Zero-field splitting of quintet conjugated dinitrenes: 2,6-biphenylenedinitrene

Tetrahedron Letters 44 (2003) 2625

Rajdeep S. Kalgutkar and Paul M. Lahti*

Department of Chemistry, University of Massachusetts, Amherst, MA 01003, USA

Photolysis of 4 yields high-spin dinitrene 5 with an unusual *anti* (180°) geometry of nitrene C-N bonds, thanks to the incorporation of a cyclobutadiene ring. The electron spin resonance quintet state zero field splitting of 5 fits a standard dipolar model for nitrene-nitrene interaction.

Addition of secondary phosphines to N-vinylpyrroles

Tetrahedron Letters 44 (2003) 2629

Boris A. Trofimov, Svetlana F. Malysheva,* Boris G. Sukhov,

Natal'ya A. Belogorlova, Elena Yu. Schmidt, Lyubov N. Sobenina, Vladimir A. Kuimov and Nina K. Gusarova

A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Branch of the Russian Academy of Sciences, 1, Favorsky Str., RUS-664033 Irkutsk, Russia

$$R^{1}_{2}PH$$
 + R^{2} radical initiation R^{2} R^{3} R^{2} R^{2} R^{3} R^{2} R^{2} R^{3} R^{2} R^{3} R^{2} R^{2} R^{3} R^{2} R^{3} R^{2} R^{3} R^{2} R^{3} R^{2} R^{3} R^{3} R^{3} R^{3} R^{3} R^{2} R^{3} R

Synthesis of a model bicyclic core related to *Microscleroderma spirophora* steroids

Anand Mayasundari, Ulf Peters and David G. J. Young*

Department of Chemistry, University of Tennessee, Knoxville, TN 37996, USA

Tetrahedron Letters 44 (2003) 2633

Aldingenin A, new brominated sesquiterpene from red algae Laurencia aldingensis

Tetrahedron Letters 44 (2003) 2637

Luciana R. de Carvalho,^a Mutue T. Fujii,^a Nídia F. Roque,^b Massuo J. Kato^c and João Henrique G. Lago^{c,*}

a Seção de Ficologia, Instituto de Botânica, CP 4005, CEP 10061-970, São Paulo-SP, Brazil

bInstituto de Química, Universidade Federal da Bahia, CEP 40170-290, Salvador-BA, Brazil

^eInstituto de Química, Universidade de São Paulo, CP 26077, CEP 05599-970, São Paulo-SP, Brazil

A new brominated bisabolene derivative, aldingenin A, was isolated from red alga *Laurencia* aldingensis Saito et Womersley (Ceramiales, Rodophyta). Its structure was determined by analysis of spectroscopic data and biogenetic considerations.

A synthetic approach to bicyclo[6.2.1]undecane ring systems

Mauricio Gomes Constantino,* Kleber Thiago de Oliveira,

Adilson Beatriz and Gil Valdo José da Silva

Departamento de Química da Faculdade de Filosofia Ciências e Letras de Ribeirão Preto, Universidade de São Paulo, Avenida dos Bandeirantes, 3900, 14040-901, Ribeirão Preto, SP, Brazil

A short and efficient synthesis of 10 is described.

Stereoselective ring contraction diverts the Mitsunobu reaction of a 6-hydroxy-1,4-diazepan-2-one

Tetrahedron Letters 44 (2003) 2645

Spencer Knapp,^{a,*} Gregori J. Morriello^a and George A. Doss^b

^aDepartment of Chemistry & Chemical Biology, Rutgers The State University of New Jersey, 610 Taylor Road, Piscataway, NJ 08854-8087, USA

^bMerck & Co., PO Box 2000, Rahway, NJ 07065-0900, USA

$$t\text{-BuPh}_2 \text{SiO} \xrightarrow{\text{CH}_3} \text{O} \text{HO}^{\text{CH}_3} \text{O} \text{PhCOO} \xrightarrow{\text{CH}_3} \text{PhCOO} \text{CH}_3$$

Unexpected reactivity between aromatic nitro compounds and PCl₃/AlCl₃. A new one-pot synthesis of phenazines

Tetrahedron Letters 44 (2003) 2649

Rodolphe Abdayem, a Graziano Baccolini, a.* Carla Boga, Magda Monari and Simona Selva

^aDipartimento di Chimica Organica 'A. Mangini', Viale Risorgimento, 4-40136 Bologna, Italy

^bDipartimento Di Chimica 'G. Ciamician', Via Selmi 2-40126 Bologna, Italy

R 1)3 PCl₃, 1 AlCl₃

$$\begin{array}{c}
 & \begin{array}{c}
 & 1)3 \text{ PCl}_3, 1 \text{ AlCl}_3 \\
 & \begin{array}{c}
 & 50-55^{\circ}\text{C} \\
 & 2) \text{ H}_2\text{O}
\end{array}
\end{array}$$
R
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}
\end{array}$$
R
$$\begin{array}{c}
 & \text{NN} \\
 & \text{R}
\end{array}$$
R
$$\begin{array}{c}
 & \text{NN} \\
 & \text{R}
\end{array}$$
A
$$\begin{array}{c}
 & \text{R} \\
 & \text{R}
\end{array}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}
\end{array}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}
\end{array}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}
\end{array}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}
\end{array}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN}
\end{array}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN} \\
 & \text{NN}
\end{array}$$
A
$$\begin{array}{c}
 & \text{NN} \\
 & \text{NN} \\
 & \text{NN}$$
A
$$\begin{array}$$

Methyltrioxorhenium-catalyzed aerobic oxidative coupling of 2-naphthols to binaphthols

Tetrahedron Letters 44 (2003) 2655

Vishal B. Sharma, Suman L. Jain and Bir Sain*

Chemical and Biosciences Division, Indian Institute of Petroleum, Dehradun 248005, India

$$R^{1}$$
 R^{2}
 R^{3}
 R^{3}
 R^{3}
 R^{3}
 R^{3}
 R^{3}
 R^{3}
 R^{3}
 R^{4}
 R^{2}
 R^{2}
 R^{3}
 R^{3}
 R^{3}
 R^{3}
 R^{3}
 R^{3}

Mono(6^A -N-allylamino- 6^A -deoxy)perphenylcarbamoylated β -cyclodextrin: synthesis and application as a chiral stationary phase for HPLC

Xianghua Lai and Siu-Choon Ng*

Department of Chemistry, National University of Singapore, Singapore 119260

A novel cyclodextrin derivative: $mono(6^A-N-allylamino-6^A-deoxy)$ perphenylcarbamoylated β -cyclodextrin was synthesized. Hydrosilylation with (EtO)₃SiH and then reaction of the reactive siloxane with pristine silica gel afforded a facile entry into a durable, structurally well-defined chiral stationary phase capable of enantioseparation of a variety of racemic drugs.

$$(OR)_{14} \qquad (EtO)_3Si-(CH_2)_3 \qquad (OR)_{14} \qquad (OR)_{14} \qquad (OR)_{14} \qquad (OR)_{14}$$

Selective reduction of carbon-carbon double and triple bonds in conjugated olefins mediated by SmI₂/H₂O/amine in THF

Tetrahedron Letters 44 (2003) 2661

Anders Dahlén and Göran Hilmersson*

Organic Chemistry, Department of Chemistry, Göteborg University, SE-412 96 Göteborg, Sweden

Conjugated double and triple bonds are reduced into alkenes using non-hazardous SmI₂/H₂O/amine mixtures as reducing agents in THF. Isolated alkenes are not reduced during these reductions. All the reactions studied are quantitative and are completed in less than five minutes.

SmI₂/H₂O/R₃N
5 min

> 99%

Synthesis of a biphenyl-based cyclophane via benzidine rearrangement of a constrained *m*-nitrophenol derivative

Tetrahedron Letters 44 (2003) 2665

Andrew C. Benniston,^{a,*} William Clegg,^b Anthony Harriman,^a Ross W. Harrington,^b Peiyi Li^a and Craig Sams^a

^aMolecular Photonics Laboratory, School of Natural Sciences (Chemistry), University of Newcastle upon Tyne, Newcastle upon Tyne NE1 7RU, UK

^bCrystallography Laboratory, School of Natural Sciences (Chemistry), University of Newcastle upon Tyne, Newcastle upon Tyne

Activity-based fluorescent probes that target phosphatases

Tetrahedron Letters 44 (2003) 2669

Qing Zhu, a Xuan Huang, Grace Y. J. Chena, and Shao Q. Yaoa, **

^aDepartment of Chemistry, National University of Singapore, 3 Science Drive 3, Singapore 117543, Singapore

^bDepartment of Biological Sciences, National University of Singapore, 3 Science Drive 3, Singapore 117543, Singapore

Two fluorescently-labeled, activity-based probes, **Probe 1** and **Probe 2** have been successfully designed and synthesized. They were shown to label selectively different types of phosphatases over other enzymes. These probes may find potential applications in large-scale proteomic experiments.

 $O_{0} \stackrel{O}{\underset{P}{\longrightarrow}} O \stackrel{F}{\longleftrightarrow} \stackrel{H}{\longleftrightarrow} O \stackrel{O}{\longleftrightarrow} O \stackrel{O}$

Probe 1

$$(HO)_2P-O \xrightarrow{CHF_2} \overset{H}{\underset{N}{\longrightarrow}} \overset{O}{\underset{N}{\longrightarrow}} Cy3$$

Probe 2

Synthesis of a new C_{60} -substituted tris(2,2'-bipyridine)ruthenium(II) complex

Tetrahedron Letters 44 (2003) 2673

François Cardinali and Jean-François Nierengarten*

Groupe des Matériaux Organiques, Institut de Physique et Chimie des Matériaux de Strasbourg, Université Louis Pasteur et CNRS, 23 rue du Loess, BP 43, 67034 Strasbourg Cedex, France

On the lithiation of oxazolinylaziridines

Tetrahedron Letters 44 (2003) 2677

Renzo Luisi, Vito Capriati, Saverio Florio* and Rosa Ranaldo

Dipartimento Farmaco-Chimico, Università di Bari, Via E. Orabona 4, I-70126, Bari C.N.R., Istituto di Chimica dei Composti Organo Metallici 'ICCOM', Sezione di Bari, Italy

An efficient asymmetric synthesis of Fmoc-L-cyclopentylglycine via diastereoselective alkylation of glycine enolate equivalent

Tetrahedron Letters 44 (2003) 2683

Satendra Singh* and Michael W. Pennington

BACHEM Bioscience Inc., 3700 Horizon Drive, King of Prussia, PA 19406, USA

A short and efficient synthesis of Fmoc-L-cyclopentylglycine is reported.

N-Alkylation of amines with alcohols catalyzed by a Cp*Ir complex

Tetrahedron Letters 44 (2003) 2687

Ken-ichi Fujita, a,* Zhenzi Li, Naohiro Ozeki and Ryohei Yamaguchi ,*

^aDepartment of Natural Resources, Graduate School of Global Environmental Studies, Kyoto University, Kyoto 606-8501, Japan

^bGraduate School of Human and Environmental Studies, Kyoto University, Kyoto 606-8501, Japan

A new effective catalytic system consisting of $[Cp*IrCl_2]_2/K_2CO_3$ (Cp*= pentamethylcyclopentadienyl) for the N-alkylation of primary amines with alcohols has been developed.

$$R^{1}NH_{2} + R^{2} R^{3} \xrightarrow{\text{Cat. } [Cp^{*}IrCl_{2}]_{2} (5.0 \text{ mol}\% Ir)} R^{2}NHR^{2} R^{3} R^{3}$$

Synthesis of 1,5-naphthylethynyl nanostructure networks with extended π -conjugation. Effective heterocoupling catalyzed by palladium under a compatible CO_2 atmosphere

J. Gonzalo Rodríguez* and J. Luis Tejedor

Departamento de Química Orgánica, Facultad de Ciencias, Universidad Autónoma, Cantoblanco, 28049 Madrid, Spain

A general protocol for the regio high yielding opening of different glycidol derivatives

Tetrahedron Letters 44 (2003) 2695

Carlo Bonini,^{a,*} Lucia Chiummiento,^a Maria Teresa Lopardo,^a Maddalena Pullez,^a Françoise Colobert^{b,*} and Guy Solladié^{b,*}

^aDipartimento di Chimica, Università della Basilicata, Via N. Sauro, 85, 85100 Potenza, Italy

^bLaboratoire de stéréochimie associé au CNRS, Université Louis Pasteur, ECPM, 25 rue Becquerel, 67087 Strasbourg cedex 2, France

Protected glycidol derivatives 1 have been tested for the regioselective ring opening with vinylmagnesium bromide in order to obtain useful five-carbon functionalised homoallylic alcohols 2.

Site-site interactions within high-loading PAMAM dendrimer resin beads

Tetrahedron Letters 44 (2003) 2699

Andrea Basso^{a,*} and Mark Bradley^b

^aUniversità degli Studi di Genova, Dipartimento di Chimica e Chimica Industriale, Via Dodecaneso 31, Genova I-16146, Italy

^bUniversity of Southampton, Department of Chemistry, Highfield, Southampton SO17 1BJ, UK

Site-site interactions on high-loading PAMAM dendrimer beads were no greater than on TentaGel (PS-PEG) resin.

ROMPgel-supported tris(triphenylphosphine)rhodium(I) chloride: a selective hydrogenation catalyst for parallel synthesis

Tetrahedron Letters 44 (2003) 2703

Erik Årstad, Anthony G. M. Barrett* and Livio Tedeschi

Department of Chemistry, Imperial College of Science, Technology and Medicine, Exhibition Road, London SW7 2AY, UK

$$R^{1} \xrightarrow[Ph PPh_{3}]{Ph PPh_{3}} \\ H_{2}, THF:EtOH_{1:1}$$

 R^1 = alkyl, aryl; R^2 = H, alkyl, NO_2

Stereospecific synthesis of eight- and nine-membered cyclic ethers by Eu(fod)₃-mediated cyclization of hydroxy epoxides

Toshikazu Saitoh,^a Toshio Suzuki,^{b,*} Norihiro Onodera,^a Hideya Sekiguchi,^a Hisahiro Hagiwara^a and Takashi Hoshi^b

^aGraduate School of Science and Technology, Niigata University, 2-nocho, Ikarashi, Niigata 950-2181, Japan ^bFaculty of Engineering, Niigata University, 2-nocho, Ikarashi, Niigata 950-2181, Japan

$$\begin{array}{c|c} \text{DH} & \text{Eu(fod)}_3 \\ \text{Next of toluene} & \text{Eu(fod)}_3 \\ \text{Next of toluene} & \text{Eu(fod)}_3 \\ \text{exo-mode} & \text{Eu(fod)}_3 \\$$

Approaches to the total synthesis of phomactins

Tetrahedron Letters 44 (2003) 2713

Andrew S. Balnaves, Graham McGowan, Peter D. P. Shapland and Eric J. Thomas*

The Department of Chemistry, The University of Manchester, Manchester M13 9PL, UK

A synthesis of the bicyclic trienyl alcohol **34** is reported which features a regio- and stereoselective 2,3-Wittig rearrangement of the propargylic ether **21**. The alcohol **34** is an advanced intermediate for a projected synthesis of phomactins.

Pyrimidine to guanine PDE inhibitors: determination of chemical course via structure elucidation

Tetrahedron Letters 44 (2003) 2717

Dinesh Gala,* Donald J. DiBenedetto, Max Kugelman and Mohindar S. Puar

Schering-Plough Research Institute, 1011 Morris Avenue, Union, NJ 07083, USA

NMR analysis of advanced intermediates in a chemical synthesis unraveled that the chlorination of pyrimidine 3 leads to the formation of 11, and not 4. Some uncommon chemical observations are also described.

A novel synthesis of guanine PDE inhibitors via tricyclic imidazopyrimidines

Tetrahedron Letters 44 (2003) 2721

Dinesh Gala,* Donald J. DiBenedetto, Max Kugelman and Michael B. Mitchell

Schering-Plough Research Institute, 1011 Morris Avenue, Union, NJ 07083, USA

The synthesis and use of tricyclic imidazopyrimidine 9 as an advanced intermediate for the preparation of multiple PDE inhibitors 1 and 2 are described.

Formanilide and carbanilide from aniline and carbon dioxide

Tetrahedron Letters 44 (2003) 2725

Pradip Munshi, David J. Heldebrant, Erin P. McKoon, Patrick A. Kelly, Chih-Cheng Tai and Philip G. Jessop*

Department of Chemistry, University of California, Davis, CA 95616-5295, USA

Earlier methods for the reduction of ${\rm CO}_2$ and amines to formamides have only been successful for dialkylamines.

Now, a synthesis of formanilide from CO₂, H₂ and aniline has been designed. In the absence of H₂, carbanilide is formed.

$$\begin{array}{c} \text{trans-RuCl}_2(\text{PMe}_3)_4 & \overset{\text{O}}{\text{II}} \\ \text{H}_2, \text{DBU} & \text{scCO}_2 \\ \\ \text{PhNH}_2 + \text{CO}_2 & \overset{\text{DBU}}{\text{scCO}_2} & \overset{\text{O}}{\text{O}} \\ \\ \text{PhHN}-\overset{\text{C}}{\text{C}}-\text{NHPh} \end{array}$$

Convenient synthesis of functionalized terphenyls

Tetrahedron Letters 44 (2003) 2729

A. A. Greenfield,* J. A. Butera and C. E. Caufield

Chemical Sciences, Wyeth Research, CN 8000, Princeton, NJ 08543-8000, USA

A highly efficient olefin metathesis initiator: improved synthesis and reactivity studies

Tetrahedron Letters 44 (2003) 2733

Aideen M. Dunne, Stefan Mix and Siegfried Blechert*

Institut für Chemie, Technische Universität Berlin, Strasse des 17. Juni 135, 10623 Berlin, Germany

A practical synthesis and reactivity studies of the catalyst shown are reported.

Isolation and structural determination of spilacleosides A and B having a novel 1,3-dioxolan-4-one ring

Tetrahedron Letters 44 (2003) 2737

1: Spilacleoside A

Akiyo Kameyama,^a Yusuke Shibuya,^a Hiroshi Kusuoku,^a Yoshinori Nishizawa,^{a,*}

Satoshi Nakano^b and Kuniaki Tatsuta^{b,*}

^aBiological Science Laboratories, Kao Corporation, 2606 Akabane, Ichikai-machi, Haga, Tochigi 321-3497, Japan ^bDepartment of Applied Chemistry, School of Science and Engineering, Waseda University, 3-4-1 Ohkubo, Shinjuku-ku, Tokyo 169-8555, Japan

Preparation of a novel 16-DPA- P_2S_5 adduct and its application as a masked α,β -unsaturated ketone in [4+2]cycloaddition reactions

Apurba Chetia, Anil Saikia, Chandan J. Saikia and Romesh C. Boruah*

Medicinal Chemistry Division, Regional Research Laboratory, Jorhat 785006, India

$$\begin{array}{c|c}
Me & Me & Me \\
\hline
O & P-S-P & Me
\end{array}$$

$$\begin{array}{c|c}
Me & Me \\
\hline
O & COOMe
\end{array}$$

$$\begin{array}{c|c}
DMAD & Me
\end{array}$$

$$\begin{array}{c|c}
COOMe \\
H & COOMe
\end{array}$$

Efficient synthesis of novel pyrido[3,2-d]pyrimidine-2,4-diones

Tetrahedron Letters 44 (2003) 2745

R. Mamouni, M. Aadil, M. Akssira, A. J. Lasri and

J. Sepulveda-Arques^b

^aLaboratoire de Chimie Bioorganique et Analytique, Université Hassan II-Mohammedia, BP 146, 20650 Mohammedia, Morocco

^bDepartamento de Química Orgánica, Facultad de Farmacia, Universidad de Valencia, Avda. Vicent Andres Estelles s/n, 46180 Burjassot, Valencia, Spain

Modification of the Cp' ring in the ferrocifen precursor and its influence on the recognition by the estrogen receptor

Konrad Kowalski,^{a,b} Anne Vessières,^a Siden Top,^a Gérard Jaouen^{a,*} and Janusz Zakrzewski^b

^aEcole Nationale Supérieure de Chimie de Paris, 11 rue Pierre et Marie Curie, 75231 Paris Cedex 05, France

^bDepartment of Organic Chemistry, University of Łódź, 90-136 Łódź, Narutowicza 68, Poland Tetrahedron Letters 44 (2003) 2749

A remarkably simple α -oximation of ketones to 1,2-dione monooximes using the chlorotrimethylsilane—isoamyl nitrite combination

Tetrahedron Letters 44 (2003) 2753

Abdulkarim H. A. Mohammed and Gopalpur Nagendrappa*

Department of Chemistry, Bangalore University (Central College Campus), Bangalore 560001, India

$$\begin{array}{c} \begin{array}{c} O \\ II \\ R-C-CH_2-R' \end{array} \xrightarrow{\begin{array}{c} Me_3SiCI+C_5H_{11}ONO \end{array}} \begin{array}{c} O \\ II \\ R-C-C-R \end{array}$$

One-pot synthesis of dihydropyrimidinones catalysed by lithium bromide: an improved procedure for the Biginelli reaction

Gourhari Maiti, a,* Pradip Kundua and Chandrani Guinb

RCHO + Me OEt +
$$H_2N$$
 NH_2 $LiBr$ (cat) CH_3CN $reflux$ Me NH_2

^aDepartment of Chemistry, Jadavpur University, Jadavpur, Calcutta 700 032, India

^bDepartment of Organic Chemistry, Indian Association for the Cultivation of Science, Jadavpur, Calcutta 700 032, India