

Tetrahedron Letters Vol. 46, No. 50, 2005

Contents

COMMUNICATIONS

Total synthesis of (±)-cyclooroidin

pp 8635-8638

Gianluca Papeo,* María Antonia Gómez-Zurita Frau, Daniela Borghi and Mario Varasi

(±)-cyclooroidin

The first total synthesis of (\pm) -cyclooroidin, a member of the pyrrole-imidazole alkaloid family recently isolated from the sponge *Agelas oroides* in optically pure form, has been described.

Indium(III) triflate catalyzed tandem azidation/1,3-dipolar cycloaddition reaction of ω,ω -dialkoxyalkyne derivatives with trimethylsilyl azide

pp 8639-8643

Hikaru Yanai and Takeo Taguchi*

 $X = C(CO_2Et)_2$, O, NTs

A convenient procedure for palladium catalyzed cyanation using a unique bidentate phosphorus ligand pp 8645–8647 Rader S. Jensen, Anil S. Gajare, Kozo Toyota, Masaaki Yoshifuji* and Fumiyuki Ozawa

A low valent phosphorus ligand has been used to facilitate the palladium catalyzed cyanation of aryl bromides.

Asymmetric Michael addition using *N*-cinnamoyl- and *N*-crotonyl-*trans*-hexahydrobenzoxazolidin-2-ones pp 8649–8652 Lydia Pérez, Sylvain Bernès, Leticia Quintero* and Cecilia Anaya de Parrodi*

$$\begin{array}{c|c}
O & CuBr \cdot S(CH_3)_2, ZnX_2 \\
\hline
N & R'MgBr, THF \\
-40 °C to 25 °C
\end{array}$$

$$\begin{array}{c}
O \\
N \\
O \\
R'
\end{array}$$

2 and 3 or ent-2 and ent-3

4 - 11 or ent-4-ent-11

Stereoselective copper promoted conjugated additions of Grignard reagents to chiral *N*-enoyl amides 2 and 3 or *ent-*2 and *ent-*3 in the presence of Zn(II) salts afforded the 1,4-addition products 4–11 and the corresponding enantiomers.

The reaction of nitrones with pyrroles and furan: an easy access to heteroaromatic hydroxylamines and bis(heteroaryl)alkanes

pp 8653-8656

Christophe Berini, Frédéric Minassian,* Nadia Pelloux-Léon* and Yannick Vallée

New sesquiterpene lactones from water extract of the root of *Lindera strychnifolia* with cytotoxicity pp 8657–8660 against the human small cell lung cancer cell, SBC-3

Takamasa Ohno, Akito Nagatsu, Munehiro Nakagawa, Makoto Inoue, Yun-Mo Li, Shinya Minatoguchi, Hajime Mizukami and Hisayoshi Fujiwara*

New sesquiterpene lactones (1) and (2) were isolated from the EtOAc soluble fraction of the water extract of Linderae Radix through bioassay-guided fractionation and isolation methods. Two isolates showed significant cytotoxicity against the human small cell lung cancer cell SBC-3, and lesser cytotoxicity against mouse fibroblast cell 3T3-L1.

Synthesis of symmetrical 1,3-butadiynes by homocoupling reactions of alkynylboronates mediated by a copper salt pp 8661–8664

Yasushi Nishihara,* Masanori Okamoto, Yoshiaki Inoue, Mikihiro Miyazaki, Mitsuru Miyasaka and Kentaro Takagi

$$\begin{array}{c|c} R & \longrightarrow & O \\ \hline & & & Cu(OAc)_2 \\ \hline & DMI \\ & 60 \, ^{\circ}C \\ & \text{in air} \end{array} \qquad R & \longrightarrow & R$$

R = aromatic, heteroaromatic, aliphatic

84-99%

Various symmetrical 1,3-butadiyne derivatives are synthesized by a copper-mediated homocoupling reaction of alkynylboronates in good to excellent yields.

Synthesis of novel organoselenium as catalyst for Baeyer–Villiger oxidation with $30\%\ H_2O_2$

pp 8665-8668

Hayato Ichikawa,* Yoshihide Usami and Masao Arimoto

Studies on the formation of porphyrinogens from monopyrroles in presence of the enzymes PBG deaminase and/or Uro'gen III synthase

pp 8669-8672

Clotilde Pichon-Santander and A. Ian Scott*

 R_1 , $R_2 = CH_2CO_2H$, $CH_2CH_2CO_2H$, $CH_2CH_2CO_2H$ or CH_3

Preparation of porphyrins from pyrroles in buffer in presence of the enzymes PBG deaminase and/or Uro'gen III synthase is investigated.

A new route to 3,4-disubstituted piperidines: formal synthesis of (-)-paroxetine and (+)-femoxetine pp 8673–8676 Shinji Yamada* and Ishrat Jahan

Enantioselective synthesis of both enantiomers of *tert*-butylphenylphosphine oxide from (S)-prolinol

Adeline Leyris, Didier Nuel, Laurent Giordano, Mathieu Achard and Gérard Buono*

Both enantiomers of *tert*-butylphenylphosphine oxide can be easily synthesised starting from (S)-prolinol-based oxazaphospholidine.

Electrosynthesis of pyridines from 'only acetonitrile'

pp 8681-8683

M. Dolores Otero, Belen Batanero and Fructuoso Barba*

Direct aldol and tandem Mannich reactions in room temperature ammonia solutions

pp 8685-8689

Lichun Feng, Lijin Xu, Kimhung Lam, Zhongyuan Zhou, C. W. Yip* and Albert S. C. Chan*

RCHO +
$$\frac{2N \text{ NH}_3 \text{ in MeOH}}{\text{r.t.}} \xrightarrow{\text{OH}} \xrightarrow{\text$$

Sulfamic acid: a novel and efficient catalyst for the synthesis of aryl-14H-dibenzo[a.j]xanthenes under conventional heating and microwave irradiation

pp 8691-8693

B. Rajitha,* B. Sunil Kumar, Y. Thirupathi Reddy, P. Narsimha Reddy and N. Sreenivasulu

Palladium-catalyzed carbonylation of arylmethyl halides: efficient synthesis of arylacetic acid and esters

pp 8695-8697

Ray V. H. Jones, W. Edward Lindsell,* Daniel D. Palmer, Peter N. Preston* and Alan J. Whitton

ArCH₂X
$$\xrightarrow{CO}$$
 ArCH₂CO₂R
 $X = Cl, Br$ $\xrightarrow{[Pd cat]}$ $R = H \text{ (solvent is aq. biphasic)}$ $R = Me \text{ (solvent is MeOH)}$

High yielding catalytic syntheses of arylacetic acids and esters have been achieved under mild conditions through the use of new types of organo-palladium compounds.

A novel solid-phase synthesis of cyclic guanidines

pp 8699-8703

Vladimir Y. Vvedensky,* Boris V. Rogovoy, Alexander S. Kiselyov and Alexandre V. Ivachtchenko

Elongation of β-hydroxyenones by cross-metathesis

pp 8705-8709

Franck A. Silva and Véronique Gouverneur*



2-Nitrofurans as dienophiles in Diels-Alder reactions

pp 8711-8714

Claudia Della Rosa, María N. Kneeteman and Pedro M. E. Mancini*

Glycosyl azide—a novel substrate for enzymatic transglycosylations

pp 8715-8718

Pavla Fialová, Ana T. Carmona, Inmaculada Robina, Rüdiger Ettrich, Petr Sedmera, Věra Přikrylová, Lucie Petrásková-Hušáková and Vladimír Křen*



An improved synthesis of isonitrosoacetanilides

pp 8719-8721

Gordon W. Rewcastle,* Hamish S. Sutherland, Claudette A. Weir, Adrian G. Blackburn and William A. Denny

A novel two-step synthesis of isonitrosoacetanilides provides efficient access to compounds not readily available using traditional procedures.

Synthesis of 1-aryl-tetralins and 4-aryl-benzopyrans by sulfoxide-mediated benzylic carbocation cyclizations

pp 8723-8726

Alessandro Volonterio* and Matteo Zanda*

$$\begin{array}{c} O \\ S \\ CONHCH_3 \end{array}$$

$$\begin{array}{c} R^1 \\ Alkylation \\ NH \\ R^1 \end{array}$$

$$\begin{array}{c} C \\ Alkylation \\ R^2 \\ R^4 \end{array}$$

$$\begin{array}{c} C \\ Alkylation \\ R^2 \\ R^4 \end{array}$$

$$X = O, CH_2$$

Geranylgeraniol formation in *Croton stellatopilosus* proceeds via successive monodephosphorylations of geranylgeranyl diphosphate

pp 8727-8731

Natsajee Nualkaew, Wanchai De-Eknamkul,* Toni M. Kutchan and Meinhart H. Zenk

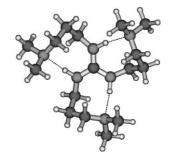
GGPP
$$CH_2O-P-P$$

GGMP CH_2O-P
 CH_2O-P
 CH_2O-P
 CH_2O-P

Synthesis and properties of novel guanidine bases. *N*,*N*′,*N*″-Tris(3-dimethylaminopropyl)-guanidine Zoran Glasovac, Borislav Kovačević, Ernest Meštrović and Mirjana Eckert-Maksić*

pp 8733–8736

The synthesis of novel N,N',N''-tris(3-dimethylaminopropyl)-guanidine 1 and X-ray structure of its hexafluorophosphate salt are described. The hydrogen bonding in protonated 1 is also discussed.





A general method for the deoxygenation of aromatic N-oxides using RuCl $_3$: xH_2O

pp 8737-8739

Sanjay Kumar, Anil Saini and Jagir S. Sandhu*

$$R^{1} \xrightarrow{\text{N}} R^{2} \xrightarrow{\text{RuCl}_{3}.\text{xH}_{2}\text{O}} R^{1} \xrightarrow{\text{R}^{2}} R^{2}$$

An efficient, simple and selective method for the deoxygenation of aromatic N-oxides, such as N-arylnitrones, azoxybenzenes, N-heteroarene N-oxides using ruthenium(III) chloride to afford deoxygenated products in excellent yields, is described.

Bargellini condensation of coumarins. Expeditious synthesis of *o***-carboxyvinylphenoxyisobutyric acids pp 8741–8743** Prabir K. Sen, Bidyut Biswas and Ramanathapuram V. Venkateswaran*

Chemoselective hydrolysis of terminal isopropylidene acetals in acetonitrile using molecular iodine as a mild and efficient catalyst

pp 8745-8748

J. S. Yadav,* M. Satyanarayana, S. Raghavendra and E. Balanarsaiah

Solid-phase synthesis of pyrido[2,3-d]pyrimidin-7-ones

pp 8749-8752

Mauro Angiolini, Domenico Fusar Bassini, Markus Gude and Maria Menichincheri*

A novel solid-phase method for the synthesis of 4-methyl-pyrido[2,3-d]pyrimidin-7-one compounds with two diversity points is described. The polymer supported methylsulfonyl derivatives A_3 , achieved by coupling compound G with different resin-bound amines A_1 followed by oxidation with MCPBA, are substituted with several amines R_1R_2NH . Final cleavage affords 126 compounds having formula H in good yield and purity.

The synthesis and characterization of phenylacetylene tripodal compounds containing boroxine cores Emily K. Perttu, Matthew Arnold and Peter M. Iovine*

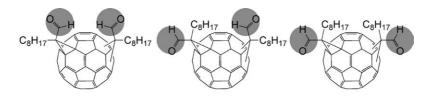
pp 8753-8756



Preparation and identification of bis(formylmethano)[60]fullerene isomers: the first systematic study on bifunctionalized [60]fullerenes with dissymmetric addends

pp 8757-8760

Hiroshi Ito, Yasuhiro Ishida and Kazuhiko Saigo*



Separation and characterization of regio- and diastereo-isomers



Synthesis of polyacetylenic acids isolated from *Nanodea muscosa* Diego Alves, Cristina W. Nogueira and Gilson Zeni*

pp 8761-8764

$$\sim$$
 COOH \rightarrow TeBu

СООН

Luminescence behaviour of calix[4]arenes bearing an increasing number of appended-pyrenes Hyun Jung Kim, Ju Han Bok, Jacques Vicens,* Il-Hwan Suh, Jaejung Ko and Jong Seung Kim* pp 8765-8768

$$\begin{array}{c} R_1 & R_2 & R_4 & R_3 \\ & 1 \ R_1 = R_3 = H, \ R_2 = R_4 = X \\ & 2 \ R_1 = C H_2 C O_2 E t, \ R_2 = R_3 = R_4 = H \\ & 3 \ R_1 = R_2 = R_3 = R_4 = C H_2 C O_2 E t \\ & 4 \ R_1 = R_2 = R_3 = R_4 = C H_2 C O_2 H \\ & 5 \ R_1 = R_2 = R_3 = R_4 = C H_2 C O_2 E t \\ & 6 \ R_1 = R_3 = C H_2 C O_2 E t, \ R_2 = R_4 = X \\ & 7 \ R_1 = C H_2 C O_2 E t, \ R_2 = R_3 = R_4 = X \\ & 8 \ R_1 = R_2 = R_3 = R_4 = X \end{array}$$

Indium-mediated mild and efficient one-pot synthesis of alkyl phenyl selenides

pp 8769-8771

Wanida Munbunjong, Eun Hwa Lee, Warinthorn Chavasiri* and Doo Ok Jang*

R-X + PhSeSePh
$$\frac{\text{In}}{\text{CH}_2\text{Cl}_2, \text{ reflux}}$$
 R-SePh

Selective reduction of thioacetal to sulfide by gallium(II) chloride

Kei-ichiro Ikeshita, Nobuhiro Kihara* and Akiya Ogawa

pp 8773-8775

$$R \stackrel{\mathsf{SR'}}{\longleftarrow} + \mathsf{Ga}_2\mathsf{Cl}_4 \stackrel{\mathsf{H}^+}{\longrightarrow} \mathsf{R} \stackrel{\mathsf{SR'}}{\longrightarrow}$$

Zr-Mediated hydroboration: stereoselective synthesis of vinyl boronic esters

pp 8777-8780

Yanong D. Wang,* Gregory Kimball, Amar S. Prashad and Yan Wang

An improved process for the preparation of (E)-vinylboronic esters via a Zr-mediated hydroboration of alkynes, especially oxygencontaining alkynes, is described.

Regioselective nitration of N,N-dialkylanilines using cerium(IV) ammonium nitrate in acetonitrile Xianghua Yang, Chanjuan Xi* and Yanfeng Jiang

pp 8781-8783

$$\begin{array}{c|c}
R & R^1 & \text{1eq CAN} \\
\hline
 & N & \text{CH}_2\text{CN}, \text{rt}
\end{array}$$

Proline-catalyzed aldol reactions of acyl cyanides with acetone: an efficient and convenient synthesis of 1,3-diketones

pp 8785-8788

Zongxuan Shen, Bin Li, Lu Wang and Yawen Zhang*

$$\begin{array}{c} \textbf{acetone} \\ \textbf{R} \\ \textbf{CN} \\ \textbf{L-proline} \\ \textbf{Aa-m} \\ \end{array} \\ \textbf{5a-m} \\ \textbf{6a-m} \\ \textbf{a:} \\ \textbf{R} = Ph; \\ \textbf{b:} \\ \textbf{R} = 4-CH_3-C_6H_4; \\ \textbf{c:} \\ \textbf{R} = 2-CH_3-C_6H_4; \\ \textbf{d:} \\ \textbf{R} = 4-CH_3O-C_6H_4; \\ \textbf{e:} \\ \textbf{R} = 2-F-C_6H_4; \\ \textbf{f:} \\ \textbf{R} = 4+F-C_6H_4; \\ \textbf{g:} \\ \textbf{R} = 2-CI-C_6H_4; \\ \textbf{h:} \\ \textbf{R} = 4-CI-C_6H_4; \\ \textbf{g:} \\ \textbf{R} = 2-Br-C_6H_4; \\ \textbf{j:} \\ \textbf{R} = 4-Br-C_6H_4; \\ \textbf{k:} \\ \textbf{R} = 4-NO_2-C_6H_4; \\ \textbf{l:} \\ \textbf{R} = Ph-CH=CH; \\ \textbf{R} = 4-R-CH_3O-C_6H_4; \\ \textbf{R} = 4-R-CH_$$

5-Nitro[2.2]paracyclophanepyran-6-one—building block for the synthesis of [2.2]paracyclophanes containing condensed benzofuran subunits

pp 8789-8792

Lucio Minuti,* Assunta Marrocchi, Ilaria Tesei and Eszter Gacs-Baitz

m: R = furan-2-vl

$$NO_2$$

R=R₁=Me; R=Me, R₁=H

New tetrakis(4-aminophenyl)ethenes: synthesis and electrochemical investigations

pp 8793-8797

Jean-François Kuntz, Raphaël Schneider,* Alain Walcarius and Yves Fort*

Four new tetrakis(4-aminophenyl)ethenes have been prepared and characterized by cyclic voltammetry, coulometry, electrolysis, UV-vis absorption, and EPR spectroscopy. The stability of (radical cation)s generated by oxidation depends on the substitution of the NR^1R^2 group.



Regioselective construction of polysubstituted pyridine ring from Baylis-Hillman adducts via sequential pp 8799-8803 introduction of tosylamide, Michael reaction, aldol condensation, and elimination of TsH

Da Yeon Park, Mi Jung Lee, Taek Hyeon Kim and Jae Nyoung Kim*

Peptide thioester preparation based on an N-S acyl shift reaction mediated by a thiol ligation auxiliary pp 8805–8807 Toru Kawakami, Megumi Sumida, Ken'ichiroh Nakamura, Thomas Vorherr and Saburo Aimoto*

OTHER CONTENTS

Corrigendump 8809Corrigendump 8811Contributors to this issuep IInstructions to contributorspp III-VI

*Corresponding author

** Supplementary data available via ScienceDirect

COVER

Regioselective construction method of polysubstituted pyridine skeleton has been developed starting from Baylis–Hillman adducts. The method involved sequential introduction of tosylamide, Michael reaction, aldol condensation, and elimination of TsH. *Tetrahedron Letters* **2005**, *46*, 8799–8803.

© 2005 J. N. Kim. Published by Elsevier Ltd.



Full text of this journal is available, on-line from **ScienceDirect**. Visit **www.sciencedirect.com** for more information.



This journal is part of **ContentsDirect**, the *free* alerting service which sends tables of contents by e-mail for Elsevier books and journals. You can register for **ContentsDirect** online at: http://contentsdirect.elsevier.com

Indexed/Abstracted in: AGRICOLA, Beilstein, BIOSIS Previews, CAB Abstracts, Chemical Abstracts, Chemical Engineering and Biotechnology Abstracts, Current Biotechnology Abstracts, Current Contents: Life Sciences, Current Contents: Physical, Chemical and Earth Sciences, Current Contents Search, Derwent Drug File, Ei Compendex, EMBASE/Excerpta Medica, Medline, PASCAL, Research Alert, Science Citation Index, SciSearch

