DIASTEREOSELECTIVE [2,3] WITTIG REARRANGEMENT OF CARBOHYDRATE-DERIVED TERTIARY ALLYLIC ETHERS. 1.

SYNTHESIS OF THE C<sub>11</sub>-C<sub>18</sub> SUBUNIT OF HERBIMYCIN A FROM D-GLUCOSE. Jill E. Eshelman, Janet L. Epps and James Kallmerten, Department of Chemistry, Syracuse University, Syracuse NY 13244-4100 USA

Intermediate 1, comprising the  $C_{11}$ - $C_{18}$  subunit of herbirnycin A, is prepared by reductive fragmentation of a D-alucose -derived iodopyranose and subsequent sigmatropic homologation of the resulting tertiary allylic alcohol.

Tetrahedron Lett. 1993, 34, 753

DIASTEREOSELECTIVE [2,3] WITTIG REARRANGEMENT OF

CARBOHYDRATE-DERIVED TERTIARY ALLYLIC ETHERS. 2.
SYNTHESIS OF AN ADVANCED RAPAMYCIN INTERMEDIATE FROM D-GLUCOSE. Ny Sin and James Kallmerten, Department of Chemistry, Syracuse University, Syracuse NY 13244-4100 USA

The 13-carbon rapamycin intermediate 1 has been prepared by an iterative sigmatropic sequence incorporating the diastereoselective [2,3] Wittig rearrangement of a D-glucofuranose-derived tertiary allytic ether.

DIRECTABLE REGIOCHEMISTRY IN NAPHTHAZARINS VIA THE USE OF MASKED DERIVATIVES J. L. Bloomer\* and K. W. Stagliano Temple University, Department of Chemistry, Philadelphia, PA 19122

Tetrahedron Lett. 1993, 34, 757

The synthesis of the masked naphthazarin I is reported and its conversion to the masked leuconapthazarin II, free naphthazarin III and nucleophilic addition product IV are described.

Tetrahedron Lett. 1993, 34, 761

NOSTOCLIDE I AND II, EXTRACELLULAR METABOLITES FROM A SYMBIOTIC CYANOBACTERIUM, NOSTOC SP. FROM THE LICHEN PELTIGERA CANINA.

Xuemin Yang and Yuzuru Shimizu\*

Department of Pharmacognosy and Environmental Health Sciences, College of Pharmacy, The University of Rhode Island, Kingston, R.I. 02881.

Jorge Rios Steiner and Jon Clardy

Department of Chemistry, Baker Laboratory, Cornell University, Ithaca, N.Y. 14853-1301

Two novel chlorine-containing metabolites were isolated from a lichen blue-green alga.

#### REGIOSELECTIVITY IN GAS-PHASE ELIMINATION REACTIONS: 1,2 VS 1,4 AND ALLENIC ION VS DIENIDE **FORMATION**

John J. Rabasco and Steven R. Kass\*, Department of Chemistry, University of Minnesota, Minneapotis, MN 55455

Competition between the different elimination pathways in deuterium labeled 1-allyl-1-methoxy-6,6-dimethyl-2-cyclohexene (1), 1methoxy-4,4-dimethyl-1-vinyl-2-cyclohexene (2) and 3-methoxy-3-methyl-1-butene (3) have been investigated.

### Synthesis of Short Chain Phosphatidylinositols

Tetrahedron Lett. 1993, 34, 769

Venkata R. Garigapati and Mary F. Roberts, Dept of Chemistry, Boston College, Chestnut Hill, MA 02176 USA

Short Chain Phosphatidylinositols were achieved in high quantities via H-Phosphonate using highly efficient coupling agent, NPCI

#### REGIOSELECTIVE NUCLEOPHILIC RING OPENING OF OXABICYCLIC COMPOUNDS.

Tetrahedron Lett. 1993, 34, 773

Mark Lautens\*, Pauline Chiu

Department of Chemistry, University of Toronto, Toronto, Ontario Canada M5S 1A1

The effect of substituents on the regioselectivity of the ring opening reaction of oxabicyclo[3.2.1] compounds with organolithium reagents has been investigated. Attack of the nucleophile occurs distal to the bridgehead substituent, generating compounds with trisubstituted olefins, 1. Subsequent oxidative cleavage of the olefin provides acyclic fragments with differentiated termini, 2 or 3.

## THE MOST CONVENIENT METHOD FOR THE PREPARATION OF **ALIPHATIC CRYPTANDS**

Tetrahedron Lett. 1993, 34, 777

Krzysztof E. Krakowiak, Patrycja A. Krakowiak, and Jeraid S. Bradehaw \* Department of Chemistry, Brigham Young University, Provo UT 84602-1022 U.S. A

#### REGIOSPECIFIC CONVERSION OF TERMINAL ALKYNES TO

Tetrahedron Lett. 1993, 34, 779

KETOL PHOSPHATES WITH AN IODINE(E)-PHOSPHATE REAGENT

Gerald F. Koser\*, Xiao Chen, Kuanchiang Chen and Guoping Sun

Department of Chemistry, The University of Akron, Akron, Ohio 44325 U.S.A.

Various terminal alkynes were converted regiospecifically to ketol phosphates 3 with the iodine(III)-phosphate 2 in acetonitrile containing water.

## Allylboronic Ester Synthesis: A Mechanistic Study

Tetrahedron Lett. 1993, 34, 783

George W. Kabalka\* and R. David Pace Department of Chemistry, University of Tennessee, Knoxville, Tennessee 37996-1600

The selective formation of allylboronic esters via the allylation of trialkylborates using allylmagnesium bromide was found to be temperature dependent. At -78°C, allylmagnesium bromide reacts cleanly with triisopropylborate to provide allyldiisopropoxyborane.

Tetrahedron Lett. 1993, 34, 785

Enantioselective Oxazaborolidine Reduction of Ketones Containing Heteroatoms George J. Quallich\* and Teresa M. Woodall Process Research and Development, Central Research Division, Pfizer Inc., Groton, CT 06340

Ketones which contain heteroatoms, particularly nitrogen, can be enantioselectively and catalytically reduced with chiral oxazaborolidines in the presence of excess horane.

Tetrahedron Lett. 1993, 34, 789

AN INTRAMOLECULAR 4+3 CYCLOADDITION-VINYLOGOUS GROB FRAGMENTATION ROUTE TO A TRICYCLO[6.3.0.02,4] UNDECENE

RING SYSTEM. Michael Harmata\* and Saleh Elahmad, Department of Chemistry, University of Missouri-Columbia, Columbia, Missouri 65211 USA

Treatment of 4+3 cycloadduct 2a with excess n-BuLi followed by triflic anhydride results in the formation of 10.

#### INTERMEDIATES IN THE DYE-SENSITIZED PHOTO-OXYGENATION OF NITRONES AND HYDRAZONES

Ihsan Erden,\* Albert Griffin, James R.Keeffe and Vibeke Brinck-Kohn San Francisco State University, Department of Chemistry and Biochemistry, San Francisco, CA 94132

In the dye-sensitized photooxygenation of C-aryl nitrones and hydrazones short-lived intermediates of the type 1,2,3,4, respectively, are observed. The structures of these intermediates clearly limit the mechanistic possibilities for the reactions of these compounds with <sup>1</sup>O<sub>2</sub>.

Tetrahedron Lett. 1993, 34, 797

FREE RADICAL CYCLIZATIONS LEADING TO NITROGEN HETEROCYCLES.
II. 2-PHENYLPYRAZOLIDIN-3-ONES VIA TRIBUTYLTIN HYDRIDE REACTION WITH ALPHA-BROMOACYLATED PHENYLHYDRAZONES

John L. Belletire,\* Chad E. Hagedorn, Douglas M. Ho, Jeanette Krause

Department of Chemistry, University of Cincinnati Cincinnati, OH 45221-0172

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AN EFFICIENT PREPARATION OF (+)-TRANS-2-(a-CUMYL)CYCLO-HEXANOL ((+)-TCC): A PRACTICAL ALTERNATIVE TO (+)-8-PHENYLMENTHOL. Tetrahedron Lett. 1993, 34, 801

Daniel L. Comins\* and James M. Salvador, Department of Chemistry, North Carolina State University, Raleigh, NC 27695-8204 USA.

An efficient preparation and resolution of trans-2-(α-cumyl)cyclohexanol gives the (+)-enantiomer in three steps from cyclohexene oxide.

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", Y"

enzymatic resolution

(-)-isomer ( laurate ester )

Tetrahedron Lett. 1993, 34, 805

A NEW SYNTHETIC APPROACH TO ENEDIYNES
RELATED TO THE NEOCARZINOSTATIN CHROMOPHORE
FROM α-TRIMETHYLSILYL α-ALLENYL CARBONYL COMPOUNDS
Nícos A. Petasis" and Kurt A. Teets

Department of Chemistry, University of Southern California, Los Angeles, California 90089-0744

Conversion of Benzal Chloride to Stilbene by Electrogenerated Cobalt(I)(Salen). A Doubly Electrocatalytic Process

Albert J. Fry, \* U. Nilantha Sirisoma, and Agnes S. Lee, Hall-Atwater Laboratory of Chemistry, Wesleyan University, Middletown, CT 06459

Benzal chloride is converted to a mixture consisting primarily of cis- and trans-stilbene by the action of electrochemically generated cobalt(I)(salen). The process involves a sequence involving electrocatalytic conversion of benzal chloride to a mixture of meso- and dl-1,2-dichloro-1,2-diphenylethane, followed by electrocatalyzed conversion of the latter to the stilbenes. Each of the diastereomeric dichlorides affords the same mixture of stilbenes under the reaction conditions.

#### [4+1]-ANIONIC ANNULATION APPROACH TO PHENYL-SULFONYL SUBSTITUTED CYCLOPENTENES

Tetrahedron Lett. 1993, 34, 813

Albert Padwa\* and Michelle A. Filipkowski

Department of Chemistry, Bmory University Atlanta, Georgia 30322 USA

2,3-Bis(phenylsulfonyl)-1,3-butadiene undergoes a [4+1]-annulation reaction with a variety of soft carbanions to give phenylsulfonyl substituted cyclopentenes in good yield.

### PREPARATION OF 3α- AND 3β-(ω-AMINOALKOXY)-7α,12α-DIHYDROXY-CHOLANOIC ACID ESTERS: VERSATILE SHUTTLES FOR DRUG TARGETING

Tetrahedron Lett. 1993, 34, 817

COOR

G. Wess\*, W. Kramer, A. Enhsen, H. Glombik, K.-H. Baringhaus K. Bock, H. Kleine, W. Schmitt Hoechst AG, Pharma Forschung, Postfach 800320, D-6230 Frankfurt-80

H<sub>2</sub>N -(CH<sub>2</sub>) O H H H OH

 $3\alpha$ - and  $3\beta$ -( $\omega$ -aminoalkoxy)-cholanoic acid esters 2 are prepared from cholic acid

## SYNTHESIS OF BILE ACID-DRUG CONJUGATES: POTENTIAL DRUG-SHUTTLES FOR LIVER SPECIFIC TARGETING

Tetrahedron Lett. 1993, 34, 819

G. Wess\*, W. Kramer, G. Schubert, A. Enhsen, K.-H. Baringhaus, H. Glombik S. Müllner, K. Bock, H. Kleine, M. John, G. Neckermann, A.Hoffmann Hoechst AG, Pharma Forschung, Postfach 800320, D-6230 Frankfurt-80

Bile acid-drug conjugates 2 are prepared with chlorambucil, HR 780 and an oxaproline peptide

Tetrahedron Lett. 1993, 34, 827

#### REACTION OF DIAZOMETHANE WITH CLEROCIDIN. Preparation of New Clerodane Diterpenoid Derivatives

J.P. Jacquet,\* D. Bouzard, and P. Remuzon. Bristol-Myers Squibb Pharmaceutical Research Institute. BP 62. 77422 Marne-la-Vallée Cedex 2, FRANCE

Reaction of diazomethane with 1 gave

an open form 2 and the diepoxide 4.

#### Dialkylation of β and γ-Diketone Monoethyleneketals by 1,8-Bis(trimethylsilyl)-2,6-octadiene.

Hélène Pellissier, Arlette Tubul and Maurice Santelli\*

Laboratoire de Synthèse Organique associé au CNRS, Centre de Si-Jérôme, Av. Esc. Normandie-Niemen, 13397, Marseille Cedex 13-France.

Dialkylation of \( \beta \)-diketone monoethyleneketals by 1.8-bis(trimethylsilyl)-2.6-octadiene leads to bi- or tricyclic alcohols (one stereoisomer) resulting from a participation reaction.

## SYNTHETIC STUDIES OF NOVEL 6-AZA- AND 6,7-DIAZA-AZULENO[1,2-a]AZULENES

Makoto Nitta, "Yukio Iino, Tomoki Sugiyama, and Akira Akaogi

Department of Chemistry, School of Science and Engineering, Waseda University, Shinjuku-ku, Tokyo 169, Japan

The title compounds were synthesized by thermal reaction of [(azulen-2-yl)imino]phosphorane and [(1-azaazulen-2-yl)imino]phosphorane with 2-bromotropone or alternative methods.

## Tetrahedron Lett. 1993, 34, 831

X=CH: X=N

## Tetrahedron Lett. 1993, 34, 835

#### STRUCTURAL AND CHEMICALPROPERTIES OF 6-AZA- AND 6.7-DIAZA-AZULENO[1,2-a]AZULENES

Makoto Nitta, \* Yukio Iino, Tomoki Sugiyama, and Azumao Toyota \* Department of Chemistry, School of Science and Engineering, Waseda University, Shinjuku-ku, Tokyo 169, Japan; \*Department of Chemistry, Faculty of General Education, Yamagata University, Kojirakawa, Yamagata 990, Japan

The NMR and electronic spectral studies including PPP-type MO calculation of the title compounds revealed that there are little contribution of peripheral  $18-\pi$  electron conjugation but those are rather composed of two azulene-type \u03c4-systems. The both compounds existing as cationic and dicationic species in acidic media are also studied.

Preparation of Linear Phosphorus Compound Containing Two Hypervalent Centers. Acetylene-bisitriphenyl(phenylethynyl)phosphoranel

Satoshi Ogawa, Yoko Tajiri, and Naomichi Furukawa\* Department of Chemistry, University of Tsukuba, Tsukuba, Ibaraki 305, Japan

A new linear acetylide-connected bis-phosphorane, acetylenebis[triphenyl(phenylethynyl)phosphorane] was synthesized.

Tetrahedron Lett. 1993, 34, 841

Tetrahedron Lett. 1993, 34, 845

## Preparation of Macrodiolides via a Common Chiral Building Block. Total Synthesis of (-)-Pyrenophorin and (-)-Pyrenophorol

### Nobuo Machinaga and Chihiro Kibayashi\*

Tokyo College of Pharmacy, Horinouchi, Hachioii. Tokyo 192-03, Japan

Macrodiolides (-)-pyrenophorin (1) and (-)pyrenophorol (2) have been synthesized utilizing the  $C_2$  symmetric (R,R)-diepoxide 3 as a common enantiopure chiral building block.

## A STEREOSELECTIVE ROUTE TO E-3-HYDROXYALKENYL PHENYL SULFIDES

Seiichi Takano,\* Yoshiaki Sugihara, and Kunio Ogasawara

Pharmaceutical Institute, Tohoku University, Aobayama, Sendai 980, Japan

$$R_{1}$$
  $R_{2}$   $O$   $R_{3}$   $R_{2}$   $O$   $R_{3}$   $R_{2}$   $O$   $R_{3}$   $R_{2}$   $O$   $R_{3}$   $R_{4}$   $R_{2}$   $O$   $R_{4}$   $R_{5}$   $R_{5}$ 

R<sub>1</sub>; R<sub>3</sub>=H, alkyl, aryl, R<sub>2</sub>+H

#### The First Diazatetrathiafulvalenes

Suk-Ling Chu, Kwok-Fai Wai, Ting-Fong Lai and Michael P. Sammes Department of Chemistry, the University of Hong Kong, Pokfulam Road, Hong Kong

Tetrahedron Lett. 1993, 34, 847

Preparation of the title compounds is described. X-Ray analysis of the trans isomer (R = H) reveals stacked, planar molecules with intra-stack distances of 3.56Å.

Catalytic Asymmetric Nitroaldol Reactions.

A New Practical Method for the Preparation of the Optically Active Lanthanum Complex

Hiroaki Sasai, Takeyuki Suzuki, Noriie Itoh, and Masakatsu Shibasaki\* Faculty of Pharmaceutical Sciences, University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113, Japan

The optically active La complex, which catalyzes asymmetric nitroaldol reactions, was found to be readily prepared from LaCl3. 7H2O. The presence of water, LiCl or LiBr and an alkali metal hydroxide was essential for the formation of the effective lanthanum binaphthoxide complex.

Tetrahedron Lett. 1993, 34, 855

Catalytic Asymmetric Nitroaldel Reactic

An Efficient Synthesis of (S) Proprancial Using Lanthanum Binaphthol Complex

Hiroaki Sassi, Noriie Itoh, Takeyuki Suzuki and Masakatsu Shibasaki\* Pacuky of Pharmaceutical Sciences, University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113, Japan

(5) Propranolol was conveniently synthesized in a highly enantioselective manner by the lanthanum-(R)-(+)-binaphthol complex catalyzed asymmetric nitroaldol reaction.

Unexpected Product from the Dakin-West Reaction of N-Acylprolines using Trifluoroacetic Anhydride: A Novel Access to 5-Trifluoromethyloxazoles

Tetrahedron Lett. 1993, 34, 859

Masami Kawase,\* A Hiroshi Miyamae, b Mariko Narita, b and Teruo Kuriharab

Faculty of Pharmaceutical Sciences and Faculty of Science, Josai University, 1-1 Keyakidai, Sakado-shi, Saitama 350-02, Japan

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\end{array}$$

AGGREGATION BEHAVIOR AND REACTIVITY OF HYDROPHOBIC VITAMIN B<sub>12</sub> COVALENTLY BOUND TO LIPID IN AQUEOUS MEDIA

Yukito Murakami,\* Yoshio Hisaeda, Akihiro Ogawa, Tetsuya Miyajima,

Osamu Hayashida, and Teruhisa Ohno Department of Chemical Science and Technology, Faculty of Engineering, Kyushu University, Fukuoka 812, Japan

Co-vesicles of B<sub>12</sub>-lipid and N<sup>+</sup>C<sub>5</sub>Ala2C<sub>16</sub> at a 1:50 molar ratio provided

(CH<sub>2</sub>)<sub>15</sub>CH<sub>3</sub> H<sub>2</sub>CO<sub>2</sub>C CO<sub>2</sub>CH<sub>2</sub>

B<sub>12</sub>-lipid : H<sub>3</sub>CO<sub>2</sub>C CO<sub>2</sub>CH<sub>3</sub> H<sub>3</sub>CO<sub>2</sub>C CO2CH2

Tetrahedron Lett. 1993, 34, 863

(CH<sub>3</sub>)<sub>3</sub>N<sup>+</sup>(CH<sub>2</sub>)<sub>5</sub> CNHCHCN

#### RADICAL-INDUCED CLEAVAGES IN RING-FUSED EPOXIDES.

John A. Murphy\* and Christopher W. Patterson, Department of Chemistry, University of Nottingham, Nottingham NG7 2RD

Carbon-carbon bond cleavage has been observed in ring-fused epoxides where stereoelctronic effects do not interfere.

# TIN(II) CHLORIDE DIHYDRATE REDUCTION OF $\beta$ , $\frac{1}{2}$ -UNSATURATED NITROALKENES

Tetrahedron Lett. 1993, 34, 869

Nalin B Das, Jadab C Sarma, Ram P Sharma\* and Manobiyoti Bordoloi

Natural Products Chemistry Division Regional Research Laboratory (CSIR) Jorhat 785 006, Assam INDIA

## BIS(TRIMETHYLSILYL)SULFIDE BASED THIONATION OF CARBONYL COMPOUNDS: SYNTHESIS OF THIOKETONES

Tetrahedron Lett. 1993, 34, 873

A. Dedi'innocenti<sup>1,a</sup>, A. Capperucci<sup>4,b</sup>, A. Mordini<sup>c</sup>, G. Reginato<sup>c</sup>, A. Ricci<sup>c,d</sup>, F. Cerreta<sup>b</sup>,

<sup>a</sup>Dipartimento di Chimica, Via N. Sauro 85, 85100 Potenza (Italy). <sup>b</sup>Dipartimento di Chimica Organica and <sup>c</sup>Centro CNR sulla Chimica e la Struttura dei Composti Eterociclici, Via G. Capponi 9, 50121 Firenze (Italy). <sup>d</sup>Dipartimento di Chimica Organica, Viale Risorgimento 4, 40136 Bologna (Italy).

Thioketones may be conveniently obtained by silyl triflate promoted bis(trimethylsilyl)sulfide thionation of ketones.

R. R'= alkvl. cvclic. α.β-unsaturated

Tetrahedron Lett. 1993, 34, 877

A PRACTICAL SYNTHESIS OF SUBSTITUTED BENZO[ $\circ$ ]CINNOLINEN,N'-DIOXIDES AND N-OXIDES.

Cristins Paradisi\*, Guadalupe Gonzalez-Trueba, Glanfranco Scorrano
Centro Meccanismi di Reazioni Organiche del CNR, Dipartimento di Chimica Organica dell'Universita' di Padova, v. Marzolo 1, 35131 Padova, Italy
Depending on the equivalents of KOH used and on the reaction time N.N\*-dioxides (2) or N-oxides (3) are formed in satisfatory violds.

a: X = H; b: X = 5.5'-Cl<sub>2</sub>; a: X = 4.4'-(CH<sub>2</sub>)<sub>2</sub>; d: X = 4.4'-(CF<sub>2</sub>)<sub>2</sub>; e: X = 5.5'-(2-PrO)<sub>2</sub>

## THE STEREOCHEMISTRY OF ENAMIDE INTERMEDIATES IN

DUPHOS-RH(I) CATALYSED ASYMMETRIC HYDROGENATION
Susan K. Armstrong and John M. Brown, Dyson Perriss Laboratory, South Perks Road, Oxford OX1 3QY
Mark J. Burk, Research and Development Laboratories, E. I. du Pont de Nemours, PO Box 80328, Wilmington, DE, 19880-0328

Stereochemical aspects of the title reaction (below) were investigated for the (S,S)-Me-DuPHOS ligand 5a, largely by NMR examination of Ir complexes. They were found to behave in an analogous fashion to that established for P-aryl diphosphine rhodium(I) complexes

## KINETIC RESOLUTION OF LARYL AND L-HETEROARYL ETHANOLS BY OXIDATION WITH BAKER'S YEAST.

Tetrahedron Lett. 1993, 34, 883

G. Pantin, M. Fogagnolo, A. Medici. P. Pedrini, S. Poli, M. Sinigaglia, Dipartimento di Chimica, Università di Ferrara, Italy;

<sup>a</sup>Dipartimento di Protezione e Valorizzazione Agroalimentare, Università di Bologna, Italy

The kinetic resolution of racemic 1-aryl and 1-heteroaryl ethanols via oxidation by Baker's yeast to give the R-enantiomer is reported.

COPOLYMERISATION OF 5,6-DIHYDROXYINDOLE AND 5,6-DIHYDROXY-INDOLE-2-CARBOXYLIC ACID IN MELANOGENESIS: ISOLATION OF A CROSS-COUPLING PRODUCT

Tetrahedron Lett. 1993, 34, 885

A. Napolitano, O.Crescenzi and G. Prota\* Department of Organic and Biological Chemistry University of Naples, Via Mezzocannone 16, I-80134 Naples, Italy

Co-oxidation of 5,6-dihydroxyindole and its 2-carboxy derivative under biomimetic conditions afforded, besides homopolymers of the two indoles, a mixed-type dimer which was isolated in the O-acetyl form and formulated as 3.