

#### Tetrahedron Letters Vol. 47, No. 47, 2006

### **Contents**

#### **COMMUNICATIONS**

#### Electrosynthesis of tryptanthrin

pp 8201-8203

Belen Batanero and Fructuoso Barba\*

Highly-loaded amphiphilic polyimino resin: quench reagent and solid support for peptide synthesis Houcine Rahali and Didier Stien\*

pp 8205-8207

$$= \bigcirc NH$$

A new liquid crystalline derivative of dibenzotetraaza[14]annulene: synthesis, characterization and the preliminary evaluation of mesomorphic properties

pp 8209-8213

Jarosław Grolik, Lesław Sieroń and Julita Eilmes\*



#### Facile electrochemical transformation of diazonium salts into carboxylic acids

pp 8215-8216

M. Dolores Otero, Belen Batanero and Fructuoso Barba\*

$$Ar-N_2^+BF_4^- + CO_2 \xrightarrow{\text{cathodic} \\ \text{reduction}} Ar-COO^-$$

#### Macrocycles with two exclusive hydrogen-bonding modes

pp 8217-8220

Min Kyung Chae, Geun-Young Cha and Kyu-Sung Jeong\*

A series of large, 44-membered macrocycles were efficiently synthesized and characterized, which have two exclusive binding modes in a diagonal manner.

### 2,2'-Biphenyldiol-bridged bis(free base porphyrin): synthesis and chiroptical probing of asymmetric amino alcohols

pp 8221-8225

Yusuke Ishii, Yoichi Onda and Yuji Kubo\*

# Kinetic resolution of d,l-1,2-diols catalyzed by amine-phosphinite bifunctional organocatalysis derived from quinidine

pp 8227-8229

Shinya Mizuta, Yutaka Ohtsubo, Takeo Tsuzuki, Tetsuya Fujimoto\* and Iwao Yamamoto

$$\begin{array}{c} \text{catalyst (30 mol\%)} \\ \text{R} & \begin{array}{c} \text{p-CF}_3\text{C}_6\text{H}_4\text{COCI (0.65 equiv.)} \\ \text{Pr}_2\text{NEt (0.5 equiv.)} \\ \end{array} \\ \text{EtCN, -78 °C, 1 h} \\ \\ \text{R} & \begin{array}{c} \text{OCOC}_6\text{H}_4\text{-}p\text{-CF}_3 \\ \end{array} \\ \text{R} & \begin{array}{c} \text{OCOC}_$$

#### Palladium-catalyzed arylation of α,α-difluoro-allylic-β-hydroxyester

pp 8231-8234

Xiang Fang, Xueyan Yang, Xianjin Yang, Min Zhao, Guorong Chen and Fanhong Wu\*

### Asymmetric allylation of N-benzoylhydrazones promoted by novel $C_2$ -symmetric bis-sulfoxide organocatalysts

pp 8235-8238

Fred García-Flores, Luz S. Flores-Michel and Eusebio Juaristi\*

NHCOPh
$$H + SiCl_3 = L^* - R^* C$$

$$CH_2Cl_2 / -78 °C - R^* C$$

$$up to 76\% ee$$

$$L^* = P_{p-tol} S_{l/l/O} = P_{$$

Solvent-free facile synthesis of novel α-tosyloxy β-keto sulfones using [hydroxy(tosyloxy)iodo]benzene pp 8239–8241 Dalip Kumar,\* M. Swapna Sundaree, Gautam Patel, V. S. Rao and Rajender S. Varma\*

$$\begin{array}{c|c}
 & O & O & O \\
 & S & \hline
 & C_6H_5I(OH)OTs \\
 & & \text{neat, rt} & \\
 & & OTs
\end{array}$$

A facile, general and high yielding protocol for the synthesis of novel  $\alpha$ -tosyloxy  $\beta$ -keto sulfones is described utilizing relatively non-toxic, [hydroxy(tosyloxy)iodo]benzene, under solvent-free conditions at room temperature.

### Synthesis of triprenylated toluquinone and toluhydroquinone metabolites from a marine-derived *Penicillium* fungus

pp 8243-8246

Brent A. Scheepers, Rosalyn Klein and Michael T. Davies-Coleman\*

Marine fungal metabolites 1 and 2 were synthesized from 2-methyl-1,4-benzoquinone in four and five steps, respectively.



# A novel method for the highly efficient synthesis of 1,2-benzisoxazoles under neutral conditions using the pp~8247-8250 $Ph_3P/DDQ$ system

Nasser Iranpoor,\* Habib Firouzabadi\* and Najmeh Nowrouzi

$$R = H$$
, alkyl, aryl 
$$X = H, CH_3, C_2H_5, OH, OCH_3, Br$$

#### 2-Chloroquinazoline. Synthesis and reactivity of a versatile heterocyclic building block

pp 8251-8254

Signe Teuber Henriksen and Ulrik Svane Sørensen\*

A Stille-type coupling using tributyltin hydride as a mild and selective way of converting 2,4-dichloroquinazoline to 2-chloroquinazoline and the reactivity of this heterocyclic building block is discussed.



#### Mild and efficient methods for the conversion of benzylic bromides to benzylic thiols

pp 8255-8258

Chien-Chung Han\* and R. Balakumar

$$BzSH \xrightarrow{\text{(ii) HSAc/K}_2CO_3/THF/0.5 h} BzBr \xrightarrow{HSAc} BzSF \xrightarrow{\text{(iii) MeOH/0.5 h}} BzSF$$

Very mild and efficient methods have been developed to synthesize benzylic thiols from benzylic bromides at room temperature to give 94-99% yield within 1 h under  $N_2$ .

#### Ugi/xanthate cyclizations as a radical route to lactam scaffolds

pp 8259-8261

Laurent El Kaïm,\* Laurence Grimaud, Luis Demetrio Miranda\* and Emilie Vieu

A new preparation of lactams by a two step Ugi/xanthate radical cyclization procedure.

Anti-AIDS agents 68. The first total synthesis of a unique potent anti-HIV chalcone from genus *Desmos* pp 8263–8266 Kyoko Nakagawa-Goto and Kuo-Hsiung Lee\*



Synthetic studies toward bryostatin 1: preparation of a C<sub>1</sub>–C<sub>16</sub> fragment by pyran annulation Gary E. Keck,\* Dennie S. Welch and Yam B. Poudel

pp 8267-8270



Synthesis of hexopyranosyl acetates and 2,3-disubstituted tetrahydropyrans via chemoselective hydrogenation of hex-2-enopyranosyl acetates

pp 8271-8274

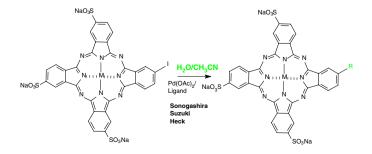
Kaname Sasaki, Takayuki Wakamatsu, Shuichi Matsumura and Kazunobu Toshima\*

$$(PO)_{n} \xrightarrow{Q} \underbrace{\begin{array}{c} 2 \text{ H}_{2}, \text{ Pd/C} \\ \text{EtOH/AcOH} \end{array}} \\ (PO)_{n} \xrightarrow{Q} \underbrace{\begin{array}{c} O \\ \text{NOAc} \end{array}} \underbrace{\begin{array}{c} H_{2}, \text{ Rh/Al}_{2}O_{3} \\ \text{EtOAc/toluene} \end{array}} \\ (PO)_{n} \xrightarrow{Q} \underbrace{\begin{array}{c} O \\ \text{NOAc} \end{array}} \\ (PO)_{n} \xrightarrow{Q} \underbrace{\begin{array}{c} O \\ \text$$

### Functionalization of sulfophthalocyanines in aqueous medium by palladium-catalyzed cross-coupling reactions

pp 8275-8278

Hasrat Ali, Olivier St-Jean, Jean-Philip Tremblay-Morin and Johan E. van Lier\*



# Synthesis and post-synthetic derivatization of a cyanine-based amino acid. Application to the preparation pp 8279–8284 of a novel water-soluble NIR dye

Bertrand Chipon, Guillaume Clavé, Cédric Bouteiller, Marc Massonneau, Pierre-Yves Renard\* and Anthony Romieu\*

### The ring opening of 3,4-dichloro-1,2,5-thiadiazole with metal amides. A new synthesis of 3,4-disubstituted-1,2,5-thiadiazoles

pp 8285-8288

Alain Merschaert,\* Pascal Boquel, Hugo Gorissen, Jean-Pierre Van Hoeck, Alfio Borghese, Luc Antoine, Vincent Mancuso, Anne Mockel and Michel Vanmarsenille

We have developed a new synthesis of 3,4-disubstituted-1,2,5-thiadiazoles. The methodology is based on the ring opening of readily available 3,4-dichloro-1,2,5-thiadiazole with metal amides to afford a stable synthon, which is then transformed into the 3,4-disubstituted-1,2,5-thiadiazole derivatives via two consecutive reactions with O-, S-, N- or C-nucleophiles.

### Microwave-assisted one-pot synthesis of substituted tetrahydrocarbazole and 8,9,10,11-tetrahydro-7*H*-pvrido[*a*]carbazoles

pp 8289-8292

Vera Barbieri and Maria Grazia Ferlin\*

Optimization procedures of process variables, power, temperature, and irradiation time are reported in detail for microwave Fischer indole synthesis of substituted tetrahydrocarbazole and tetrahydropyridocarbazole derivatives.



#### Palladium-catalyzed aerobic oxidation of amines

pp 8293-8297

Jia-Rui Wang, Yao Fu, Bei-Bei Zhang, Xin Cui, Lei Liu\* and Qing-Xiang Guo\*



### Synthesis of 1,7-epoxycyclononanes and 1,8-epoxycyclodecanes by $\beta$ -fragmentation reactions using LTA $\,$ pp 8299–8304 and $I_2$

Ángel M. Montaña\* and Stefano Ponzano

X=H, I X=I, OAc The treatment of derivatives on C3 of 6-hydroxy-2,7-dimethyl-11-oxatricyclo[6.2.1.0<sup>2.6</sup>]undecan-4-one with lead tetraacetate and iodine, gave, in a good yield, 1,7-epoxycyclononanes or 1,8-epoxycyclodecanes, depending on the type of substitution in  $\alpha$  to the ketone on C4 of substrates.

#### Synthetic studies on a phenyl-laulimalide analogue

pp 8305-8308

Christelle Faveau, Martine Mondon, Jean-Pierre Gesson,\* Tobias Mahnke, Sandra Gebhardt and Ulrich Koert\*

# Heteropolyacid encapsulated into mesoporous silica framework for an efficient preparation of 1,1-diacetates from aldehydes under a solvent-free condition

pp 8309-8312

Jianmin Wang, Liang Yan, Guang Qian, Keli Yang, Haitao Liu and Xiaolai Wang\*

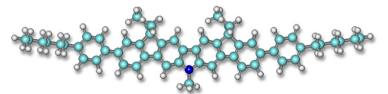
RCHO + Ac<sub>2</sub>O 
$$\xrightarrow{\text{SiPW-8}}$$
 RCH(OAc)<sub>2</sub> room temperature, 10-30min R = alkyl, aryl

Acylals were efficiently prepared from aliphatic and aromatic aldehydes using mesoporous silica with heteropolyacid encapsulated into their framework (SiPW-8) as catalysts by a solvent-free procedure.

#### Novel bisindenocarbazole derivative exhibiting a nematic mesophase

pp 8313-8317

Martin Sonntag and Peter Strohriegl\*



In this letter we describe the synthesis of the first liquid crystalline bisindenocarbazole derivative. The novel bisindenocarbazole exhibits a broad nematic mesophase between 180 and 250 °C, which was characterized by polarizing microscopy and small angle X-ray scattering. The material shows an excellent electrochemical stability and a strong blue fluorescence with a quantum yield of 49% in solution.

#### Reactions of dicarbanion equivalents generated from complexation of 1,3-dienes on Ti(II) moiety

pp 8319-8322

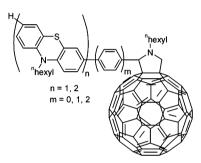
Johann Baraut, Arnaud Perrier, Virginie Comte, Philippe Richard, Pierre Le Gendre\* and Claude Moïse\*

#### First syntheses and electronic properties of (oligo)phenothiazine– $C_{60}$ dyads

pp 8323-8327

Nadine Bucci and Thomas J. J. Müller\*

(Oligo)phenothiazine– $C_{60}$ -dyads are synthesized by a three-component condensation–cycloaddition. Cyclic voltammetry shows that donor and acceptor are decoupled in the ground state, whereas upon UV excitation a considerable fluorescence quenching is observed.



### Synthesis and electronic properties of (oligo)phenothiazine-ethynyl-hydro- $C_{60}$ dyads

pp 8329-8332

Nadine Bucci and Thomas J. J. Müller\*

(Oligo)phenothiazine-ethynyl-hydro- $C_{60}$  dyads are synthesized by addition of the corresponding acetylides to  $C_{60}$  followed by protonation. Cyclic voltammetry shows that donor and acceptor are decoupled in the ground state, whereas upon UV excitation considerable fluorescence quenching is observed.

### 1,3-Thiazoline-2-thione-4,5-dithiolato, an efficient building block towards functionalized dithiadiazafulvalenes

pp 8333-8336

Samar Eid, Michel Guerro and Dominique Lorcy\*

$$\begin{bmatrix} S & S & S & N & N \\ S & S & S & N & N \\ N & S & S & S \end{bmatrix} (NEt_4)_2 \qquad \begin{bmatrix} Me & Me \\ S & N & S \\ N & S & N & S \\ N & Me & S \end{bmatrix}$$

[NEt4]<sub>2</sub>[Zn(Me-thiazdt)<sub>2</sub>]

BEDT-DTDAF

A simple and efficient approach to *N*-methyl-1,3-thiazoline-2-thione-4,5-dithiolate, easily isolated as its zinc dithiolene complex, has been developed. The reactivity of this dithiolene complex as a nucleophilic dithiolate synthon and as a precursor of the air sensitive electron rich olefine, the bis ethylenedithiodithiadiazafulvalene (BEDT–DTDAF), is also described.

#### The lithiation of 2-chloroglucal derivatives

pp 8337-8341

Ewan Boyd, Michael R. Hallett, Ray V. H. Jones, James E. Painter, Prakash Patel, Peter Quayle\* and Anita J. Waring (née Potts)

The metallation of 2-chloroglucals is described.

Pyridinium *N*-2'-pyridylaminide: radical cyclization in the synthesis of annulated 2-aminopyridines Aránzazu Sánchez, Araceli Núñez, Carolina Burgos\* and Julio Alvarez-Builla\*

pp 8343-8346

### **(i)**+

#### CAN-mediated rearrangement of 4-benzhydrylidenepiperidines

Meng-Yang Chang,\* Tsun-Cheng Wu, Chun-Yu Lin and Ching-Yi Hung

pp 8347-8350

#### Direct-type catalytic three-component Mannich reaction in aqueous media

Thierry Ollevier,\* Etienne Nadeau and Andrée-Anne Guay-Bégin

pp 8351-8354

### Total synthesis of the putative structure of the novel triquinane based sesquiterpenoid natural product pp 8355-8360 dichomitol

Goverdhan Mehta\* and Kotapalli Pallavi

### Alternative synthetic routes to 2',3'-didehydro-2',3'-dideoxy-5-hydroxymethyluridine

pp 8361-8363

Raymond Chung and Karen S. Anderson\*

#### 36- and 42-Membered cyclophosphazene-containing macrocycles

pp 8365-8368

Vadapalli Chandrasekhar,\* Gurusamy Thangavelu Senthil Andavan, Ramachandran Azhakar and Balasubramanian Murugesa Pandian

Novel 36- and 42-membered cyclophosphazene-containing macrocycles were obtained by [2+2] condensation reactions of  $N_3P_3(O_2C_{12}H_8)_2[-O-C_6H_4-p\text{-}CHO]_2$  with  $PhP(O)[N(Me)NH_2]_2$  or 1,6-diaminohexane.

### $Hg(OTf)_2\hbox{-}Catalyzed\ cyclization\ of\ alkynyl\ \textit{tert-} butyl carbonate\ leading\ to\ cyclic\ enol\ carbonate$

pp 8369-8373

Hirofumi Yamamoto, Mami Nishiyama, Hiroshi Imagawa and Mugio Nishizawa\*



#### Microwave-promoted synthesis of polyhydroxydeoxybenzoins in ionic liquids

pp 8375-8378

Ullastiina Hakala and Kristiina Wähälä\*

A microwave-promoted synthesis of polyhydroxydeoxybenzoins and -phenylpropanones has been developed, using a bis{(trifluoromethyl)sulfonyl}amine (HNTf<sub>2</sub>) or BF<sub>3</sub>·OEt<sub>2</sub> in an ionic liquid solvent.

### Use of a solid-supported coupling reagent for a selective phosphitylation of the primary alcohol of $N^2$ -isobutyryl-2'-deoxy or 2'-O-methyl guanosine

pp 8379-8382

Ivan Zlatev, Yukiko Kato, Albert Meyer, Jean-Jacques Vasseur and François Morvan\*

HO 
$$G^{iBu}$$
  $PV$   $NH$   $O_3S$   $NC$   $R$   $R$   $O$   $N$   $O$   $N$ 

#### Capping the upper and lower rims of calix[4]arenes by aryl dinitrile oxide reactions

pp 8383-8386

Ya-Jiun Shiao, Pei-Chen Chiang, Annamalai Senthilvelan, Ming-Tsung Tsai, Gene-Hsiang Lee and Wen-Sheng Chung\*

### **(i)**+

#### Lewis acid-promoted $\alpha$ -hydroxy $\beta$ -dicarbonyl to $\alpha$ -ketol ester rearrangement

pp 8387-8390

Xuechuan Hong, José M. Mejía-Oneto and Albert Padwa\*

#### Addition of benzyltrimethylsilane to imines triggered by tetrabutylammonium fluoride

pp 8391-8393

Wan-Xuan Zhang, Chang-Hua Ding, Zhi-Bin Luo, Xue-Long Hou\* and Li-Xin Dai

$$R^{1}$$
  $N$   $R^{2}$   $\frac{\text{4A MS}}{\text{PhCH}_{2}\text{SiMe}_{3}}$   $\frac{\text{HN}}{\text{5% - 15\% TBAF}}$   $R^{1}$  = alkyl, aryl;  $R^{2}$  = aryl, sulfinyl 31-85% yield 59-86% de

### A facile and catalytic method for selective deprotection of *tert*-butyldimethylsilyl ethers with copper(II) bromide

pp 8395-8399

Suchitra Bhatt and Sandip K. Nayak\*

R-OTBDMS 
$$\xrightarrow{\text{CuBr}_2 (30 \text{ mol}\%)}$$
 R-OH  $\xrightarrow{\text{R-OH}}$  R-OH 67-100%

# Synthesis of novel dimeric cationic lipids based on an aromatic backbone between the hydrocarbon chains and headgroup

pp 8401-8405

Bishwajit Paul, Avinash Bajaj, S. S. Indi and Santanu Bhattacharya\*

$$H_{33}C_{16}O$$
 $OC_{16}H_{33}$ 
 $OC_{16}H_{33}$ 
 $OC_{16}H_{33}$ 

Novel dimeric cationic lipids with an aromatic anchor between the hydrocarbon chains and headgroup bearing different spacers have been synthesized.

# Color modulation for intramolecular charge-transfer-induced chemiluminescence of bicyclic dioxetanes bearing a 3-hydroxy-5-naphthylphenyl moiety in the coordination sphere

pp 8407-8411

Masakatsu Matsumoto,\* Kazutaka Yamada, Harumi Ishikawa, Naoyuki Hoshiya, Nobuko Watanabe and Hisako K. Ijuin



### Synthesis towards complex bridged alkaloids derived from diketopiperazines: a cationic cascade approach to stephacidins, paraherquamides and related systems

pp 8413-8417

Mark Pichowicz, Nigel S. Simpkins,\* Alexander J. Blake and Claire Wilson

#### Chiral single-stranded metallohelix: metal-mediated folding of linear oligooxime ligand

pp 8419-8422

Shigehisa Akine, Takanori Taniguchi and Tatsuya Nabeshima\*

Effective chirality induction  $(M = Ca^{2+})$ 

# Aryl pyrrolidinones via radical 1,4-aryl migration and 5-endo-trig cyclisation of N-(2-bromoallyl)arylcarboxamides

pp 8423-8425

Matthew J. Palframan, Kirill Tchabanenko\* and Jeremy Robertson

# Formation of substituted N-oxide hydroxyquinolines from o-nitrophenyl Baylis-Hillman adduct: a new key intermediate intercepted by ESI-(+)-MS(IMS) monitoring

pp 8427-8431

Giovanni W. Amarante, Mario Benassi, Adão A. Sabino, Pierre M. Esteves, Fernando Coelho\* and Marcos N. Eberlin\*

Data collected from ESI-(+)-MS(/MS) monitoring and probed by experiments in solution reveals the participation of a new key intermediate (4) in the mechanism of formation of N-oxide hydroxyquinolines (3) from o-nitrophenyl Baylis–Hillman adducts (1).



### Preparation and structural characterization of a new class of stable thioketones: *ortho*-hydroxythioacetophenones

pp 8433-8435

Trung Thanh Nguyen, Thach Ngoc Le, Poul Erik Hansen and Fritz Duus\*

# The first example of Diels–Alder cycloaddition of $\it ortho$ -xylylenes to $\it meso$ -tetraarylporphyrins containing electron-deficient $\beta$ , $\beta$ -double bonds

pp 8437-8440

Stanisław Ostrowski\* and Przemysław Wyrębek

β-Nitro-*meso*-tetraphenylporphyrin and the 2,7-dinitro-derivative, or their zinc complexes, react with *ortho*-xylylenes, giving rise to chlorins, bacteriochlorins or isobacteriochlorins.



\*Corresponding author

(1) Supplementary data available via ScienceDirect

Available online at www.sciencedirect.com



Abstracted/indexed 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. Also covered in the abstract and citation database SCOPUS<sup>®</sup>. Full text available on ScienceDirect<sup>®</sup>

