W. Theilheimer

Synthetic Methods of Organic Chemistry

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Mit deutschem Registerschlüssel

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der Organischen Chemie

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Preface

The present volume contains mostly abstracts of papers published between 1977 and 1979. New references to material in the preceding volumes have again been included in the text. The index covers this volume only, inasmuch as the next volume will contain both a cumulative index and a classified arrangement of all titles in the volumes 31 through 35, thus concluding the seventh series.

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 1980. 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 (see page 481) 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 496) 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 (⇓), rearrangement (⇂), exchange (↓↑), and elimination (↑). 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 XVII.

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 481.

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.

Systematik

Für die Reihenfolge der Methoden gelten folgende Richtlinien:

1. Reaktionszeichen

Die Einteilung erfolgt zuerst nach den Bindungen, die bei einer Reaktion entstehen. Der erste Teil des Reaktions-Formelzeichens besteht somit aus den Symbolen der an der entstehenden Bindung beteiligten Elemente, z. B. HN bei einer Bindung zwischen Wasserstoff und Stickstoff. Die Reihenfolge der Elemente ist wie folgt: H, O, N, Hal (Halogen), S, Rem (Übrige Elemente). C steht an letzter Stelle.

Das *«Princip der letzten Stelle»* ist nach Möglichkeit immer angewandt worden.

Die Methoden zur Herstellung einer bestimmten Bindung werden nach deren Bildungsweise eingeteilt. Es werden 4 Fälle unterschieden: Aufnahme (⇓), Umlagerung (↶), Austausch (↓↑) und Abgabe (⇑).

Der letzte Teil des Reaktionszeichens gibt die Bindung an, die gelöst wird, oder ein charakteristisches Element, das eliminiert wird.

Die Bildung des Reaktionszeichens wird durch folgende vereinfachende Annahmen erleichtert:

1. Die Bindungen für die Registrierung ergeben sich rein formal aus den Strukturformeln, ohne dass auf Reaktionsmechanismen Rücksicht genommen wird.
2. Doppel- und Dreifachbindungen werden 2 bzw. 3 Einfachbindungen gleichgesetzt.
3. Es werden in der Regel nur stabile organische Verbindungen berücksichtigt. Zwischenprodukte, wie z. B. Grignard-Verbindungen, Na-Malonester und anorganische Reaktionspartner, wie z. B. Salpetersäure, werden deshalb nicht zur Bildung des Reaktionszeichens herangezogen.

Beispiele: siehe Band 2, Seite VI.
Systematische Übersicht: siehe Seite XVII.

2. Hilfsstoffe

Eine weitere Unterteilung, die im Reaktionszeichen nicht mehr zum Ausdruck kommt, wird nach den für die Reaktion charakteristischen

Hilfsstoffen vorgenommen. Eine Tabelle der Reihenfolge der Hilfsstoffe befindet sich auf Seite 481.

3. Innerhalb dieser Unterteilung sind die einzelnen Referate von einfachen zu komplizierten Beispielen fortschreitend angeordnet.

4. Treten bei einer Reaktion Veränderungen an mehreren Bindungen ein, wie z. B. bei Ringschlüssen, oder kann sie auf verschiedene Art durchgeführt werden, dann wird sie, falls notwendig, an mehreren Stellen eingeordnet. Das Hauptzitat steht in diesen Fällen in der Regel an der letzten Stelle; an den übrigen Stellen befinden sich Hinweise auf dieses.

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]
designed for both current awareness and retrospective retrieval. Its monthly publication, the Journal of Synthetic Methods, covers the journal and patent literature, and provides 3,000 abstracts of recently published papers annually, together with 3,000 supplementary references.
One-line access is available to over 45,000 reactions, these including the data in all the abstracts in Synthetic Methods.

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 1980

The achievements in synthetic organic sulfur chemistry [1] have inspired an extensive investigation of analogous selenium compounds [2] and, moving further down the Periodic Table, of organic tellurium compounds [3]. Thus, phenylselenimides have recently been recommended as carriers of the phenylseleno group, a widely applicable synthon owing to its easily manipulated nature [4]. Tellurides, and also selenides, can be efficiently and selectively oxidized to telluroxides and selenoxides respectively by using a positive halogen source such as *tert*-butyl hypochlorite or N-chlorosuccinimide [5]. Bis-(p-methoxyplenyl)telluroxide has been recommended as a mild and selective oxidant [6]. High yields of aryltellurocyanates have been obtained by reductive cyanation of aryltellurium trihalides [7]. Novel applications of organic sulfur chemistry continue, of course: Recently, phenyl vinyl sulfone has been found to be an excellent equivalent for ethylene and, indirectly, a wide range of terminal olefins in diene syntheses [8].

High asymmetric induction in aliphatic systems has been achieved with B-3-pinanyl-9-borabicyclo[3.3.1]nonane. A number of α,β-acetyleneketones have thus been reduced to optically active 2-acetylenealcohols [9].

Nucleophilic substitutions can be facilitated by vapor-solid phase transfer catalysis [10]. Methylations with diazomethane are effectively cat-

[1] Reviews s. Synth. Meth. *34*, 198.
[2] Reviews s. Synth. Meth. *29*, 912s*34*.
[3] Cf. review of telluranes, I. D. Sadekov, A. Y. Bushkov, V. I. Minkin, Russ. Chem. Rev. *48*, 343 (1979) (Eng. transl.).
[4] K. C. Nicolaou et al., J. Am. Chem. Soc. *101*, 3704 (1979).
[5] M. R. Detty, J. Org. Chem. *45*, 274 (1980).
[6] D. H. R. Barton, S. V. Ley, C. A. Meeholz, J. Chem. Soc. Chem. Commun. *1979*, 755.
[7] S. J. Falcone, M. P. Cava, J. Org. Chem. *45*, 1044 (1980).
[8] R. V. C. Carr, L. A. Paquette, J. Am. Chem. Soc. *102*, 853 (1980).
[9] M. M. Midland et al., J. Am. Chem. Soc. *102*, 867 (1980).
[10] P. Tundo, J. Org. Chem. *44*, 2048 (1979).

alyzed by silica gel[11]. Mannich salts can be efficiently prepared from aminals with trimethyliodosilane[12].

1,3-Dienes can be easily metalated by Lochmann's base, a mixture of K-*tert*-butoxide and *n*-butyllithum[13]. Through 1-siloxy-1,3-dienes, γ-subst. α,β-ethylenecarbonyl compounds have been obtained[14]. High yields of α,β-ethyleneketones and unsym. β-dicarbonyl compounds have been reported, the former by reaction of epoxides with carboxylic acid chlorides in the presence of Na-tetracarbonylferrate(II)[15] and the latter by acylation of ketones with α-chloroacylsilanes in the presence of lithium diisopropylamide[16].

1,3-Dithiane protective groups can be efficiently removed from oxo compounds under mild conditions by indirect electrolysis with tris-(p-tolyl)amine as a homogeneous electron transfer agent[17]. Mercaptals have been smoothly converted into ketene mercaptals with 2,2'-dipyridyl disulfide and *n*-butyllithum[18].

Rapid and mild esterification, including the preparation of macrocyclic lactones, can be carried out via carboxylic 2,4,6-trichlorobenzoic anhydrides[19]. Esters have been converted directly into acid chlorides with chlorosulfonic acid and phthaloyl chloride[20] and into nitriles with dimethylaluminum amide[21].

Isocyanates, ureas, and urethans have been conveniently prepared with 4,6-diphenylthieno[3,4-d]dioxol-2-one 5,5-dioxide, an active cyclic carbonic ester, as phosgene substitute[22].

Replacement of N-functional groups, such as isothiocyanato groups, by hydrogen has been achieved with tri-*n*-butyltin hydride in the presence of azodiisobutyronitrile[23].

[11] H. Nishiyama, H. Nagase, K. Ohno, Tetrahedron Letters *1979*, 4405, 4671.
[12] T. A. Bryson et al., J. Org. Chem. *45*, 524 (1980).
[13] J. J. Bahl, R. B. Bates, B. Gordon III, J. Org. Chem. *44*, 2290 (1979); cf. H. Yasuda et al., Bull. Chem. Soc. Japan *52*, 2036 (1979).
[14] I. Fleming, J. Goldhill, I. Paterson, Tetrahedron Letters *1979*, 3205, 3209.
[15] M. Yamashita et al., Chem. Letters *1979*, 1067.
[16] I. Kuwajima, K. Matsumoto, Tetrahedron Letters *1979*, 4095.
[17] M. Platen, E. Steckhan, Tetrahedron Letters *21*, 511 (1980).
[18] Y. Nagao, K. Seno, E. Fujita, Tetrahedron Letters *1979*, 4403.
[19] J. Inanaga et al., Bull. Chem. Soc. Japan *52*, 1989 (1979).
[20] W. J. Middleton, J. Org. Chem. *44*, 2291 (1979).
[21] J. L. Wood, N. A. Khatri, S. M. Weinreb, Tetrahedron Letters *1979*, 4907.
[22] H. Schmidt et al., Chem. Ber. *112*, 727 (1979).
[23] D. H. R. Barton, G. Bringmann, G. Lamotte, Tetrahedron Letters *1979*, 2291; D. I. John, E. J. Thomas, N. D. Tyrrell, J. Chem. Soc. Chem. Commun. *1979*, 345.

Mixtures of chlorosilanes and lithium sulfide [24], as well as O-silyl O-alkyl acetals [25], are powerful silylating agents.

Ar. acylations have been performed with selenolic esters in the presence of cuprous triflate, which has also been used for the synthesis of sulfides from mercaptals and aryl derivs [26].

Various convenient syntheses with polymer-based triphenylphosphine-lithium diorganocuprates have been published [27]. Exclusive α-substitution of heterosubstituted allylic carbanions can be achieved via allylic aluminum 'ate' complexes [28]. Allylic 2'-substitution of thioenolethers has been performed via acetoxylation [29].

A facile highly stereoselective synthesis of α- or β-disaccharides has been reported [30]. Peptide synthesis with simultaneous resolution has been achieved by using the proteolytic enzyme thermolysin [31].

Krypton ion laser irradiation gave 88% carvonecamphor by cycloisomerization of carvone [32]. High yield diene syntheses with unstable quinones can be performed by generating the quinone *in situ* from the respective hydroquinone with silver oxide [33].

A rhodium(I)-catalyzed intramolecular hydroacylation of γ,δ-ethylenealdehydes conveniently produces various cyclopentanones [34]. Regio- and stereo-specific ar. thallation-carbonylation furnishes excellent yields of lactones from alcohols, as well as ar. esters, anhydrides, and a number of heterocycles [35]. Highly strained and reactive bicyclic β-lactams are accessible via carbene insertion with diazo compounds [36]. A rather short synthesis of biotin features a remarkable intramolecular [3+2]-cycloaddition whereby a 3-(2-nitroethylthio)cycloheptene is converted stereospecifically into an octahydrocycloheptano[5,5a,6-f,g]thieno[3,4-c]isoxazole [37]. 8-Oxoprotoberberin derivs. can be obtained efficiently by

[24] G. A. Olah et al., J. Org. Chem. *44*, 4272 (1979).
[25] Y. Kita et al., Tetrahedron Letters *1979*, 4311.
[26] A. P. Kozikowski, A. Ames, J. Am. Chem. Soc. *102*, 860 (1980).
[27] R. H. Schwartz, J. San Filippo, Jr., J. Org. Chem. *44*, 2705 (1979).
[28] Y. Yamamoto, H. Yatagai, K. Maruyama, J. Org. Chem. *45*, 195 (1980).
[29] B. M. Trost, Y. Tanigawa, J. Am. Chem. Soc. *101*, 4413 (1979).
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[35] R. C. Larock, C. A. Fellows, J. Org. Chem. *45*, 363 (1980).
[36] R. W. Ratcliffe, T. N. Salzmann, B. G. Christensen, Tetrahedron Letters *21*, 31 (1980).
[37] Synth. Meth. 34, 920; s. a. J. Am. Chem. Soc. *102*, 1954 (1980).

intramolecular alkylation of the well known Reissert compds.[38], which have recently been prepared also from phthalazines[39]. The use of Vilsmeier-compds. to generate the pyrimidine ring can be superior to the conventional base-catalyzed approach[40]. In the presence of organolithium compds. such as n-butyllithium, 5-alkoxyoxazoles undergo an unusually facile ring opening leading to lithiated α-isocyanoesters, which in turn are versatile intermediates for the preparation of amino acid derivs. and a variety of heterocyclics[41].

Commercial neutral alumina has been found to act as a 3-phase catalyst if suspended in the toluene soln. of the startg. m. E. g. in the presence of potassium permanganate – also suspended in toluene – a mild oxidation of cyclododecanol to the ketone occurs. This solid-liq.-solid phase method has been applied to various displacement reactions[42] as well.

Sodium superoxide smoothly converts nitriles to amides at room temp. and also cleaves esters rapidly to acids[43].

Borane · 1,4-oxathiane is a new convenient hydroboration agent[44]. A thorough investigation of lithium hydridotriethylborate, a powerful and selective reducing agent has been published[45]. Diphosphorus tetraiodide, a stable inexpensive compound easy to prepare and handle, is a useful deoxygenating agent, e.g. for sulfoxides and selenoxides. It also converts prim. nitro compds. efficiently to nitriles[46]. Various deoxygenations, sometimes with accompanying condensation, as in the preparation of sym. ethylene derivs. by ketone dimerization, have been carried out with bis(benzene)titanium[47].

cis-Hydroxylations of C-double bonds have been performed at –70° with methyltriphenyl phosphonium permanganate in methylene chloride[48]. A mixture of chlorosulfonyl isocyanate and dimethyl sulfoxide

[38] S. Ruchirawat et al., Tetrahedron Letters *21*, 189 (1980).
[39] D. Bhattacharjee, F. D. Popp, J. Pharm. Sci. *69*, 120 (1980).
[40] R. L. N. Harris, J. L. Huppatz, T. Teitei, Australian J. Chem. *32*, 669 (1979).
[41] P. A. Jacobi, S. Ueng, D. Carr, J. Org. Chem. *44*, 2042 (1979).
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[43] N. Kornblum, S. Singaram, J. Org. Chem. *44*, 4727 (1979).
[44] H. C. Brown, A. K. Mandal, Synthesis *1980*, 153.
[45] H. C. Brown, S. C. Kim, S. Krishnamurthy, J. Org. Chem. *45*, 1 (1980).
[46] J. N. Denis, A. Krief, Tetrahedron Letters *1979*, 3995; cf. R. H. Hall, A. Jordan, M. Malherbe, J. Chem. Soc. Perkin Trans. I *1980*, 126.
[47] H. Ledon, I. Tkatchenko, D. Young, Tetrahedron Letters *1979*, 173.
[48] W. Reischl, E. Zbiral, Tetrahedron *35*, 1109 (1979).

is another new oxidant that converts alcohols to oxo compds. under mild conditions [49]. 5-Valent bismuth reagents, especially Ph_3Bi-carbonate, show remarkable functional group specificity, permitting alcohol oxidation in the presence of thiophenols, pyrroles, or indoles [50]. Bromobenzene catalyzed by $(Ph_3P)_4Pd(O)$ has been used to oxidize sec. alcohols to ketones [51]. 2-Hydroperoxyperfluoro-2-propanol is a cheap catalytic oxidant, e.g. for epoxidation. It also is recommended for the oxidation of aldehydes to acids [52].

Bromodimethylsulfonium bromide has been used for various reactions, such as the preparation of α-halogenoketones from enamines [53], the reconversion of mercaptals to oxo compds., and the preparation of sym. disulfides from mercaptans [54].

Reactions with polyphosphoric acid can be performed with advantage in a 2-phase medium by using a co-solvent such as xylene and a reduced quantity of the acid [55].

$C_{19}XeF_6$, a xenon hexafluoride-graphite intercalate, is a selective fluorination agent, e.g. for uracil and certain β-dicarbonyl compds. [56].

The following references in Vol. 33 under Trends have been entered in this volume [57]:

8/771; 9/63; 10/55; 11/523; 12/997; 13/219; 16/852; 17/532; 18/988; 19/595; 22/577; 23/977; 24/874; 26/649; 27/386; 28/786; 29/711; 31/804; 32/856, 884; 33/855; 34/871, 872, 888; 35/696; 37/664; 38/432; 39/436; 40/828; 42/239; 45/552; 46/327; 47/999; 48/41; 51/194; 53/222; 54/155; 55/256; 56/197.

[49] G. A. Olah, Y. D. Vankar, M. Arvanaghi, Synthesis *1980*, 141.
[50] D. H. R. Barton et al., J. Chem. Soc. Chem. Commun. *1979*, 705.
[51] T. Tamaru et al., Tetrahedron Letters *1979*, 1401.
[52] R. P. Heggs, B. Ganen, J. Am. Chem. Soc. *101*, 2484 (1979); Tetrahedron Letters *21*, 685, 689 (1980).
[53] G. A. Olah, Y. D. Vankar, M. Arvanaghi, Tetrahedron Letters *1979*, 3653.
[54] G. A. Olah et al., Synthesis *1979*, 720.
[55] A. Guy, J.-P. Guetté, Synthesis *1980*, 222.
[56] S. S. Yemul, H. B. Kagan, R. Setton, Tetrahedron Letters *21*, 277 (1980).
[57] The first figure refers to the footnote in Trends, Vol. 33, the second figure to the entry number of this volume.

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