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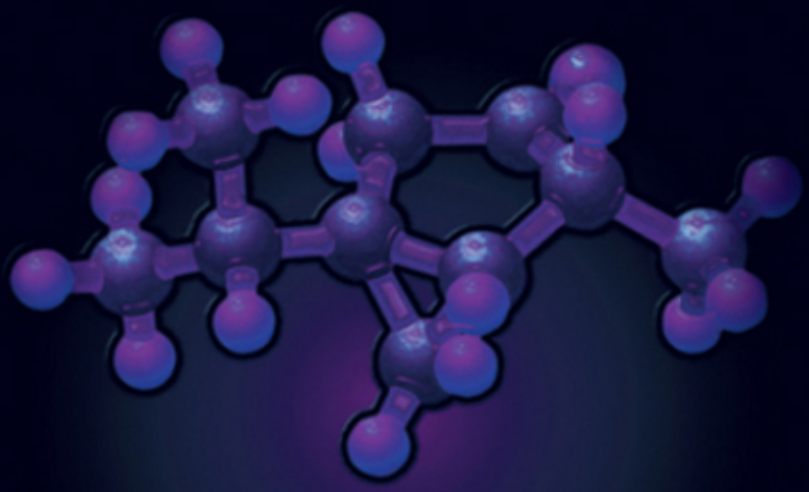

Handbook of Reagents for Organic Synthesis

Sulfur-Containing Reagents

Edited by

Leo A. Paquette

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for Organic Synthesis

Sulfur-Containing Reagents

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Sulfur-Containing Reagents

Edited by

Leo A. Paquette
The Ohio State University, Columbus, OH, USA

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This volume is dedicated to Dr. Gerald Berkelhammer who introduced me to the field of organosulfur chemistry during summer employment (1957) as a fledgling graduate student while working at the American Cyanamid Company in Stamford, Connecticut.

e-EROS Editorial Board

Contents

Preface

The eight-volume *Encyclopedia of Reagents for Organic Synthesis* (*EROS*), authored and edited by experts in the field, and published in 1995, had the goal of providing an authoritative multivolume reference work describing the properties and reactions of approximately 3000 reagents. With the coming of the Internet age and the continued introduction of new reagents to the field as well as new uses for old reagents, the electronic sequel, *e-EROS*, was introduced in 2002 and now contains in excess of 4000 reagents, catalysts, and building blocks making it an extremely valuable reference work. At the request of the community, the second edition of the encyclopedia, *EROS II*, was published in March 2009 and contains the entire collection of reagents at the time of publication in a 14 volume set.

Although the comprehensive nature of *EROS* and *EROS II* and the continually expanding *e-EROS* render them invaluable as reference works, their very size limits their practicability in a laboratory environment. For this reason, a series of inexpensive one-volume *Handbooks of Reagents for Organic Synthesis* (*HROS*), each focused on a specific subset of reagents, was introduced by the original editors of *EROS* in 1999:

Reagents, Auxiliaries, and Catalysts for C–C Bond Formation
Edited by Robert M. Coates and Scott E. Denmark

Oxidizing and Reducing Agents
Edited by Steven D. Burke and Rick L. Danheiser

Acidic and Basic Reagents
Edited by Hans J. Reich and James H. Rigby

Activating Agents and Protecting Groups
Edited by Anthony J. Pearson and William R. Roush

This series has continued over the past several years with the publication of another series of *HROS* volumes, each edited by a current or past member of the *e-EROS* editorial board:

Chiral Reagents for Asymmetric Synthesis
Edited by Leo A. Paquette

Reagents for High-Throughput Solid-Phase and Solution-Phase Organic Synthesis
Edited by Peter Wipf

Reagents for Glycoside, Nucleotide, and Peptide Synthesis
Edited by David Crich

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Fluorine-Containing Reagents
Edited by Leo A. Paquette

Catalyst Components for Coupling Reactions
Edited by Gary A. Molander

Reagents for Radical and Radical Ion Chemistry
Edited by David Crich

This series now continues with this volume entitled *Sulfur-Containing Reagents*, edited by Leo Paquette, the originator and longtime guiding light of the *EROS* and *HROS* volumes, in addition to the online version, *e-EROS*. This 12th volume in the *HROS* series, like its predecessors, is intended to be an affordable, practicable compilation of reagents arranged around a central theme that it is hoped will be found at arm's reach from synthetic chemists worldwide. The reagents have been selected to give broad relevance to the volume, within the limits defined by the subject matter of organosulfur reagents. We have enjoyed putting this volume together and hope that our colleagues will find it as much enjoyable and useful to read and consult.

David Crich
Centre de Recherche de Gif-sur-Yvette
Institut de Chimie des Substances Naturelles
Gif-sur-Yvette, France

Introduction

The widespread use of sulfur-containing reagents by synthetic chemists is a reflection of the diversity of properties offered by this broad class of compounds. The enormous divergence in the reactivity of divalent and tetravalent sulfur reagents, the stereochemical issues offered by sulfoxides, and the ionizability of sulfonic acids represent only a few of the many facets of this collective group of reagents. The resulting demands placed on chemists for the appropriate adaptation of a given reagent emerge in many forms, not the least of which bear on one's knowledge of reactivity, availability of building blocks, appreciation of the consequences of associated changes in electronic character, and the like. As a consequence of many recent developments, it was deemed appropriate to incorporate into a single volume a compilation that brings together a large fraction of the more useful organosulfur reagents as well as key sulfur-containing promoters that are currently in vogue. As usual, the compilation has been arranged alphabetically in order to facilitate searching. An added benefit is to foster the scanning for information since proximal arrangements by compound type are not prevalent.

The selection covered in this volume is built on the many important discoveries at the hands of numerous practitioners of our science. These achievements have been realized in conjunction with a very extensive physical organic base.

The featured reagents have been culled from three sources. A minority of the entries appeared initially in the *Encyclopedia of Reagents for Organic Synthesis (EROS)*, which was published in 1995. Since that time, a significant number of entries involving these classical reagents have been updated (*e-EROS*), and this important add-on information is also found herein in the form of extensions to the original articles. The third pool of reagents is constituted of entirely new entries that detail the chemical and physical properties of an added subset of sulfur-containing compounds. In some instances, groups of reagents coincidentally appear in close proximity, thereby facilitating comparative analysis. Subsets consisting of *meta*- and *para*-nitrobenzenesulfonyl peroxide and of β-tosylethyl amine, hydrazine, and hydroxylamine are exemplary.

Among the opening segments of this volume is a section that illustrates those procedures relevant to the field that have appeared in volumes 65–85 of *Organic Syntheses*. It is hoped that these tried and tested protocols will provide a useful level of added guidance in selecting what course of action one might pursue. All in all, it is hoped that this handbook will prove to be a valued adjunct to researchers experienced in sulfur chemistry, and a particularly useful compilation for others seeking to gain a foothold in the field as expediently as possible. These goals will have been realized if advances materialize from exposure to its contents.

Leo A. Paquette
Department of Chemistry
The Ohio State University
Columbus, OH, USA

Short Note on InChIs and InChIKeys

The IUPAC International Chemical Identifier (InChI™) and its compressed form, the InChiKey, are strings of letters representing organic chemical structures that allow for structure searching with a wide range of online search engines and databases such as Google and PubChem. While they are obviously an important development for online reference works, such as *Encyclopedia of Reagents for Organic Reactions* (e-EROS), readers of this volume may be surprised to find printed InChi and InChIKey information for each of the reagents.

We introduced InChi and InChIKey to e-EROS in autumn 2009, including the strings in all HTML and PDF files. While we wanted to make sure that all users of e-EROS, the second print edition of EROS and all derivative Handbooks would find the same information, we appreciate that the strings will be of little use to the readers of the print editions, unless they treat them simply as reminders that e-EROS now offers the convenience of InChIs and InChIKeys, allowing the online users to make best use of their browsers and perform searches in a wide range of media.

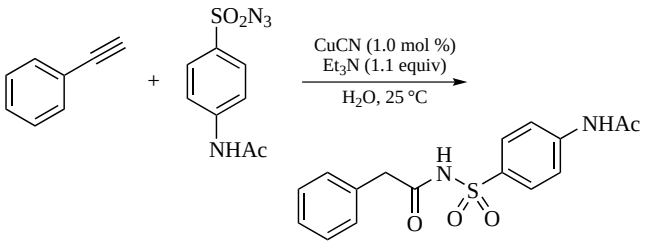
If you would like to know more about InChIs and InChIKeys, please go to the e-EROS website: www.mrw.interscience.wiley.com/eros/ and click on the InChI and InChiKey link.

Organic Synthesis Procedures Featuring the Synthesis of Organosulfur Compounds and Preparative Applications thereof, Volumes 65–85

Synthesis of Organosulfur Compounds

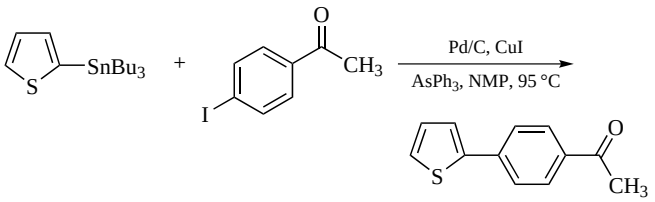
N-(4-Acetamidophenylsulfonyl)-2-phenylacetamide

S. H. Chol, S. J. Hwang, and S. Chang; *Org. Synth.* **2008**, *85*, 131.



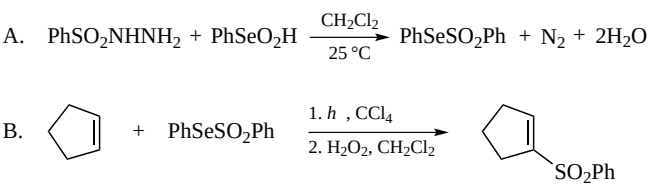
2-(4′-Acetylphenyl)thiophene

L. S. Liebeskind and E. Peña-Cabrera; *Org. Synth.* **2000**, *77*, 135.



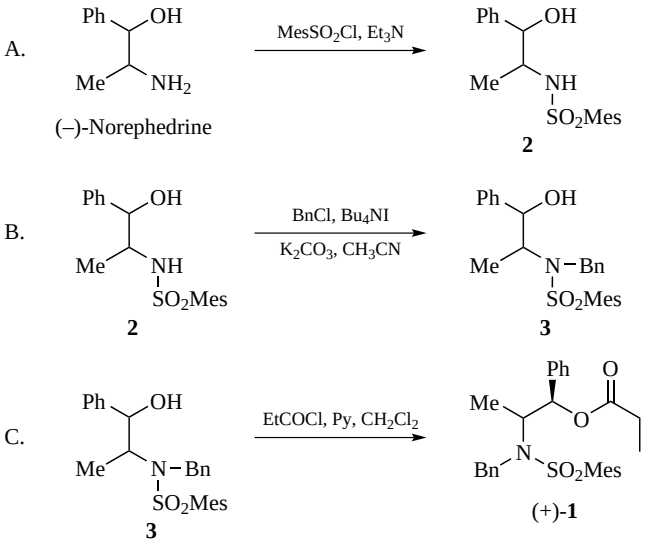
1-(Benzenesulfonyl)cyclopentane

H. S. Lin, M. J. Coghlan, and L. A. Paquette; *Org. Synth.* **1989**, *67*, 157.



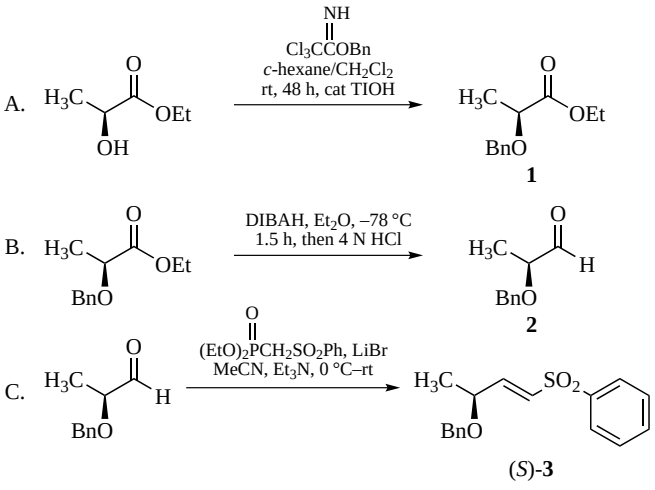
2-(*N*-Benzyl-*N*-mesitylenesulfonyl)amino-1-phenyl-1-propyl Propionate

A. Abiko; *Org. Synth.* **2003**, *19*, 109.



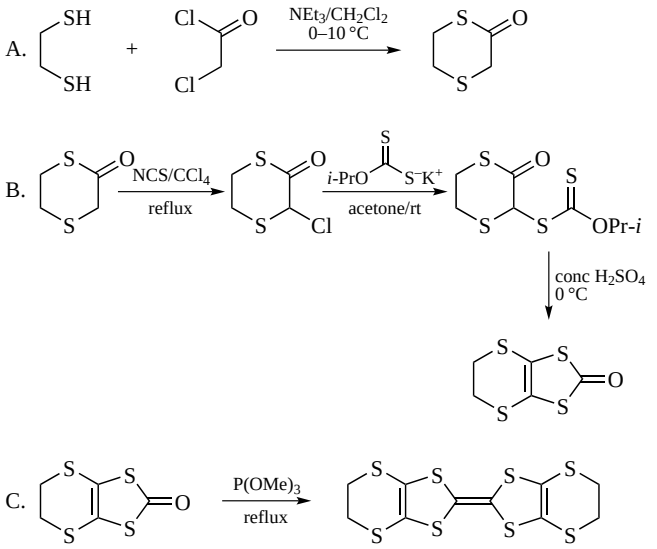
(−)-(*E*,*S*)-3-(Benzyloxy)-1-butenyl Phenyl Sulfone

D. Enders, S. Von Berg, and B. Jandeleit; *Org. Synth.* **2002**, *78*, 177.



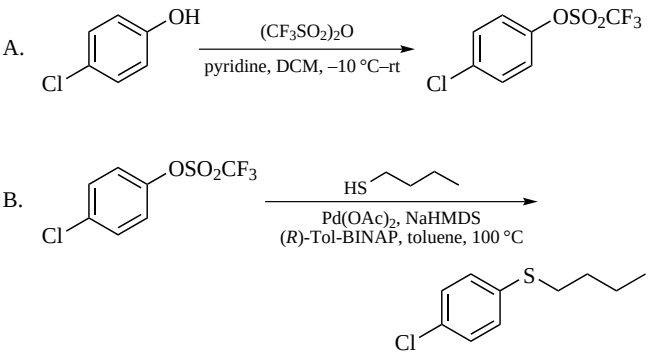
2,2′-Bi-5,6-dihydro-1,3-dithiolo[4,5-*b*][1,4]dithiinylidene (BEDT-TTF)

J. Larsen and C. Lenoir; *Org. Synth.* **1995**, *72*, 265.



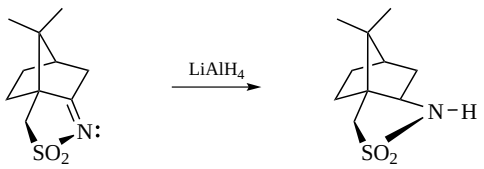
n-Butyl 4-Chlorophenyl Sulfide

J. C. McWilliams, F. Fleitz, N. Zheng, and J. D. Armstrong III; *Org. Synth.* **2003**, *79*, 43.



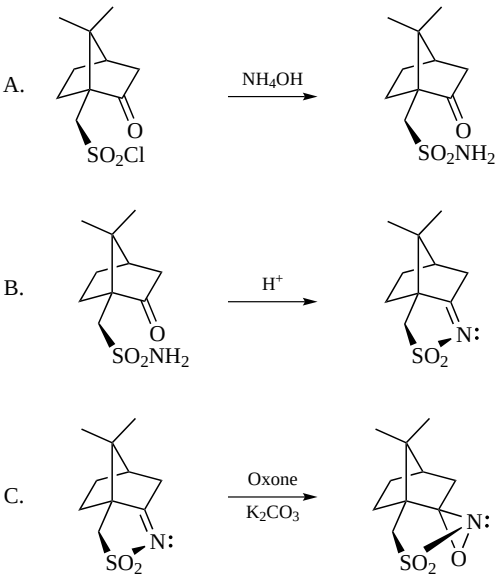
(−)-D-2, 10-Camphorsultam

M. C. Weismiller, J. C. Towson, and F. A. Davis; *Org. Synth.* **1990**, *69*, 154.



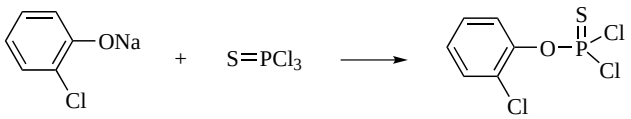
(+)-(2*R*,8a*S*)-10-(Camphorylsulfonyl)oxaziridine

J. C. Towson, M. C. Weismiller, G. S. Lal, A. C. Sheppard, and F. A. Davis; *Org. Synth.* **1990**, *69*, 158.



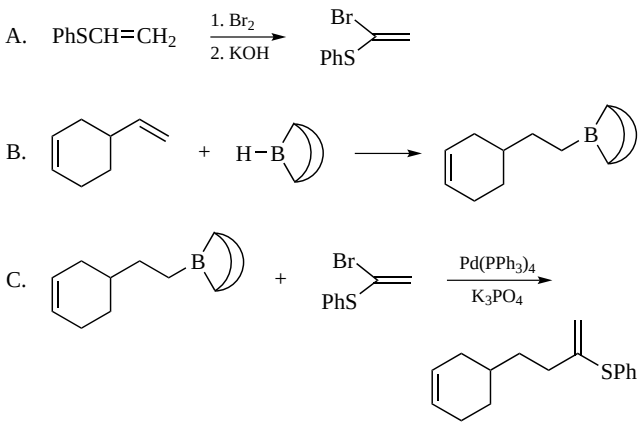
2-Chlorophenyl Phosphorodichloridothioate

V. T. Ravikumar and B. Ross; *Org. Synth.* **1999**, *76*, 271.



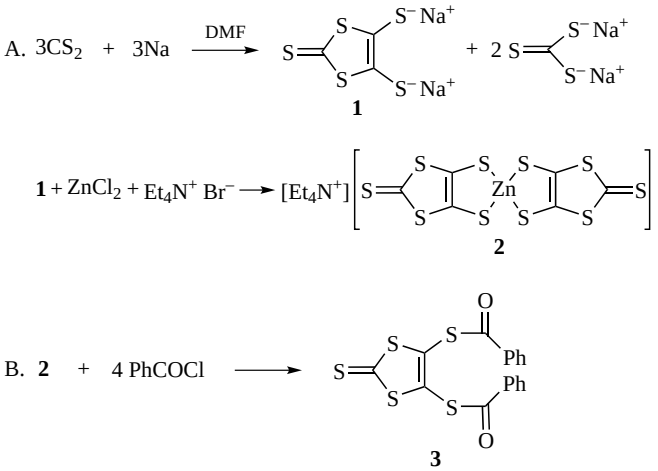
4-(3-Cyclohexenyl)-2-phenylthio-1-butene

T. Ishiyama, N. Miyaura, and A. Suzuki; *Org. Synth.* **1993**, *71*, 89.



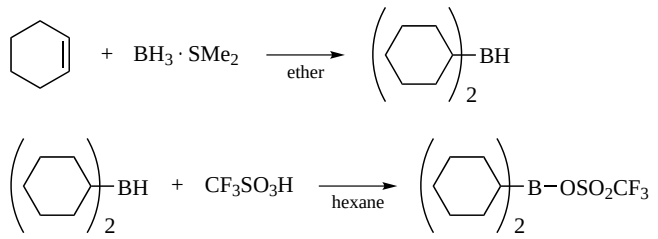
4,5-Dibenzoyl-1,3-dithiole-1-thione

T. K. Hansen, J. Becher, T. Jørgensen, K. S. Varma, R. Khedekar, and M. P. Cava; *Org. Synth.* **1976**, *73*, 270.



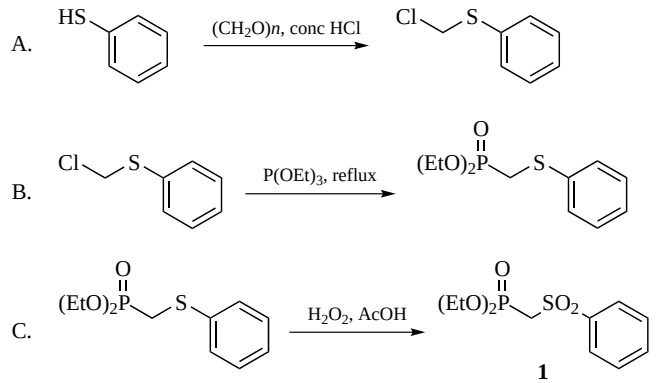
Dicyclohexylboron Trifluoromethanesulfonate

A. Abiko; *Org. Synth.* **2003**, *78*, 103.



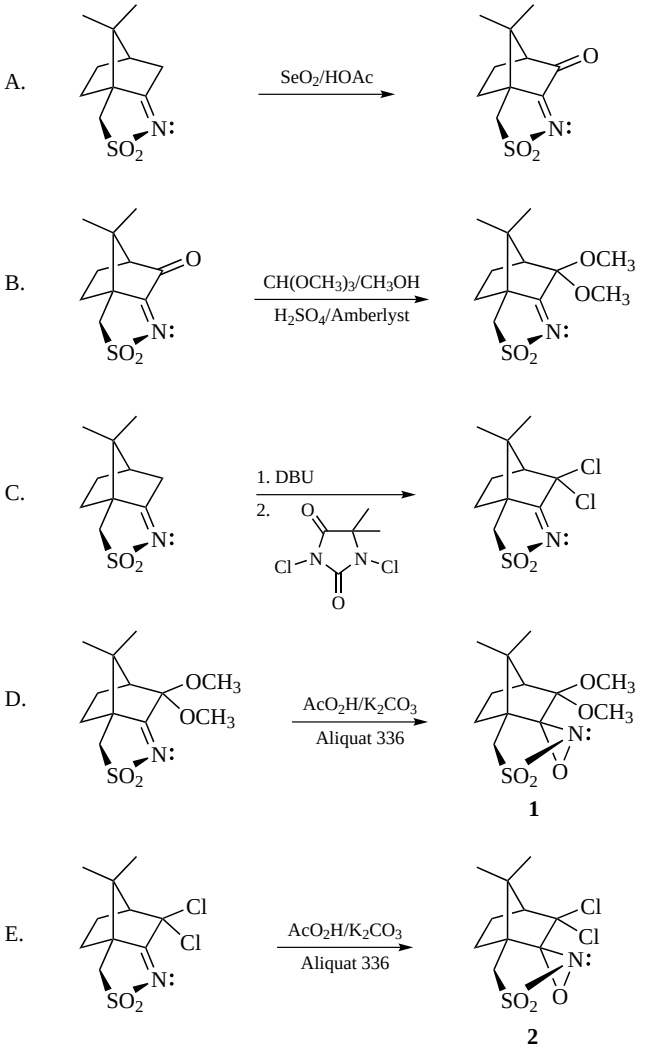
Diethyl [(Phenylsulfonyl)methyl]phosphonate

D. Enders, S. von Berg, and B. Jandeleit; *Org. Synth.* **2002**, *78*, 169.



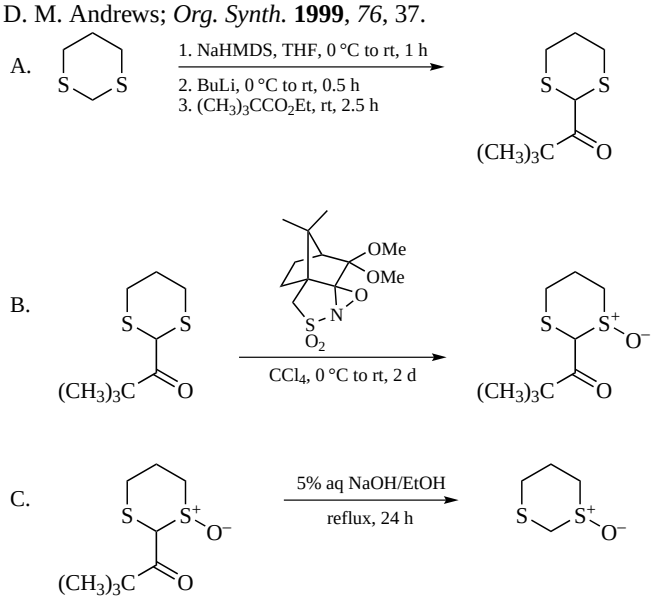
(+)-(2*R*,8a*R)-[(8,8-Dimethoxycamphoryl)sulfonyl]oxaziridine and (+)-(2*R*,8a*R**)-[(8,8-Dichlorocamphoryl)sulfonyl]oxaziridine**

B.-C. Chen, C. K. Murphy, A. Kumar, R. T. Reddy, C. Clark, P. Zhou, B. M. Lewis, D. Gala, I. Mergelsberg, D. Scherer, J. Buckley, D. DiBenedetto, and F. A. Davis; *Org. Synth.* **1995**, *93*, 159.



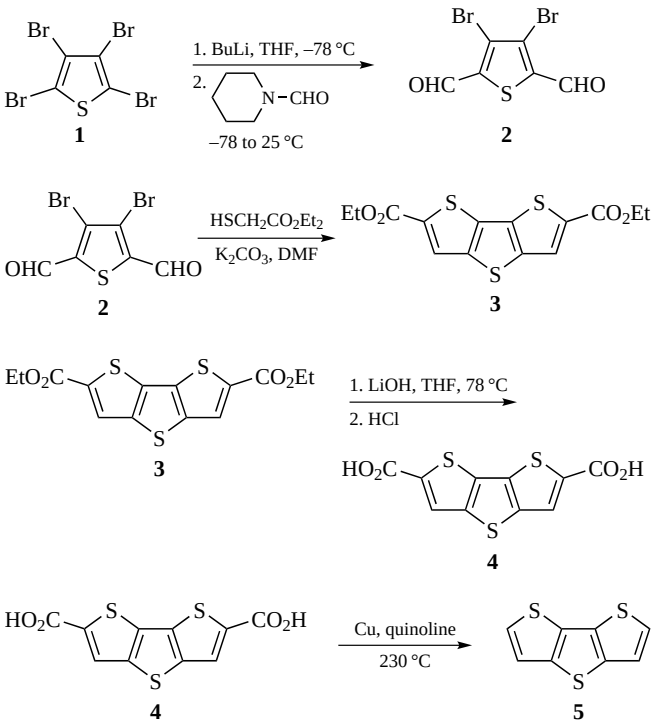
1*S*-(−)-1,3-Dithiane 1-Oxide

P. C. Bulman Page, J. P. Heer, D. Bethell, E. W. Collington, and D. M. Andrews; *Org. Synth.* **1999**, *76*, 37.



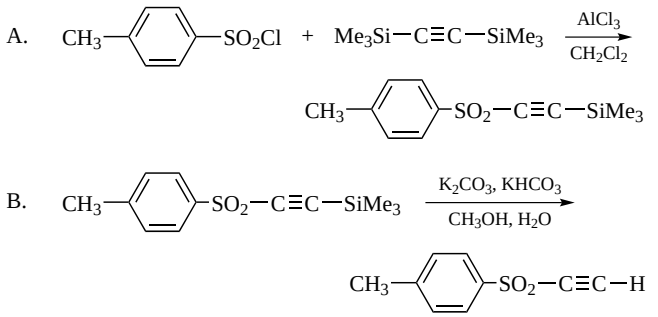
Dithieno[3,2-*b*:2′,3′-*d*]thiophene

J. Frev, S. Proemmel, M. A. Armitage, and A. B. Holmes; *Org. Synth.* **2006**, *83*, 209.



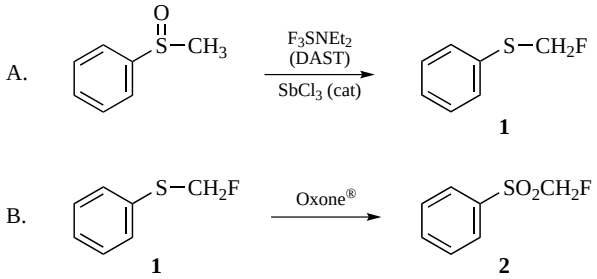
Ethynyl *p*-Tolyl Sulfone

L. Waykole and L. A. Paquette; *Org. Synth.* **1989**, *67*, 149.



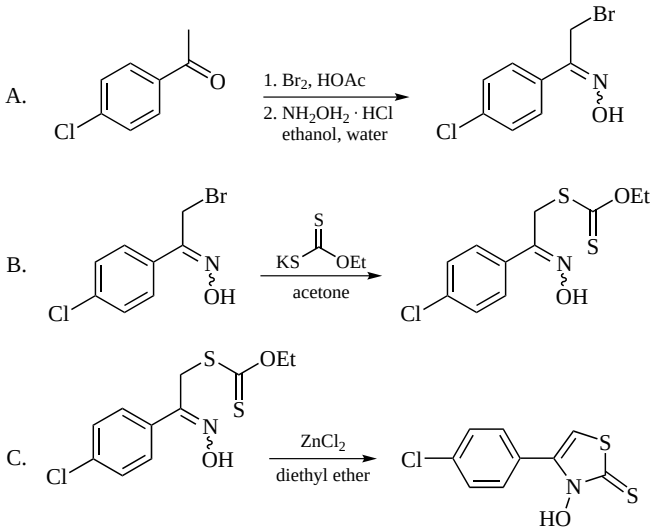
Fluoromethyl Phenyl Sulfone

J. R. McCarthy, D. P. Matthews, and J. P. Paolini; *Org. Synth.* **1995**, *72*, 209.



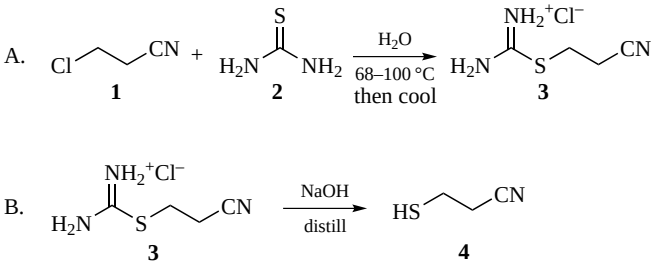
N-Hydroxy-4-(*p*-chlorophenyl)thiazole-2(3*H*)-thione

J. Hartung and M. Schwarz; *Org. Synth.* **2003**, *79*, 228.



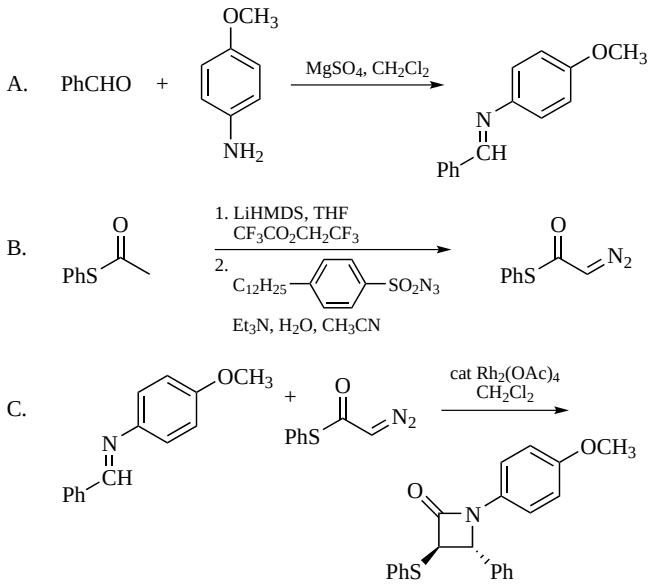
β-Mercaptopropionitrile (2-Cyanoethanethiol)

R. E. Gerber, C. Hasbun, L. G. Dubenko, M. F. King, and D. E. Bierer; *Org. Synth.* **2000**, *77*, 186.



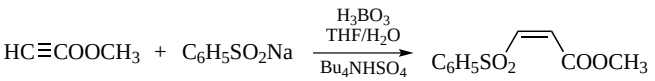
trans-1-(4-Methoxyphenyl)-4-phenyl-3-(phenylthio)azetidin-2-one

R. L. Danheiser, I. Okamoto, M. D. Lawlor, and T. W. Lee; *Org. Synth.* **2003**, *80*, 160.



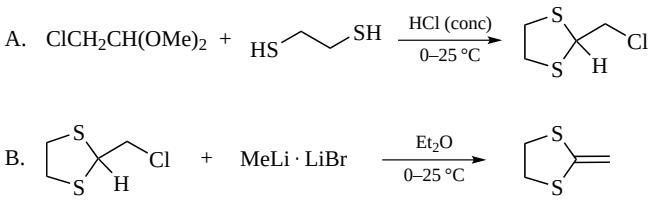
Methyl (*Z*)-3-(Benzenesulfonyl)prop-2-enoate

G. C. Hirst and P. J. Parsons; *Org. Synth.* **1990**, *69*, 169.



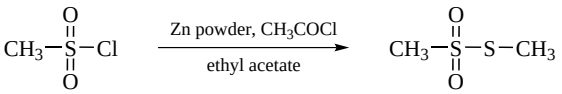
2-Methylene-1,3-dithiolane

K. R. Dahnke and L. A. Paquette; *Org. Synth.* **1993**, *71*, 175.



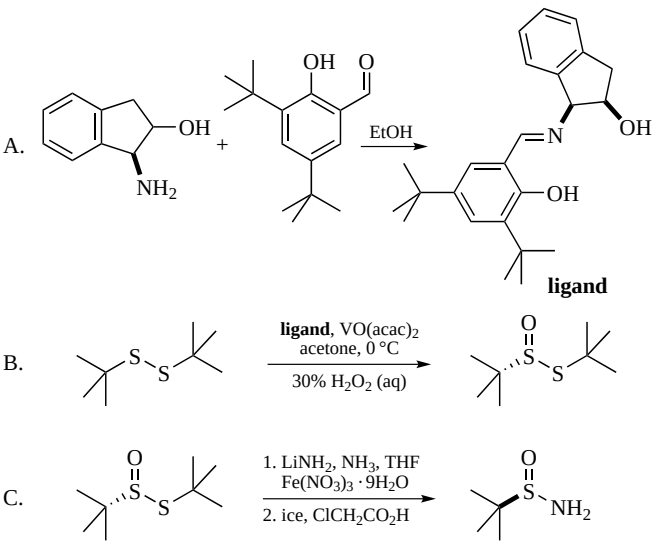
***S*-Methyl Methanethiosulfonate**

F. Chemla and P. Karoyan; *Org. Synth.* **2002**, *78*, 99.



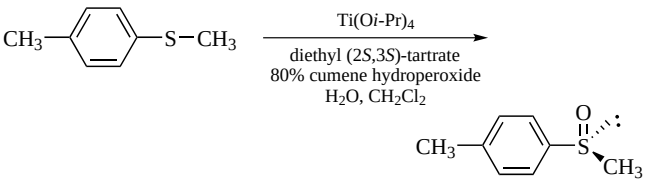
(R_s)-(+)-2-Methyl-2-propanesulfinamide

D. J. Weix and J. A. Ellman; *Org. Synth.* **2005**, *82*, 157.



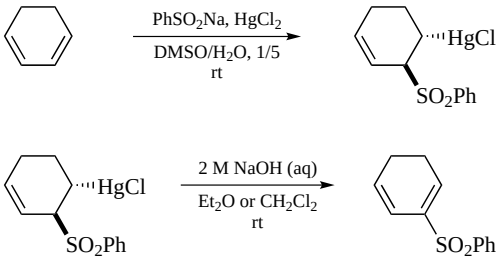
(*S*)-(−)-Methyl *p*-Tolyl Sulfoxide

S. H. Zhao. O. Samuel, and H. B. Kagan; *Org. Synth.* **1990**, *68*, 49.



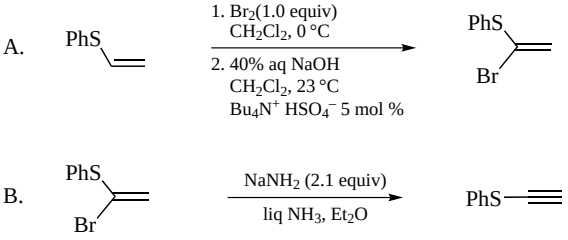
2-(Phenylsulfonyl)-1,3-cyclohexadiene

J.-E. Bäckvall, S. K. Juntunen, and O. S. Andell; *Org. Synth.* **1990**, *68*, 148.



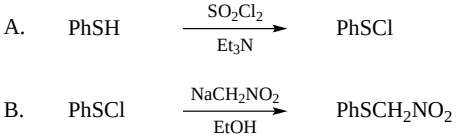
Phenylthioacetylene

P. A. Magriotis and J. T. Brown; *Org. Synth.* **1995**, *72*, 252.



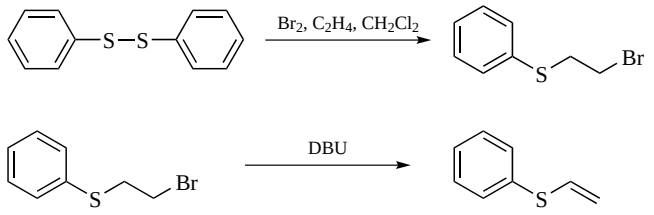
(Phenylthio)nitromethane

A. G. M. Barrett, D. Dhanak, G. G. Graboski, and S. J. Taylor; *Org. Synth.* **1990**, *68*, 8.



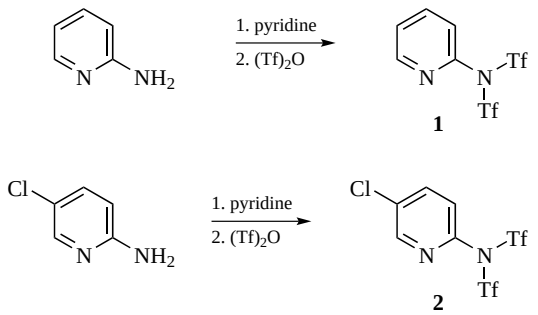
Phenyl Vinyl Sulfide

D. S. Reno and R. J. Pariz; *Org. Synth.* **1997**, *74*, 124.



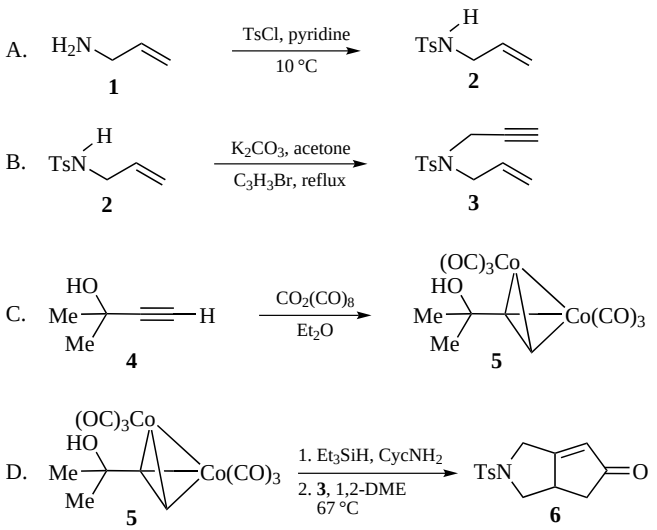
***N*-(2-Pyridyl)triflimide and *N*-(5-Chloro-2-pyridyl)triflimide**

D. L. Comins, A. Dehghani, C. J. Foti, and S. P. Joseph; *Org. Synth.* **1997**, *74*, 77.



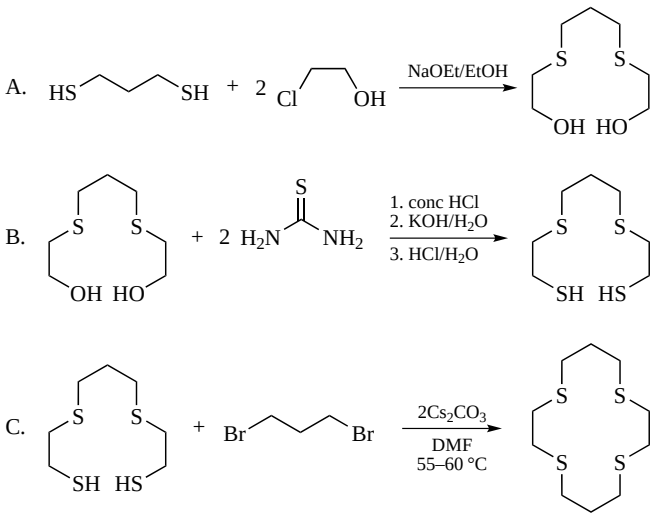
2,3,3α,4-Tetrahydro-2-[(4-methylbenzene) sulfonyl]cyclopenta[*c*]pyrrol-5(1*H*)-one

M. C. Patel, T. Livinghouse, and B. L. Pagenkopf; *Org. Synth.* **2003**, *80*, 93.



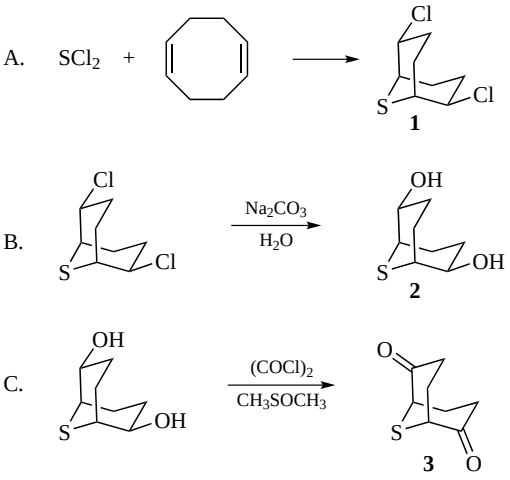
1,4,8,11-Tetrathiacyclotetradecane

J. Buter and R. M. Kellogg; *Org. Synth.* **1987**, *65*, 150.



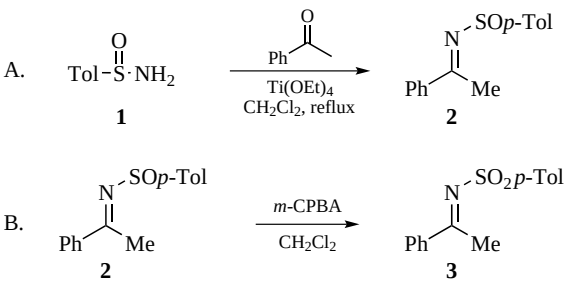
9-Thiabicyclo[3.3.1]nonane-2,6-dione

R. Bishop; *Org. Synth.* **1992**, *70*, 120.



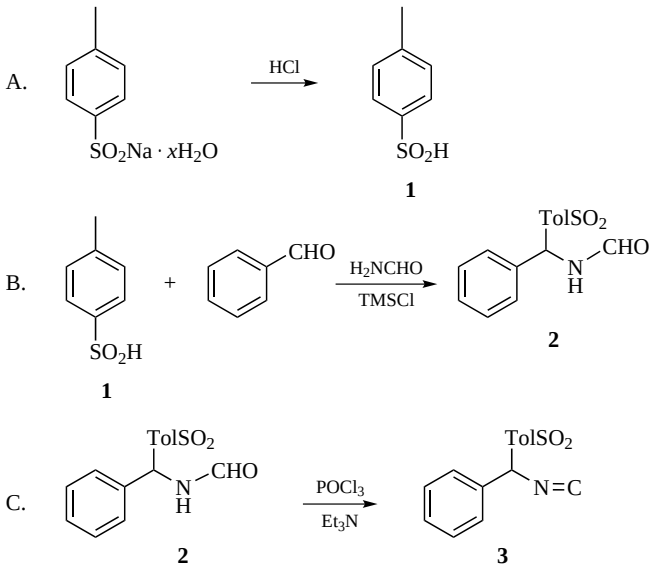
***N*-*p*-Tolylsulfonyl-(*E*)-1-Phenylethylideneimine**

J. L. G. Ruano, J. Aleman, A. Parra, and M. B. Cid; *Org. Synth.* **2007**, *84*, 129.



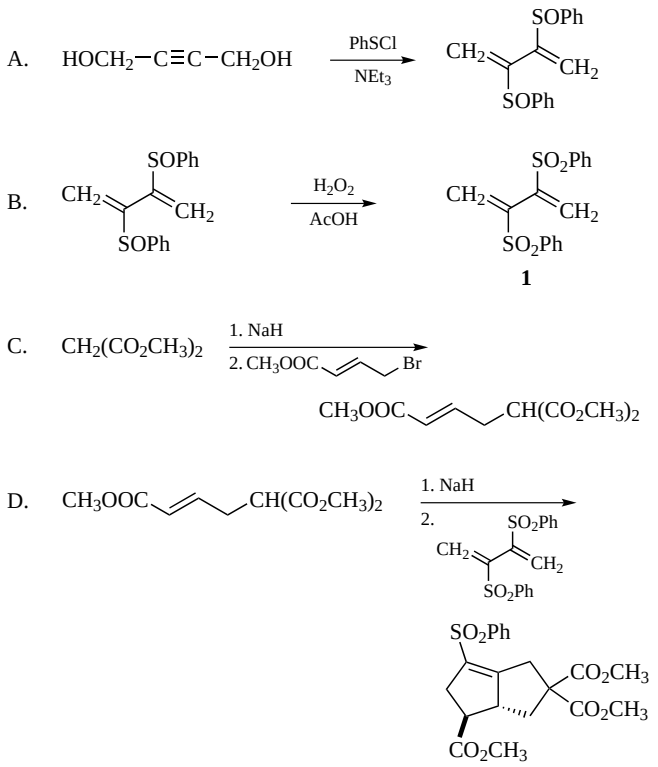
α-Tosylbenzyl Isocyanide

J. Sisko, M. Mellinger, P. W. Sheldrake, and Neil H. Baine; *Org. Synth.* **2000**, *77*, 198.



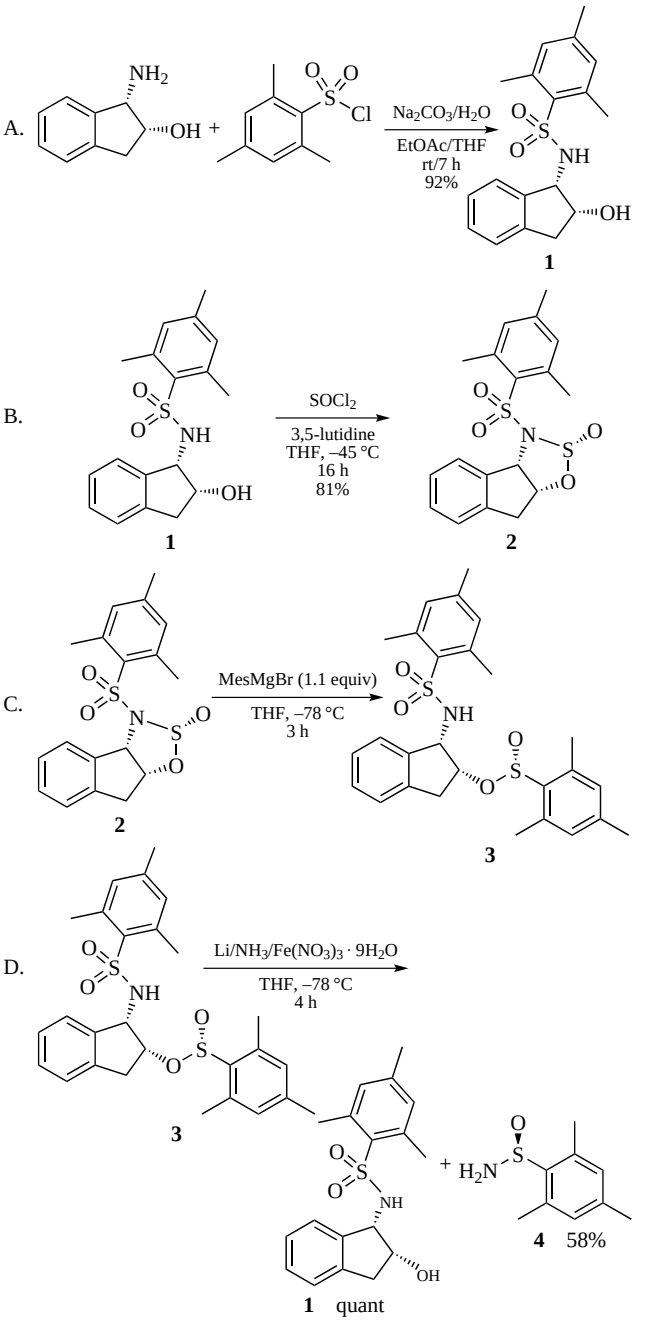
trans-4,7,7-Tricarbomethoxy-2-phenylsulfonylbicyclo[3.3.0]oct-1-ene

A. Padwa, S. H. Watterson, and Z. Ni; *Org. Synth.* **1997**, *74*, 147.



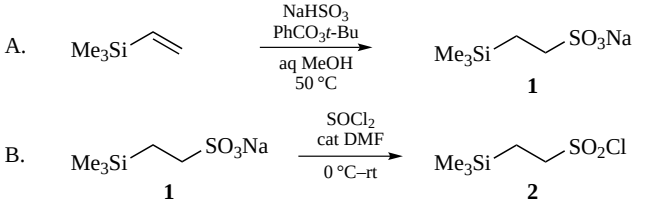
(*S*)-(+)-2,4,6-Trimethylbenzenesulfinamide

T. Ramachandar, Y. Wu, J. Zhang, and F. A. Davis; *Org. Synth.* **2006**, *83*, 131.



2-Trimethylsilylethanesulfonyl Chloride

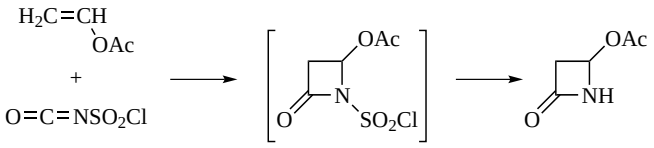
S. M. Weinreb, C. E. Chase, P. Wipf, and S. Venkatraman; *Org. Synth.* **1998**, *75*, 161.



Synthetic Applications of Sulfur-Containing Compounds

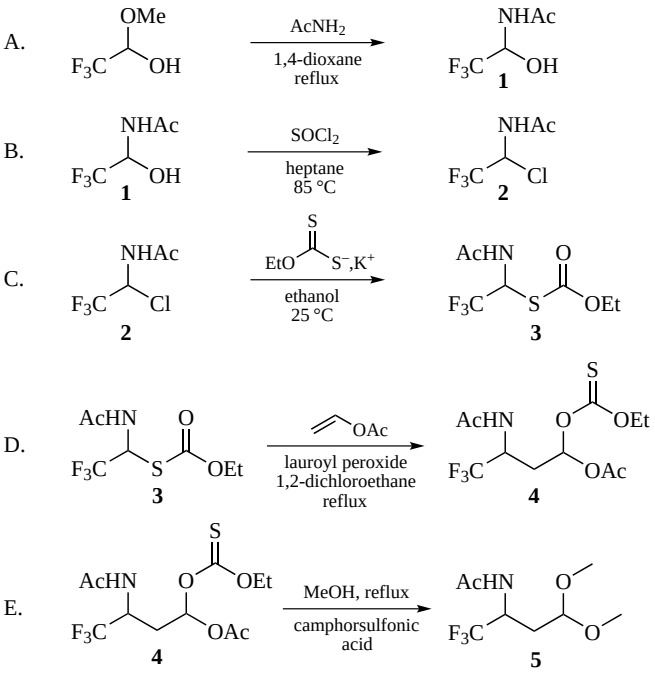
4-Acetoxyazetidin-2-one

S. J. Mickel, C.-N. Hsiao, and M. J. Miller; *Org. Synth.* **1987**, *65*, 135.



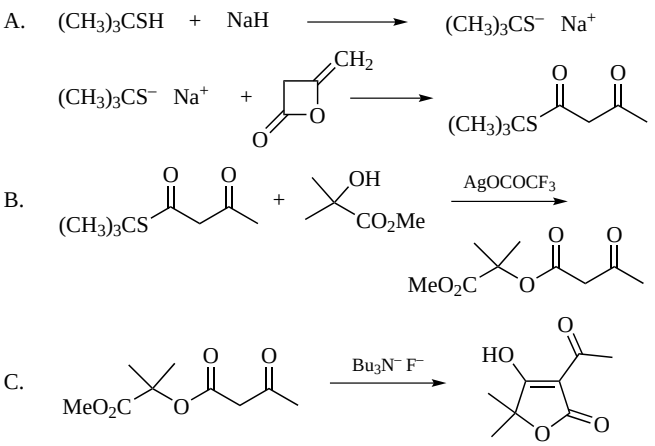
2-(*N*-Acetylamino)-4,4-dimethoxy-1,1,1-trifluorobutane

F. Gagosz and S. Z. Zard; *Org. Synth.* **2007**, *84*, 32.



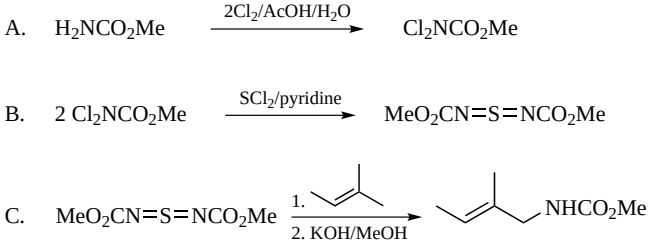
3-Acetyl-4-hydroxy-5,5-dimethylfuran-2(5*H*)-one

C. M. J. Fox and S. V. Ley; *Org. Synth.* **1988**, *66*, 108.



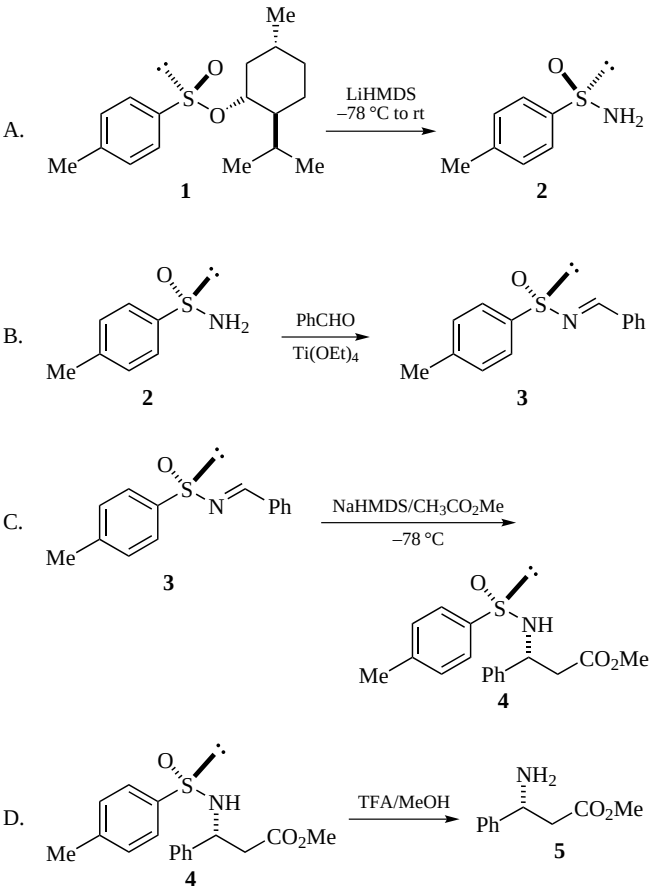
Allylcarbamates by the Aza-ene Reaction: Methyl *N*-(2-methyl-2-butenyl)carbamate

G. Kresze, H. Braxmeier, and H. Münsterer; *Org. Synth.* **1987**, *65*, 159.



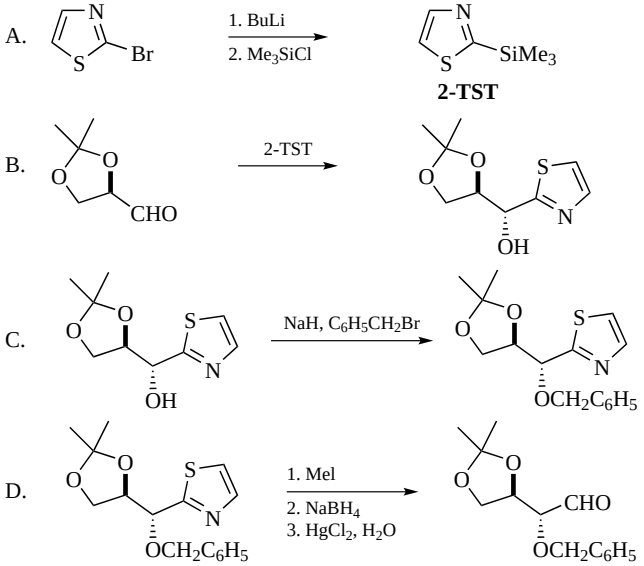
Asymmetric Synthesis of Methyl (*R*)-(+)-*β*-Phenylalanate from (*S*)-(+)*N*-(Benzylidene)-*p*-toluenesulfinamide

D. L. Fanelli, J. M. Szewczyk, Y. Zhang, G. V. Reddy, D. M. Burns, and F. A. Davis; *Org. Synth.* **2000**, *77*, 50.



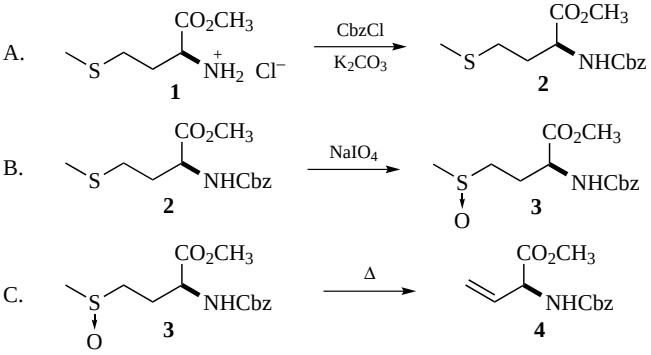
2-*O*-Benzyl-3,4-isopropylidene-D-erythrose

A. Dondoni and P. Merino; *Org. Synth.* **1995**, *72*, 21.



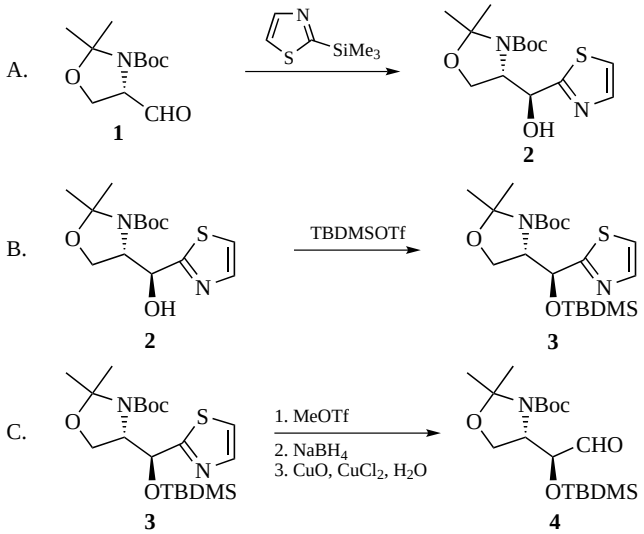
N-(Benzyloxycarbonyl)-L-vinylglycine Methyl Ester

M. Carrasco, R. J. Jones, S. Kamel, H. Rapoport; *Org. Synth.* **1992**, *70*, 29.



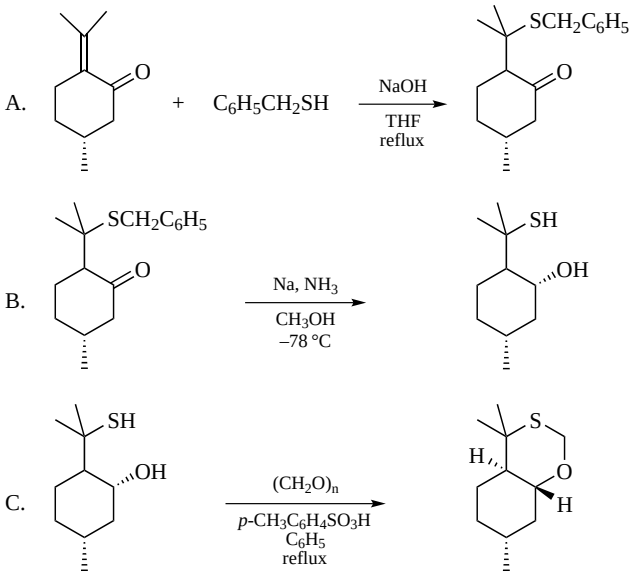
(*S*)-2-[(4*S*)-*N*-*tert*-Butoxycarbonyl-2,2-dimethyl-1,3-oxazolidinyl]-2-*tert*-butyldimethylsiloxyethanal

A. Dondoni and D. Perrone; *Org. Synth.* **2000**, *77*, 78.



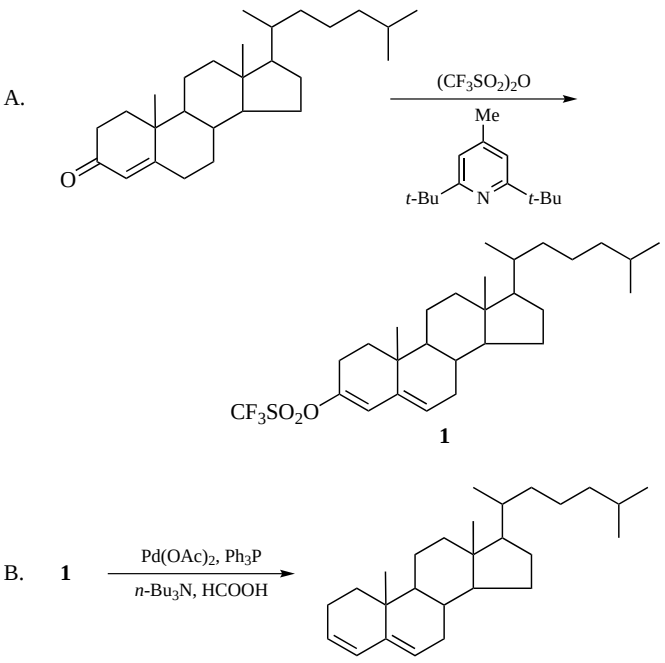
Chiral 1,3-Oxathiane from (+)-Pulegone: Hexahydro-4,4,7-Trimethyl-4*H*-1,3-benzoxathiin

E. L. Eliel, J. E. Lynch, F. Kume, and S. V. Frye; *Org. Synth.* **1987**, *65*, 215.



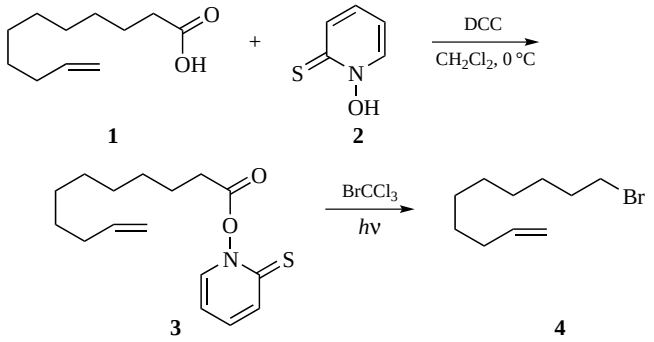
Cholesta-3,5-diene

S. Cacchi, E. Morera, and G. Ortar; *Org. Synth.* **1990**, *68*, 138.



Dec-9-enyl Bromide from 10-Undecenoic Acid

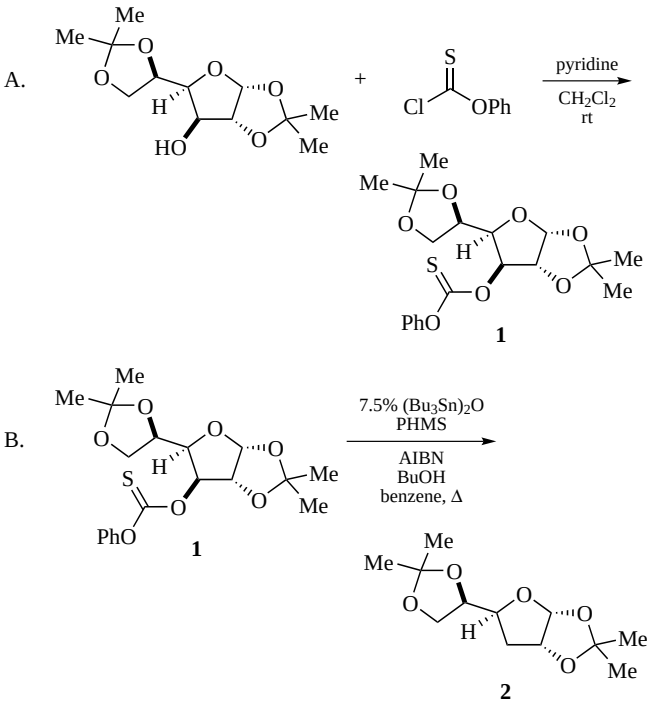
D. H. R. Barton, J. MacKinnon, R. N. Perchel, and C.-L. Tse; *Org. Synth.* **1998**, *75*, 124.



1 **2** **3** **4**

3-Deoxy-1,2:5,6-bis-*O*-(1-methylethylidene)-α-D-ribo-hexofuranose

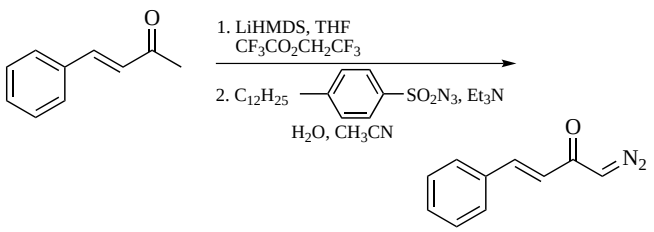
J. Tormo and G. C. Fu; *Org. Synth.* **2002**, *78*, 239.



1 **2**

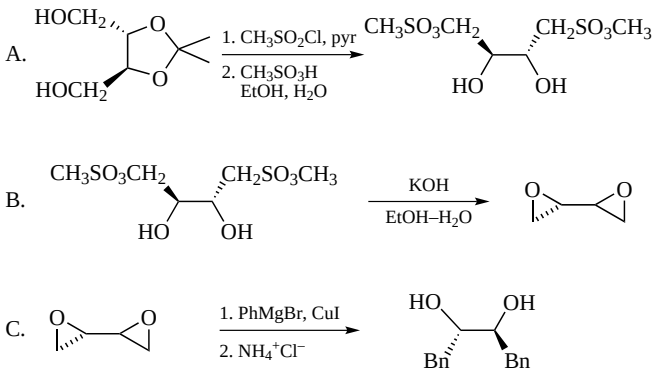
(*E*)-1-Diazo-4-phenyl-3-buten-2-one

R. L. Danheiser, R. F. Miller, and R. G. Brisbois; *Org. Synth.* **1995**, *73*, 134.



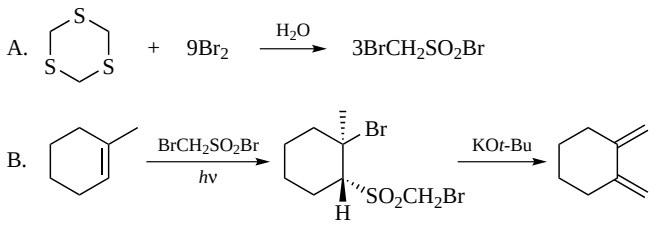
(2*S*,3*S*)-Dihydroxy-1,4-diphenylbutane

M. A. Robbins, P. N. Devine, and T. Oh; *Org. Synth.* **1999**, *76*, 101.



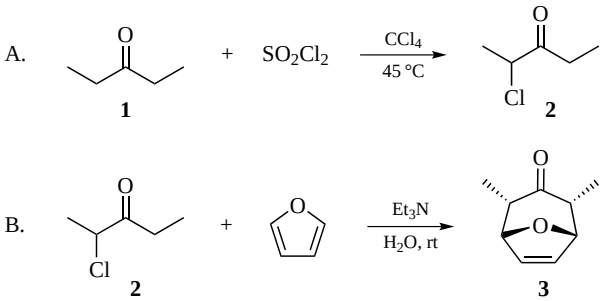
1,2-Dimethylenecyclohexane

E. Block and M. Aslam; *Org. Synth.* **1987**, *65*, 90.



2,4-*endo*,*endo*-Dimethyl-8-oxabicyclo[3.2.1]oct-6-en-3-one

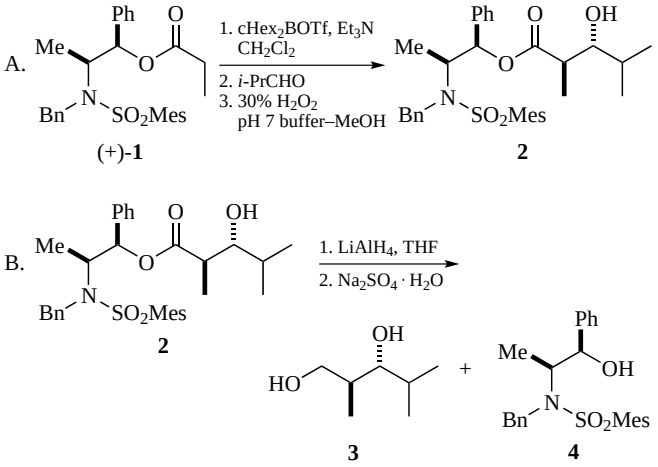
M. Lautens and G. Bouchain; *Org. Synth.* **2003**, *79*, 251.



1 **2** **3**

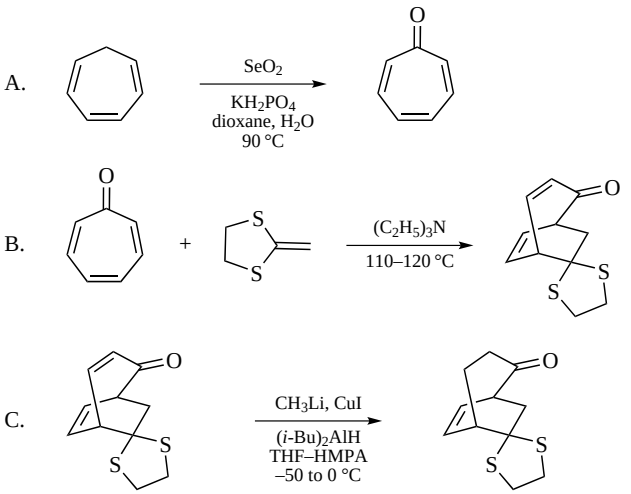
(2*S*,3*R*)-2,4-Dimethyl-1,3-pentanediol

A. Abiko; *Org. Synth.* **2003**, *79*, 116.



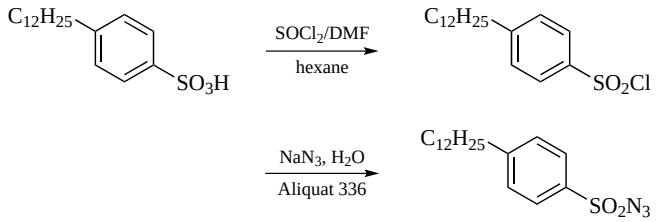
9-Dithiolanobicyclo[3.2.2]non-6-en-2-one

K. R. Dahnke and L. A. Paquette; *Org. Synth.* **1993**, *71*, 181.



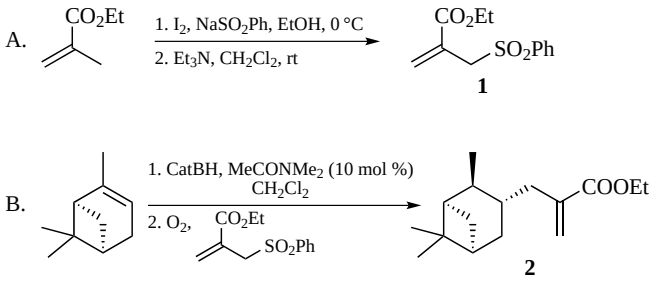
4-Dodecylbenzenesulfonyl Azides

G. G. Hazen, F. W. Bollinger, F. E. Roberts, W. K. Russ, J. J. Seman, and S. Staskiewicz; *Org. Synth.* **1995**, *73*, 144.



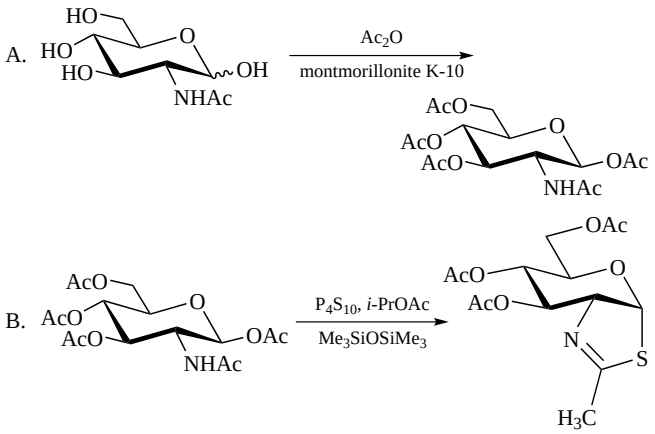
Ethyl 2-{[(1*S*,2*R*,3*R*,5*S*-2,6,6-Trimethylbicyclo[3.1.1]hept-3-yl]methyl}acrylate

V. Darmency, E. M. Scanlan, A. P. Schaffner, and P. Renaud; *Org. Synth.* **2006**, *83*, 24.



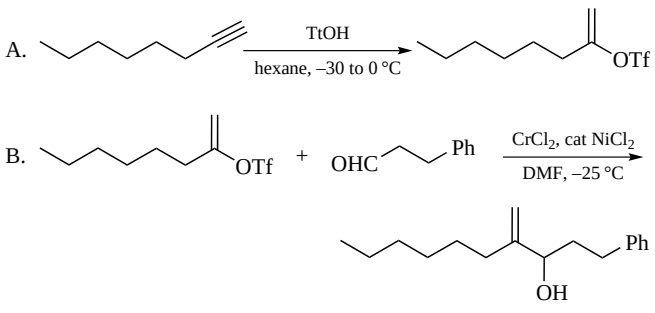
GlcNAc-thiazoline Triacetate

S. Knapp, R. A. Huhn, and B. Amorelli; *Org. Synth.* **2007**, *84*, 68.



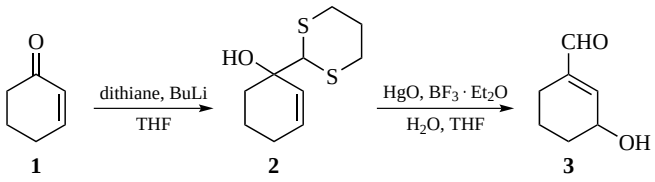
2-Hexyl-5-phenyl-1-penten-3-ol

K. Takai, K. Sakogawa, Y. Kataoka, K. Oshima, and K. Utimoto; *Org. Synth.* **1995**, *72*, 180.



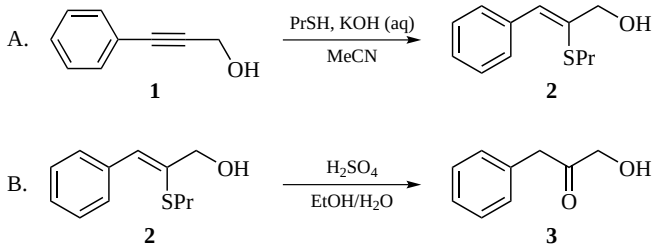
3-Hydroxy-1-cyclohexene-1-carboxaldehyde

H. L. Rigby, M. Neveu, D. Pauley, B. C. Ranu, and T. Hudlicky; *Org. Synth.* **1989**, *67*, 205.



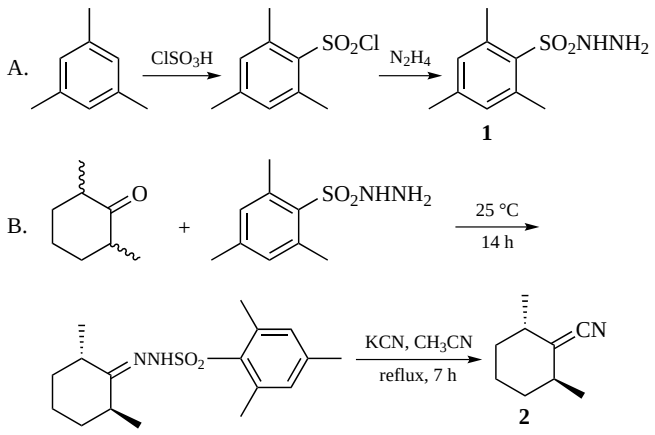
1-Hydroxy-3-phenyl-2-propanone

M. S. Waters, K. Snelgrove, and P. Maligres; *Org. Synth.* **2003**, *80*, 190.



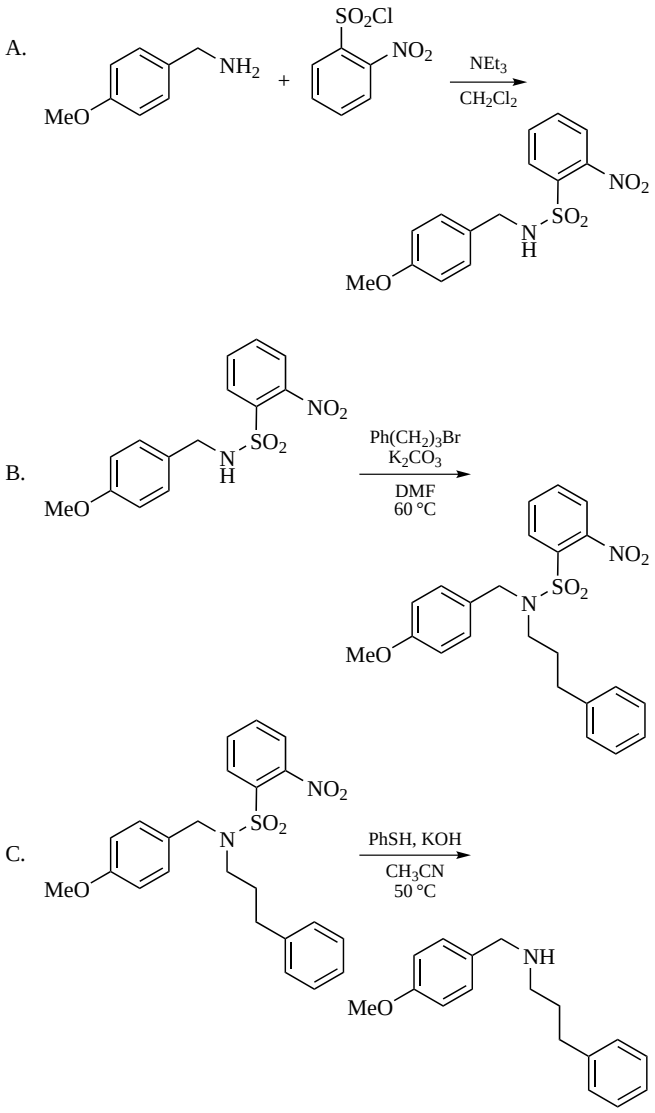
Mesitylenesulfonylhydrazine, and (1α,2α,6β)-2,6-Dimethylcyclohexanecarbonitrile and (1α,2β,6α)-2,6-Dimethylcyclohexanecarbonitrile as a Racemic Mixture

J. R. Reid, R. F. Dufresne, and J. J. Chapman; *Org. Synth.* **1997**, *74*, 217.



N-(4-Methoxybenzyl)-3-phenylpropylamine

W. Kurosawa, T. Kan, and T. Fukuyama; *Org. Synth.* **2003**, *79*, 186.



5-Methyl-2,2′-bipyridine

A. P. Smith, S. A. Savage, J. C. Love, and C. L. Fraser; *Org. Synth.* **2002**, *78*, 51.