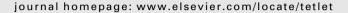


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## **Tetrahedron Letters**





## Tetrahedron Letters Vol. 50, No. 34, 2009

## **Contents**

#### COMMUNICATIONS

**A** microwave-assisted, green procedure for the synthesis of *N*-aryl sulfonyl and *N*-aryl pyrroles Matthew A. Wilson \*, Gary Filzen, Gregory S. Welmaker

pp 4807-4809

A simplified approach to the uncatalyzed Paal-Knorr condensation using microwave irradiation in water is described.

#### Generation of hexahydroazulenes

pp 4810-4812

Guido Krämer, Heiner Detert, Herbert Meier

Isolation, structure elucidation, and biological evaluation of the unusual heterodimer chrysoxanthone from the ascomycete IBWF11-95A

pp 4813-4815

Anja Schüffler, Johannes C. Liermann, Heinz Kolshorn, Till Opatz, Heidrun Anke

Chrysoxanthone

Chrysoxanthone, an unusual heterodimer linked through a diaryl ether bridge, was isolated from the ascomycete IBWF11-95A.



#### Lycochinines A-C, novel C<sub>27</sub>N<sub>3</sub> alkaloids from Lycopodium chinense

pp 4816-4819

Yusuke Hirasawa, Tomoyuki Tanaka, Koichiro Koyama, Hiroshi Morita

Three novel  $C_{27}N_3$ -type *Lycopodium* alkaloids, lycochinines A–C (**1-3**) consisting of an octahydroquinoline or a decahydroquinoline, a quinolizidine, and a piperidine, were isolated from the club moss *Lycopodium chinense*. The relative stereochemistry of **1-3** was determined by combination of NOESY correlations and chemical transformations.

Heterogeneous Suzuki and Stille coupling reactions using highly efficient palladium(0) immobilized MCM-41 catalyst pp 4820–4823 Sreyashi Jana, Satyajit Haldar, Subratanath Koner \*

Palladium(0) has been immobilized into the mesoporous silica to develop a catalyst for Suzuki and Stille coupling reactions. This recyclable heterogeneous catalyst shows excellent catalytic efficacy with high turnover frequency.



#### Andrographolactone, a unique diterpene from Andrographis paniculata

pp 4824-4826

Guo-Cai Wang, Ying Wang, Ian D. Williams, Herman Ho-Yung Sung, Xiao-Qi Zhang, Dong-Mei Zhang, Ren-Wang Jiang, Xin-Sheng Yao, Wen-Cai Ye $^{\circ}$ 

Andrographolactone (1), possessing an unprecedented diterpene skeleton, was isolated from the EtOAc extract of the aerial parts of Andrographis paniculata. Its structure was established by NMR, IR, UV, and HRESIMS data and subsequently confirmed by X-ray diffraction analysis. A possible biogenetic pathway of 1 was also proposed. Bioassay showed that 1 exhibited cytotoxic activity.



**Significant solvent effect in anomerization reaction of pyranosides with 2,3-***trans* **carbamate and carbonate** Shino Manabe \*, Yukishige Ito \*

pp 4827-4829

PO SPh 
$$\xrightarrow{30 \text{ °C}, 12h}$$
 PO or NBn anomerization BF<sub>3</sub>•OEt<sub>2</sub> PO SPh  $\xrightarrow{30 \text{ °C}, 12h}$  PO or NBn

A pronounced solvent effect was observed in the anomerization reaction of pyranosides with 2,3-trans carbamate and carbonate.

#### Acutaxylines A and B, two novel triterpenes from Dysoxylum acutangulum

pp 4830-4832

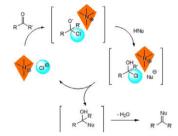
Intan Safinar Ismail, Yuta Nagakura, Yusuke Hirasawa, Takahiro Hosoya, Mohd Izwan Mohd Lazim, Nordin Hj Lajis, Hiroshi Morita

Two novel triterpenes, acutaxylines A (1) and B (2) were isolated from the leaves of *Dysoxylum acutangulum*. The relative stereochemistry of 1 and 2 was determined by NOESY correlations. Acutaxyline B showed moderate cytotoxicity against human blood premyelocytic leukemia cells.

#### Chloride ion pairs as catalysts for the alkylation of aldehydes and ketones with C-H acidic compounds

pp 4833-4837

Camille Carrignon, Philippe Makowski, Markus Antonietti, Frédéric Goettmann



Chloride anions associated with various soft cations (like tetraalkyl ammoniums, alkyl imidazoliums or pyridiniums) were shown to be able to promote the reaction of C–H acidic nucleophiles with aldehydes and ketones under relatively mild conditions.



An efficient green MCR protocol for the stereoselective synthesis of  $\beta$ -acetamido ketones catalyzed by Selectfluor

pp 4838-4843

V. S. Shinu, B. Sheeja, E. Purushothaman, D. Bahulayan  $^{\ast}$ 

CHO + CH<sub>3</sub>COCI 
$$\frac{\text{CH}_3\text{CN}}{\text{Selectfluor}^{\text{TM}} \text{ (5 mol}\%)}$$
  $\stackrel{\circ}{\text{NH}}$   $\stackrel{\circ}{\text{O}}$  2 h, rt



# A Pd-mediated intramolecular ketalization of alkynediols: construction of the central [3.2.1]-bicyclic ketal core of cyclodidemniserinol trisulfate

pp 4844-4847

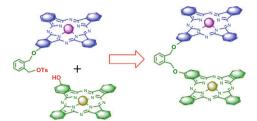
C. V. Ramana \*, Rosy Mallik, Gokarneswar Sahoo



# Heteroligand and heteronuclear clamshell-type phthalocyanines: selective preparation, spectral properties, and synthetic application

pp 4848-4850

Alexander Yu. Tolbin \*, Victor E. Pushkarev, Gennadiy F. Nikitin, Larisa G. Tomilova \*



A direct synthetic method to produce heteronuclear and heteroligand *clamshell*-type binuclear phthalocyanines is developed with the target compounds demonstrating the possibility to form sandwich-type heterocomplexes for the first time.



# A novel alkyne-induced recyclization of 4-hydroxymethyl or 4-formyl-1*H*-2,3-dihydroisoindoles—an effective pathway to substituted isobenzofurans

pp 4851-4853

Leonid G. Voskressensky \*, Larisa N. Kulikova, Alexey Kleimenov, Natalia Guranova, Tatiana N. Borisova, Alexey V. Varlamov

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



#### Organocatalytic asymmetric syn-selective direct aldol reactions in water

pp 4854-4856

Yong-Chua Teo \*, Guan-Leong Chua, Chin-Yee Ong, Chai-Yun Poh

A practical and convenient organocatalytic strategy is developed to provide a direct route to *syn*-selective aldol products in the presence of water. The siloxy serine organocatalyst mediates the direct aldol reaction of TBSO-protected hydroxyacetone with a variety of aldehydes to provide the aldol products in good yields and enantioselectivities up to 92%.



#### Novel synthesis of 3-aminopropionitriles by ring opening of 2-oxazolidinones with cyanide ion

pp 4857-4858

Tsuyoshi Taniguchi, Naoya Goto, Hiroyuki Ishibashi

$$R^{1}$$
 $N$ 
 $R^{2}$ 
 $+$ 
 $KCN$ 
 $\frac{18\text{-crown-6}}{100 \text{ °C}}$ 
 $R^{1}$ 
 $N$ 
 $R^{2}$ 
 $R^{2}$ 
 $R^{2}$ 



#### Chiral synthetic pseudopeptidic derivatives as triplet excited state quenchers

pp 4859-4862

Xavier J. Salom-Roig, Jean Martínez, M. Isabel Burguete, Francisco Galindo \*, Santiago V. Luis \*, Miguel A. Miranda, María C. Morant-Miñana, Julia Pérez-Prieto \*

A series of new synthetic pseudopeptidic quenchers of the triplet excited state of tiaprofenic acid derivatives are described. Intermolecular quenching constants have been determined by means of laser flash photolysis and significant stereodifferentiation has been found in some cases.

# Control over C-O and C-C bond formation: ruthenium catalyzed regiospecific addition of carboxylic acid to alkyne and stereoselective dimerization of alkyne

pp 4863-4865

Jyotsna Tripathy, Manish Bhattacharjee

 $Cat = [Ru(PPh_3)_2(CH_3CN)_3CI][BPh_4]$ 

A cationic ruthenium(II) complex, [Ru(PPh<sub>3</sub>)<sub>2</sub>(CH<sub>3</sub>CN)<sub>3</sub>CI][BPh<sub>4</sub>] (1), has been found to be an effective catalyst for stereoselective dimerization of alkynes in the presence of a base, and for regiospecific addition of carboxylic acids to alkynes in the presence of the Lewis acid, BF<sub>3</sub>·Et<sub>2</sub>O.



## $Pd(0) - catalyzed\ in tramolecular\ Heck\ reaction:\ a\ versatile\ route\ for\ the\ synthesis\ of\ dibenzo azocinone\ derivatives$

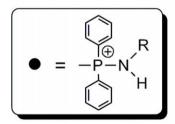
pp 4866-4869

K. C. Majumdar \*, Srikanta Samanta, Buddhadeb Chattopadhyay

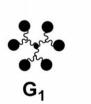
## Design of phosphonium ended dendrimers bearing functionalized amines

pp 4870-4873

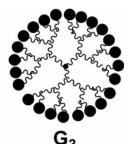
Marie-Agnés Lacour, Maria Zablocka, Anne-Marie Caminade \*, Marc Taillefer \*, Jean-Pierre Majoral \*



R = alkyl, heteroalkyl







#### Stereoselective synthesis of (22Z)-25-hydroxyvitamin $D_2$ and (22Z)-1 $\alpha$ , 25-dihydroxyvitamin $D_2$

pp 4874-4877

Zoila Gándara, Manuel Pérez, Xenxo Pérez-García, Generosa Gómez \*, Yagamare Fall \*



# C-N bond forming reaction under copper catalysis: a new synthesis of 2-substituted 5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinolines

pp 4878-4881

Mohosin Layek, A. V. Dhanunjaya Rao, Vikas Gajare, Dipak Kalita, Deepak Kumar Barange, Aminul Islam, K. Mukkanti, Manojit Pal \*



#### Novel synthetic route to the tricyclic core of (±)-galanthamine

pp 4882-4884

S. Chandrasekhar \*, Debjit Basu, M. Sailu, S. Kotamraju

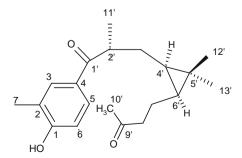
$$(-)- galanthamine$$

$$\begin{array}{c} & & & \\ &$$

## ${\bf Multidione,\ a\ novel\ diterpenoid\ from\ \it Jatropha\ multifida}$

pp 4885-4887

Biswanath Das<sup>\*</sup>, Keetha Laxminarayana, Martha Krishnaiah, Yallamalla Srinivas, Tuniki Venugopal Raju



## $Development\ of\ palladium (II) \hbox{-} catalyzed\ oxidative\ cyclization\ of\ olefinic\ keto\ and/or\ lactone\ esters$

pp 4888-4891

Ayaka Hibi, Masahiro Toyota

# Water-promoted highly regio- and stereoselective synthesis of $\alpha$ -dehydro- $\beta$ -amino esters and nitriles from Baylis-Hillman acetates

pp 4892-4895

Sudip Ghosh, Raju Dey, Kalicharan Chattopadhyay, Brindaban C. Ranu

$$R^1$$
 = H, Me, OMe, CI, Br  $X = CO_2Me$ , CN  $X = CO_2Me$   $X = CO_2Me$ 

# Enantioselective conjugate addition of fluorobis(phenylsulfonyl)methane to $\alpha,\beta$ -unsaturated ketones catalyzed by chiral bifunctional organocatalysts

pp 4896-4898

Hyoung Wook Moon, Min Je Cho, Dae Young Kim

Ph 
$$\frac{O}{Me} + F + \frac{SO_2Ph}{SO_2Ph} + \frac{cat.}{MTBE} + \frac{PhO_2S}{PhO_2S} + \frac{Cat.}{Me} + \frac{O}{NH_2} + \frac{O}{N$$



## A new strategy for the stereoselective synthesis of 2,2'-bipyrrolidines

pp 4899-4902

Mary J. Gresser, Paul A. Keller \*, Steven M. Wales

A new strategy for the stereoselective synthesis of the 2,2'-bipyrrolidine scaffold is presented using a metathesis reaction followed by asymmetric dihydroxylation for the introduction of the stereogenic elements. This straightforward high-yielding process is suitable for application to the synthesis of additional heterocycles.

# Methyl 2-methoxytetrafluoropropionate as a synthetic equivalent of methyl trifluoropyruvate in the Claisen condensation. The first synthesis of 2-(trifluoroacetyl)chromones and 5-aryl-2-hydroxy-2-(trifluoromethyl)furan-3(2H)-ones

pp 4903-4905

Roman A. Irgashev, Vyacheslav Ya. Sosnovskikh \*, Nataliya Kalinovich, Olesya Kazakova, Gerd-Volker Röschenthaler

## Direct reductive aminations with catalytic molybdenum dioxide dichloride and phenylsilane

pp 4906-4911

Clive A. Smith \*, Laura E. Cross, Kimberley Hughes, Rebecca E. Davis, Duncan B. Judd, Andrew T. Merritt

$$\begin{array}{c} R^{1} & \text{MoO}_{2}\text{Cl}_{2}, \text{PhSiH}_{3}, \text{AcOH}, \\ & \text{MeOH}, \text{H}_{2}\text{O}, \text{RT} \\ \\ R^{2} & \text{HN} \\ \\ R^{3} & \text{R}^{4} \\ \\ R^{1} = \text{H}, \text{R}^{2} = \text{SO}_{3}\text{PFP} \\ R^{1} = \text{H}, \text{R}^{2} = \text{OMe} \\ R^{1} = \text{H}, \text{R}^{2} = \text{OMe} \\ R^{1} = \text{R}^{2} = \text{OMe} \\ \\ R^{3} = \text{R}^{4} = \text{cycloalkyl} \\ \\ R^{3} = \text{H}, \text{R}^{4} = \text{aryl} \\ \end{array}$$

A powerful direct reductive amination (DRA) method is developed, using catalytic MoO<sub>2</sub>Cl<sub>2</sub> and with PhSiH<sub>3</sub> as the reducing agent.



# An efficient metal-free reduction using diphenylsilane with (tris-perfluorophenyl)borane as catalyst MeiXuan Tan, Yugen Zhang $^{*}$

pp 4912-4915

2 eq. Ph<sub>2</sub>SiH<sub>2</sub>
2 mol% B(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>
Yield: 99%

An efficient metal-free reduction of various C=X (X=O,N,C) bonds to their corresponding amines or hydrocarbons using the  $Ph_2SiH_2/B(C_6F_5)_3$  catalytic system is demonstrated. This protocol reduces enamines, enol esters, carbonyls, amides, and isocyanates.



## A one-pot synthesis of imidazo[1,5-a]pyridines

pp 4916-4918

James M. Crawforth \*, Melissa Paoletti

$$R^1$$
 $NH_2 + R^2CO_2H$ 

A one-pot synthesis of imidazo[1,5-a]pyridines starting from a carboxylic acid and 2-methylaminopyridines is achieved using propane phosphoric acid anhydride in ethyl or n-butyl acetate at reflux.

#### Tandem nitrosation/cycloaddition of heterocyclic enamines using nitrolic acids

pp 4919-4921

Cevher Altuğ, Yasar Dürüst \*, Mark C. Elliott \*, Benson M. Kariuki

#### Stabilisation of alkylcarbamate anions using neutral hydrogen bond donors

pp 4922-4924

Peter R. Edwards, Jennifer R. Hiscock, Philip A. Gale

A series of urea-based anion receptors are shown to bind to alkylcarbamate anions formed by the reaction of primary amines with carbon dioxide in DMSO-d<sub>6</sub>.



#### Microwave-assisted synthesis of pyrrolidine derivatives

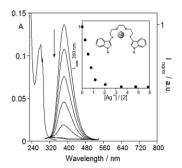
pp 4925-4929

Yuan-Chun Chang, Jiun-Ly Chir, Shuan-Yi Tsai, Wei-Fu Juang, An-Tai Wu \*

# Synthesis and characterization of novel indole-containing half-crowns as new emissive metal probes

pp 4930-4933

Angelo Rocha, M. Manuel B. Marques \*, Carlos Lodeiro



# A novel method for enzymatic asymmetric reduction of ketones in a supercritical carbon dioxide/water biphasic system

pp 4934-4936

Tadao Harada, Yuki Kubota, Takashi Kamitanaka, Kaoru Nakamura, Tomoko Matsuda \*

## Total synthesis of the proposed structures of hyacinthacines C2, C3, and their C5-epimers

pp 4937-4940

Tetsuya Sengoku, Yasutaka Satoh, Masaki Takahashi, Hidemi Yoda

# Preparation of all stereoisomers of 2-allyl-2-methyl-3-hydroxycyclopentanone by desymmetric processes based on a microbial oxidation and reduction system

pp 4941-4944

Mikio Fujii \*, Minoru Takeuchi, Hiroyuki Akita, Kaoru Nakamura

\*Corresponding author

(f) Supplementary data available via ScienceDirect

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