depends upon catalysis by N-oxide radicals⁶. Optically active labile sec. bromides can be conveniently prepared from chiral alcohols by cautious reaction with PBr_3 and pyridine⁷.

gem-Dialkylations of carbanions can be easily performed in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene⁸. α -Cyanoguanamines are new intermediates which can be used for various syntheses such as the preparation of α -diketones from amides⁹. The latter can be conveniently obtained from acids with BF_3 in the presence of triethyl-

¹ E. F. Rothgery, R. J. Holt, and H. A. McGee, Jr., *Am. Soc.* **97**, 4971 (1975).

² V. Calò et al., *Synthesis* **1976**, 200.

³ M. T. Reetz and M. Plachky, *Synthesis* **1976**, 199.

⁴ S. R. Jones and J. M. Mellor, *Synthesis* **1976**, 32.

⁵ K. B. Sharpless et al., *Am. Soc.* **97**, 2305 (1975); cf. *J. Org. Chem.* **41**, 177 (1976).

⁶ J. A. Cella, J. A. Kelley, and E. F. Kenehan, *J. Org. Chem.* **40**, 1860 (1975); cf. B. Ganem, *ibid.* **40**, 1998.

⁷ R. O. Hutchins, D. Masilamani, and C. A. Maryanoff, *J. Org. Chem.* **41**, 1071 (1976); cf. D. Landini, S. Quici, and F. Rolla, *Synthesis* **1975**, 430.

⁸ H. Oediger and F. Möller, *A.* **1976**, 348.

⁹ J. Toye and L. Ghosez, *Am. Soc.* **97**, 2276 (1975).

amine¹⁰. α,β -Ethylene- β -chloroketones can be easily prepared by treating β -diketones or β -ketoaldehydes with oxalyl chloride in an inert solvent¹¹. Of several new methods for the preparation of nitriles from aldehydes¹², a particularly simple one merely uses hydroxylamine hydrochloride in refluxing dimethylformamide¹³. Ar. thioamides can be obtained in one step by Friedel-Crafts thioacylation with ethoxy-carbonyl isothiocyanate¹⁴.

A convenient preparation of isothiocyanates from amines, carbon disulfide, and Grignard reagents, such as ethylmagnesium bromide, has been published¹⁵. Phenyl N-phenylphosphoramidochloridate is a new phosphorylation agent¹⁶.

Acid chlorides have been found to add easily to carbon-nitrogen double bonds of heterocyclics, e.g. of Δ^1 -azirines, to form N-acyl-2-chloraziridines¹⁷.

Hydroxyl groups, even if tertiary¹⁸, can be protected as methylthiomethyl ethers. The protective group can be electively removed under neutral conditions in the presence of mercuric ion¹⁹. β -Methoxyethoxymethyl, a new protective group for hydroxyl, can be removed with Lewis acids such as TiCl_4 or ZnBr_2 under mild conditions²⁰. Protection of carboxyl groups as 5-methylene-1,3-dioxanes has been recommended. This protective group avoids the introduction of new asymmetrical centers, while amenable to selective removal under mild conditions²¹. Removal of certain O-protective groups by radical anions can be performed under a series of controlled conditions to make it suitably preferential and selective²².

The preparation of ketones by ozonization of sec. alcohols is recommended with certain limitations²³. A convenient preparation of

¹⁰ J. Tani, T. Oine, and I. Inoue, *Synthesis* 1975, 714.

¹¹ R. D. Clark and C. H. Heathcock, *J. Org. Chem.* 41, 636 (1976).

¹² *Synth. Meth.* 30, 277.

¹³ J. Liebscher and H. Hartmann, *Z. Chem.* 15, 302 (1975).

¹⁴ E. P. Papadopoulos, *J. Org. Chem.* 41, 962 (1976).

¹⁵ S. Sakai, T. Fujinami, and T. Aizawa, *Bull. Chem. Soc. Japan* 48, 2981 (1975).

¹⁶ W. S. Zielinski and Z. Leńnikowski, *Synthesis* 1976, 185.

¹⁷ A. Hassner, S. S. Burke, and J. Cheng-fan I, *Am. Soc.* 97, 4692 (1975); cf. B. T. Golding and D. R. Hall, *Soc. Perkin I* 1975, 1302.

¹⁸ K. Yamada et al., *Tetrah. Let.* 1976, 65.

¹⁹ E. J. Corey and M. G. Bock, *Tetrah. Let.* 1975, 2643, 3269.

²⁰ E. J. Corey, J.-L. Gras, and P. Ulrich, *Tetrah. Let.* 1976, 809.

²¹ E. J. Corey and J. W. Suggs, *Tetrah. Let.* 1975, 3775.

²² R. L. Letsinger and J. L. Finnan, *Am. Soc.* 97, 7197 (1975).

²³ W. L. Waters et al., *J. Org. Chem.* 41, 889 (1976); by dry ozonization cf. ref. 52.

3-arylated aldehydes and ketones from allylic alcohols has been published²⁴. Tris(triphenylsilyl)vanadate is an excellent catalyst for the Meyer-Schuster rearrangement; α,β -unsatd. steroidal aldehydes have thus been produced from the corresponding ethynylcarbinols in high yield²⁵. Novel and mild procedures for synthetically useful interchanges of acetal, thioacetal, and hemithioacetal groups have been described²⁶. The preparation of glycosides, including di- and pseudo-saccharides, under mild conditions with amide acetals has been reported²⁷.

A regiospecific Baeyer-Villiger oxidation with ceric ion converts cyclic ketones into lactones, which may be different from those obtained by conventional peracid oxidation²⁸.

A simple total synthesis of prostaglandins uses monomeric formaldehyde as trapping agent for kinetic enolates²⁹. α -Lithiated N,N-dimethylhydrazones have been successfully used as enolate equivalents³⁰.

Reactions via organocesium compounds have been published³¹. Protonation of lithium alkynyltrialkylborates with acid can be directed to achieve a double migration of alkyl groups from boron to carbon³².

High yields of linear esters can be obtained by carbonylation of α -olefins in the presence of homogeneous platinum complexes and a Group IVB metal halide, such as SnCl_4 , as co-catalyst³³. Cuprous methyltrialkylborates are convenient intermediates for the synthesis of saturated nitriles from acrylonitrile, esters from acrylates, as well as *trans*- γ,δ -ethyleneketones from 1-acyl-2-vinylcyclopropanes³⁴. Cyclohexyl- and β -phenethyl-trihalogenomethylmercury compounds are carbene transfer agents effective at room temperature³⁵. β -Stannylalkylidenephosphoranes have been introduced as promising novel intermediates³⁶. Carbonylchromium complexes may be used to increase reactivity, enhance selectivity, or protect substituents of arene rings. The activating power of the carbonylchromium group can be modified

²⁴ J. B. Melpolder and R. F. Heck, *J. Org. Chem.* **41**, 265 (1976); A. J. Chalk and S. A. Magennis, *ibid.* **41**, 273.

²⁵ G. L. Olson, K. D. Morgan, and G. Saucy, *Synthesis* 1976, 25.

²⁶ E. J. Corey and T. Hase, *Tetrah. Let.* 1975, 3267.

²⁷ S. Hanessian and J. Banoub, *Tetrah. Let.* 1976, 657, 661.

²⁸ G. Mehta, P. N. Pandey, and T.-L. Ho, *J. Org. Chem.* **41**, 953 (1976).

²⁹ G. Stork and M. Isobe, *Am. Soc.* **97**, 6260 (1975); cf. *ibid.* **98**, 1583 (1976).

³⁰ E. J. Corey and D. Enders, *Tetrah. Let.* 1976, 3, 11.

³¹ N. Collignon, *J. Organometal. Chem.* **96**, 139 (1975); *Bl.* 1975, 1821.

³² M. M. Midland and H. C. Brown, *J. Org. Chem.* **40**, 2845 (1975).

³³ J. F. Knifton, *J. Org. Chem.* **41**, 793 (1976).

³⁴ N. Miyaura, M. Itoh, and A. Suzuki, *Tetrah. Let.* 1976, 255.

³⁵ D. Seyferth, C. K. Hsiao, and D. Dagani, *J. Organometal. Chem.* **104**, 9 (1976).

³⁶ S. J. Hannon and T. G. Traylor, *Chem. Commun.* 1975, 630.

by replacing one of the CO-groups by other ligands³⁷. The complexes are also useful for the preparation of optically active compounds³⁸.

Both the increased yield and lower reaction temperature of anionic oxy-Cope processes should improve the synthetic utility of these and related rearrangements³⁹.

An aldol-type ring closure of steroid intermediates with high asymmetrical selectivity has been achieved in the presence of L-phenylalanine⁴⁰. A highly stereoselective cyclopentene ring annelation has been published⁴¹. An efficient double cycloisomerization of dienic keto esters in the presence of stannic chloride has been reported, affording a direct entry into functionalized decalins⁴².

A facile chroman ring closure is the starting point of a new synthesis of vitamin E and related compounds⁴³. An efficient synthesis of *exo*- and *endo*-brevicomins, bicyclic ketals, has been reported⁴⁴. A one-step 6,7-benzomorphan ring synthesis has been achieved by *m*-bridging of electron-deficient aromatics⁴⁵. Uracils substituted in 5-position by a carbon chain can be obtained from 5-fluorouracil via a novel 1,4-fragmentation of a regioselectively 4,5-fused cyclobutane ring⁴⁶.

D-Biotin has been obtained by a stereospecific total synthesis from L-(+)-cysteine without a chemical resolution series characteristic of all previous syntheses. Part of this process is a remarkable rearrangement of a 4-vinylthiazolidine ring by bromination resulting in a 3-amino-4-bromotetrahydrothiophene ring⁴⁷.

The formation of undesirable stable emulsions in phase-transfer catalyzed reactions can be avoided by triphase catalysis with a polymer-based quaternary ammonium salt as catalyst, which can be removed by a simple filtration⁴⁸.

³⁷ G. Jaouen, A. Meyer, and G. Simonneaux, *Chem. Commun.* 1975, 813.

³⁸ *Synth. Meth.* 30, 540.

³⁹ D. A. Evans and A. M. Golob, *Am. Soc.* 97, 4765 (1975).

⁴⁰ S. Danishefsky and P. Cain, *Am. Soc.* 97, 5282 (1975).

⁴¹ B. M. Trost and D. E. Keeley, *Am. Soc.* 98, 248 (1976).

⁴² R. W. Skeean, G. L. Trammell, and J. D. White, *Tetrah. Let.* 1976, 525.

⁴³ J. W. Scott et al., *Helv.* 59, 290 (1976).

⁴⁴ P. J. Kocienski and R. W. Ostrow, *J. Org. Chem.* 41, 398 (1976).

⁴⁵ R. R. Bard and M. J. Strauss, *Am. Soc.* 97, 3789 (1975).

⁴⁶ A. Wexler and J. S. Swenton, *Am. Soc.* 98, 1602 (1976); 5-C-subst. pyrimidine nucleosides via organopalladium intermediates cf. D. E. Bergstrom and J. L. Ruth, *ibid.* 98, 1587.

⁴⁷ P. N. Confalone et al., *Am. Soc.* 97, 5936 (1975).

⁴⁸ S. L. Regen, *Am. Soc.* 97, 5956 (1975).

Useful reactions on alumina surface have been reported, including displacements under mild conditions⁴⁹ and a preferential reduction of aldehydes to prim. alcohols with isopropanol⁵⁰. Dehydrated chromatographic alumina has been recommended for the preparation of olefins from tosylates⁵¹. A novel oxidation method is the dry ozonization of saturated compounds absorbed on silica gel. As one application, tertiary alcohols can be conveniently obtained with retention of configuration⁵².

Reductions with tetrabutylammonium borohydride can be performed in the absence of protic solvents⁵³. A mixture of TiCl_3 and LiAlH_4 is a convenient reducing agent, which has been used for the reductive dimerization of oxo compounds to sym. ethylene derivatives⁵⁴, and of alcohols⁵⁵, also for the preparation of ethylene derivatives from oxido compounds⁵⁶. Magnesium amalgam-titanium tetrachloride is a useful reagent for inter- and intra-molecular pinacolic coupling of oxo compounds⁵⁷. Recently, active titanium metal powder has been found to be a superior reagent for the production of olefins by coupling of carbonyls or by reduction of glycols⁵⁸. Phosphine complexes of transition metals, such as rhodium, have been chemically bonded to the surface of silica. Most of these catalysts retain substantial hydrogenation activity in the presence of mercaptans⁵⁹.

Anhydrous H_2O_2 can be conveniently stored and handled as the solid triethylenediamine $\cdot 2\text{H}_2\text{O}_2$ complex⁶⁰.

Pyridinium chlorochromate, a readily available stable reagent oxidizes a wide variety of alcohols to oxo compounds with high efficiency⁶¹. Also, a convenient synthesis of (-)-pulegone from (-)-citronellol through an oxidative ring closure with this reagent has been published⁶².

⁴⁹ G. H. Posner et al., *Tetrah. Let.* 1975, 3597.

⁵⁰ G. H. Posner and A. W. Runquist, *Tetrah. Let.* 1975, 3601.

⁵¹ G. H. Posner and G. M. Gurria, *J. Org. Chem.* 41, 578 (1976).

⁵² Z. Cohen et al., *J. Org. Chem.* 40, 2141 (1975).

⁵³ D. J. Raber and W. C. Guida, *J. Org. Chem.* 41, 690 (1976).

⁵⁴ *Synth. Meth.* 30, 561.

⁵⁵ J. E. McMurry and M. Silvestri, *J. Org. Chem.* 40, 2687 (1975).

⁵⁶ *Synth. Meth.* 30, 662 suppl. 30.

⁵⁷ E. J. Corey, R. L. Danheiser, and S. Chandrasekaran, *J. Org. Chem.* 41, 260 (1976).

⁵⁸ J. E. McMurry and M. P. Fleming, *J. Org. Chem.* 41, 896 (1976).

⁵⁹ K. G. Allum et al., *J. Organometal. Chem.* 107, 390 (1976).

⁶⁰ P. G. Cookson, A. G. Davies, and N. Fazal, *J. Organometal. Chem.* 99, C31 (1975).

⁶¹ E. J. Corey and J. W. Suggs, *Tetrah. Let.* 1975, 2647.

⁶² E. J. Corey, H. E. Ensley, and J. W. Suggs, *J. Org. Chem.* 41, 380 (1976).

The following references in Vol. 29 under Trends have been entered in this volume⁶³:

4/366; 5/33; 6/581; 8/25; 11/591; 15/603; 16/499; 17/506; 18/144; 19/462; 20/619; 21/674; 23/487; 24/118; 30/135; 27/414; 28/414; 29/540; 30/266; 31/255; 32/98; 33/195; 34/361; 37/574; 38/168; 40/178; 41/336; 42/672; 43/338; 44/406; 50/40; 51/322; 52/365; 53/66; 54/80.

⁶³ The first figure refers to the footnote in Trends, Vol. 29, the second figure to the entry number to this volume.

有机化学合成方法 第30卷

本丛书第1—26卷已全部编目，编目情况和丛书简介请参见F44/110。第27、28卷编目号为F75/96—97。第29卷编目号为F87/96。这是丛书的第30卷，也是丛书第六辑的最后一册（每五册为一辑）。本卷摘入700种新的有机反应提要，还包括第26—30卷的累积索引和全部反应名称，还有最近的补充参考文献。大部文献是1973到1975年间印行的。在每种反应提要中均写出反应式、实验步骤、试剂、反应条件、产率、文献来源等资料。书前有本卷序言、分类法（附有德文），1976年合成有机化学趋向。

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Formation of H—O Bond

Uptake



Addition to Oxygen

HO↓OO

Triethylamine

 Et_3N

5-Hydroxytropolones

s. 26, 1

Thiourea

 $(H_2N)_2CS$

Diols from cyclic peroxides

C

1.



3,6-Peroxy- α -damascone stirred 8 hrs. with thiourea in methanol \rightarrow 3,6-dihydroxy- α -damascone. Y: 75%. K. H. Schulte-Elte, M. Gadola, and G. Ohloff, *Helv. 56*, 2028 (1973); s. a. Y. Itô, M. Oda, and Y. Kitahara, *Tetrah. Let.* 1975, 239.

Addition to Oxygen and Carbon

HO↓OC

Sodium acetate

 CH_3COONa

Glycols from oxido compds.

s. 26, 2

 $\rightarrow C(OH)C(OH)$

Benzopinacol

 \leftarrow

Quinols from quinones

 \leftarrow

s. 28, 31

Hydrazobenzene

 $PhNHNHPh$

s. 26, 263; with N,N -diethylhydroxylamine cf. S. Fujita and K. Sano, *Tetrah. Let.* 1975, 1695

 N,N -Diethylhydroxylamine Et_2NOH

s. 26, 263 suppl. 30

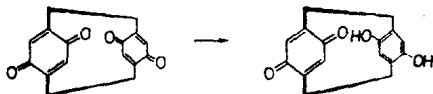
Palladium-carbon

 $Pd-C$

Intramolecular quinhydrone

 \leftarrow

2.



Startg. bisquinone hydrogenated with $Pd-C$ in dioxane until the calculated amount of H_2 has been absorbed \rightarrow product. Y: 70%. W. Rebafka and H. A. Staab, *Ang. Ch.* 85, 831 (1973); 86, 234 (1974).