



Tetrahedron Letters Vol. 45, No. 7, 2004

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COMMUNICATIONS

First isolation of 1,3,2-dithiaphosphetane 2-sulfide

pp 1331-1334

Hideaki Oshida, Akihiko Ishii* and Juzo Nakayama*

Ans-P-Ans
$$S = P - Ans$$

$$S = P - Ans$$

$$R = 1 - Ad, t - Bu$$

$$R = S = P - MeOC_6H_4$$

$$R = 1 - Ad, t - Bu$$

$$R = S = R$$

$$R = R - Ans$$

The reaction of *tert*-alkyl phenyl thioketones and 2-adamantanethione with Lawesson's reagent yielded the corresponding 1,3,2-dithiaphosphetane 2-sulfides in high yields.

Efficient trapping of the intermediates in the photooxygenation of sulfides by aryl selenides and selenoxides

pp 1335-1337

Nikoletta Sofikiti, Constantinos Rabalakos and Manolis Stratakis*

No Reaction
$$Ar \stackrel{1}{\searrow} Se$$
 $Ar \stackrel{1}{\searrow} Ar \stackrel{1}{\searrow} Ar \stackrel{0}{\searrow} Ar \stackrel{0}{\Longrightarrow} Ar$

Unidirectional helical assembly via triple hydrogen bonds between chiral tris(oxazoline) and achiral tris(imidazoline)

pp 1339-1342

Seong Ryong Nam, Hae-Jo Kim, Shigeru Sakamoto, Kentaro Yamaguchi and Jong-In Hong*

A novel one-pot synthesis of derivatives of aryldioxins and aryldithiins

pp 1343-1346

Patcharee Preedasuriyachai, Porntip Charoonniyomporn, Osit Karoonnirun, Tienthong Thongpanchang* and Yodhathai Thebtaranonth

Ar
$$\longrightarrow$$
 OH \longrightarrow HX XH \longrightarrow Ar \longrightarrow Ar \longrightarrow XX R

Ar = naphthalene, phenanthrene X = O, S

Efficient modification of steroid 20S-hydroxy functionality for industrial preparation of $1\alpha,25$ -dihydroxy-22-oxavitamin D_3 , Maxacalcitol

pp 1347–1350

Hitoshi Shimizu,* Kazuki Shimizu, Noboru Kubodera, Kenichi Yakushijin and David A. Horne

Applications of chiral allenylzinc additions and Noyori asymmetric reductions to an enantioselective synthesis of a C3–C13 precursor of the polyketide phosphatase inhibitor cytostatin

pp 1351-1353

James A. Marshall* and Keith Ellis

Preparation of novel analgesics via diastereoselective nucleophilic addition to 1-dimethylamino-2-methylpentan-3-one

pp 1355-1357

Ornella Azzolina,* Simona Collina, Gloria Brusotti, Daniela Rossi, Laura Linati, Enrica Lanza and Victor Ghislandi

Relative Solvent, T°C
$$C_2H_5COCH-CH_2N(CH_3)_2$$
 $R = H, OH, OTHP$

Naphthyl amino alcohols were prepared via nucleophilic addition to racemic 1-dimethylamino-2-methylpentan-3-one. The use of the appropriate experimental conditions allowed the synthesis of both diastereoisomers.

Neoorthosiphonone A; a nitric oxide (NO) inhibitory diterpene with new carbon skeleton from *Orthosiphon stamineus*

pp 1359-1362

Suresh Awale, Yasuhiro Tezuka, Mitsuo Kobayashi, Jun-ya Ueda and Shigetoshi Kadota*

N-Tosylaziridine, a new substrate for chemical fixation of carbon dioxide via ring expansion reaction under atmospheric pressure

pp 1363-1365

Atsushi Sudo, Yosuke Morioka, Fumio Sanda and Takeshi Endo*

$$\begin{array}{c}
Ts \\
N \\
R
\end{array}
+ CO_2$$

$$\begin{array}{c}
O \\
1 \text{ atm} \\
LiBr
\end{array}$$

Sonogashira cross-coupling reactions catalysed by heterogeneous copper-free Pd-zeolites Laurent Djakovitch* and Patrick Rollet

pp 1367-1370

 $R = p\text{-OCH}_3$, $o\text{-CH}_3$, H, p-F, $p\text{-COCH}_3$, $p\text{-NO}_2$, $o\text{-NO}_2$

Cetyltrimethylammonium dichromate: a mild oxidant for coupling amines and thiols Sabita Patel and B. K. Mishra*

pp 1371-1372

$$Ar - NH_2 \xrightarrow{[C_{16}H_{33}N(CH_3)_3]_2Cr_2O_7} Ar - N=N-Ar$$

$$[C_{16}H_{33}N(CH_3)_3]_2Cr_2O_7$$

$$R - SH \xrightarrow{R-SS-R} R-SS-R$$

A new 'one-pot' synthesis of 2-substituted 3-nitro pyrrolidines through a multicomponent domino reaction

pp 1373-1375

Nikla Baricordi, Simonetta Benetti,* Gisella Biondini, Carmela De Risi and Gian P. Pollini

R-CHO +
$$R^1NH_2$$
 $\xrightarrow{MgSO_4}$ R-CH=N-R¹ $\xrightarrow{O_2N}$ $\xrightarrow{$

Dilactams. Synthesis of nonsymmetrical dibenzodiazocinediones

Alfred Hassner,* Bin Sun, Gary Gellermann and Simcha Meir

pp 1377-1379

Two step synthesis of nonsymmetrical dibenzodiazocinediones.

Solid phase synthesis of mandelic acid-derived thioethers by α-keto carbocation trapping Jean-Sébastien Fruchart, Jean-Bernard Behr and Oleg Melnyk*

pp 1381-1383

A new type of phase-transfer catalysis via continuous transfer of fluoride anions to the organic phase in the form of potassium difluorotriphenylstannate

pp 1385-1386

Mieczysław Mąkosza* and Robert Bujok

$$R - X_{org} + KF_{solid} \xrightarrow{Ph_3SnF} R - F_{org} + KX_{solid}$$

Ph₃SnF acts as phase-transfer catalyst

Complex formation between water-soluble sulfonated calixarenes and C_{60} fullerene

pp 1387-1390

Sándor Kunsági-Máté,* Kornélia Szabó, István Bitter, Géza Nagy and László Kollár

$$\begin{array}{c|c}
SO_3Na & SO_3Na \\
\hline
OH & OH \\
1 & 2
\end{array}$$

Photoluminescence studies revealed that water-soluble calixarene sulfonate derivatives 1 and 2 form complexes with the C_{60} fullerene with 2:1 and 1:1 stoichiometry, respectively.

Metathetic approach to naphthoxepin and spirocyclic molecular frameworks

pp 1391-1394

Sambasivarao Kotha* and Kalyaneswar Mandal

$$0 \mapsto 0 \mapsto 0$$

An efficient method for the synthesis of naphthoxepin and spirocyclic skeletons starting from β -naphthol has been developed. The Claisen rearrangement and the ring-closing metathesis reaction are used as key steps for their assembly.

Synthesis of a new bifunctional chelating agent for samarium complexation

pp 1395-1397

Ali Ouadi,* Karine Bultel, Agnes de France-Robert, Anthony Loussouarn, Laurence Morandeau and Jean-Francois Gestin

$$H_2N$$
 H_2N
 H_2N

Cystine mimetics—solid phase lanthionine synthesis

pp 1399-1401

Mizio Matteucci, Gurdip Bhalay and Mark Bradley*

The total solid phase synthesis of an analogue of the B ring of nisin was achieved, in a biomimetic fashion, via the solid phase diastereoselective cyclisation of a dehydrothiol-containing peptide.

Regioselective oxidation of N-alkylpyrrolidines to pyrrolidin-5-ones by RuCl₃/NaIO₄

pp 1403-1406

Nagendra K. Sharma and Krishna N. Ganesh*

RuCl₃/NaIO₄ under EtOAc/H₂O biphasic conditions, selectively oxidizes the N α -endo-methylene group of pyrrolidine derivatives, without affecting the exo-methylene group adjacent to the N-heteroatom.



Synthesis of benzo[4,5]imidazo[2,1-a]phthalazines

pp 1407-1408

Kirill M. Shubin,* Viktor A. Kuznetsov and Vladimir A. Galishev

$$R^{1}$$
 O
 $O_{2}N$
 R^{2}
 R^{2}

Activation of silylphosphines by diethyl azodicarboxylate: novel silylation of alcohols

pp 1409-1411

Minoru Hayashi,* Yutaka Matsuura and Yutaka Watanabe*

R-OH +
$$Ph_2P-Si$$
 $EtO_2C-N=N-CO_2Et$ PPTS, r.t., 5 min R-OS
$$Si = SiMe_2{}^tBu, SiEt_3, Si^tPr_3$$



5-Hydroxy-2*H*-pyrrol-2-ones and not 2-aminofurans are the cycloaddition products between alkyl isocyanides and benzyliden-1,3-diketones

pp 1413-1416

Monica Quai,* Sara Frattini, Ugo Vendrame, Maurizio Mondoni, Simona Dossena and Enzo Cereda

$$+ R-N \stackrel{\downarrow}{=} C^{-} \longrightarrow 0$$

Iodination of organotrifluoroborates: synthesis of vinyl and alkynyl iodides

pp 1417-1419

George W. Kabalka* and Arjun Reddy Mereddy

$$RBF_{3}K \qquad \frac{\text{Na I}}{\text{Chloramine-T}} \qquad R \longrightarrow I$$

$$1 \qquad \qquad R.T. \qquad \qquad 2$$

$$R = \text{Vinyl or alkynyl}$$

Vinyl- and alkynyltrifluoroborates are rapidly converted to vinyl and alkynyl iodides under mild conditions using sodium iodide in the presence of chloramine-T.

Efficient synthesis of novel cytotoxic cis-fused α-methylene γ-lactones from 7,14-dihydroxy-entkaurenes by transformation under Mitsunobu reaction conditions

pp 1421-1425

Yutaka Aoyagi, Ming-Yu Gui, Yong-Ri Jin, Xu-Wen Li, Takeshi Noguchi, Haruhiko Fukaya, Tomoyo Hasuda and Koichi Takeya*

Efficient nucleophilic substitution reactions of highly functionalized allyl halides in ionic liquid media pp 1427-1431 S. R. S. Saibabu Kotti, Xin Xu, Guigen Li* and Allan D. Headley*

$$\frac{[\text{Bmim}][\text{BF}_4], 50 \, ^{\circ}\text{C}}{\text{NaNu or KNu, 2 h}}$$

$$Nu = N_3, \text{ AcO and PhSO}_2$$

$$(75\% - \text{quant.})$$

Efficient synthesis of core 2 class glycosyl amino acids by one-pot glycosylation approach Hiroshi Tanaka, Masaatsu Adachi and Takashi Takahashi*

pp 1433-1436



The influence of the ring size of thiolactams in the Eschenmoser coupling reaction in presence of DBU. Formation of bicyclic thiazolidinones or thioimines

pp 1437-1440

Dennis Russowsky* and Brenno Amaro da Silveira Neto



A robust palladium-catalyzed cyanation procedure: beneficial effect of zinc acetate Ramakrishnan Chidambaram*

pp 1441-1444

A more robust procedure to convert aryl bromides to aryl cyanides that uses zinc dust and zinc acetate has been described.

Hexafluoroacetone as a protecting and activating reagent: 5,5-difluoro- and *trans*-5-fluoropipecolic pp 1445–1447 acids from glutamic acid

Alexander S. Golubev, Hartmut Schedel, Gabor Radics, Marco Fioroni, Sven Thust and Klaus Burger*

$$HO_2C$$
 HO_2C
 HO_2

Starting from hexafluoroacetone-protected (S)-glutamic acid, trans-5-fluoropipecolic and 5,5-difluoropipecolic acid have been synthesized. The piperidine ring was constructed by an intramolecular metal carbenoid NH insertion.

A practical, solvent free, one-pot synthesis of C_2 -symmetrical secondary amines

pp 1449-1451

Alexandre Alexakis,* Ségolène Gille, Fabrice Prian, Stéphane Rosset and Klaus Ditrich

A novel one-pot reductive amination of ketones using the combination $Ti(O^{i}Pr)_{4}/H_{2}/Pd$ —C is reported. This practical procedure does not require any solvent and affords C_{2} -symmetrical secondary amines in high yields and excellent diastereoselectivities.

An efficient approach for monosulfide bridge formation in solid-phase peptide synthesis

pp 1453-1456

Pietro Campiglia, Isabel Gomez-Monterrey, Luigi Longobardo, Teresa Lama, Ettore Novellino and Paolo Grieco*

A novel approach for the synthesis of cyclic peptides containing unnatural thioether side-chain bridges, is reported.

Tandem Diels-Alder reaction/radical cyclizations for the rapid construction of bridged ring systems pp 1457–1459 George A. Kraus* and Junwon Kim

Bridged tricyclic ring systems can be prepared in a one-pot reaction using a tandem Diels–Alder reaction/radical cyclization strategy. The regiochemistry of the radical addition is unexpected.

First synthesis of 3-O-methyl-scyllo-inosamine, a natural product which favors the Rhizobium-Leguminosae symbiosis

pp 1461-1463

Alain Krief,* Willy Dumont, Denis Billen, Jean-Jacques Letesson, Pascal Lestrate, Peter J. Murphy and Damien Lacroix

The first enantioselective synthesis of 4-acetyl-3(R)- and 3(S)-(hydroxymethyl)-3,4-dihydro-2H-pyrido[3,2-b]oxazine

pp 1465-1468

N. Henry, G. Guillaumet and M. D. Pujol*

Ruthenium-catalyzed addition of sulfenamides to alkynes leading to selective synthesis of polyfunctional pp 1469–1471 alkenes

Teruyuki Kondo, Atsushi Baba, Yumiko Nishi and Take-aki Mitsudo*

Novel applications of 2-cyanoethylanilines in the synthesis of conjugated primary and secondary anilines pp 1473–1475 Yi Liao* and Bruce H. Robinson

Synthesis of pyranoid δ -sugar amino acids and their oligomers from per-benzylated β -C-vinyl glucoside pp 1477–1479 François Durrat, Juan Xie* and Jean-Marc Valéry

Synthesis of orthogonally protected 3,8-diazabicyclo[3.2.1]octane-2-carboxylic acid—a versatile building block for the synthesis of cocaine analogues

pp 1481-1483

Stefan Pichlmair,* Kurt Mereiter and Ulrich Jordis*

Vicinal alkylation—carboxymethylation of electron-poor alkenes by radical-chain reactions with *O*-alkyl *O*-silyl ketene acetals and their [3+2] annulation by reaction with *O*-cyclopropylcarbinyl *O*-silyl ketene acetals

pp 1485-1488

Yudong Cai and Brian P. Roberts*

Conditions: i Dilauroyl peroxide (0.10 equiv.) initiator, refluxing benzene; ii TBAF, Mel

Two-step synthesis of β -alkyl chalcones and their use in the synthesis of 3,5-diaryl-5-alkyl-4,5-dihydropyrazoles

pp 1489-1493

Christopher D. Cox,* Michael J. Breslin and Brenda J. Mariano

$Samarium (II) \hbox{-induced ring-expansion reaction of 1,2-cyclobutane dicarboxy lates to produce cyclopentanones \\$

pp 1495-1498

Ikuo Shinohara and Hiroto Nagaoka*

$$(\bigcirc)_{n} \qquad \qquad \\ CO_{2}Me \qquad \qquad \\ \\ CO_{$$

Indium(III)-catalysed aryl sulfonylation reactions

pp 1499-1501

Vincenzo Garzya,* Ian T. Forbes, Stephanie Lauru and Paolo Maragni

A convenient synthesis of dihydro- and tetrahydro-1,3-thiazine derivatives from β -aryl- β -amino acids Nicolas Leflemme, Patrick Dallemagne* and Sylvain Rault

pp 1503-1505

Synthesis, optical, and electrochemical properties of conjugated oligomers derived from 4-bromo-4'-(n-butyl)-2,2'-biphenyl

pp 1507-1510

Xue-Ming Liu, Jingmei Xu and Chaobin He*

Br +
$$n$$
-BuLi $\frac{\mathsf{THF}}{\mathsf{0}\,^{\circ}\mathsf{C}\,\mathsf{to}\,\mathsf{r.t.}}$ n -Bu Br $\frac{\mathsf{R-X}}{\mathsf{i}\,\mathsf{or}\,\mathsf{ii}}$ n -Bu MgBr R-X S MgBr H S 2 B(OH)2 H S 3 B(OH)2 MgBr H₃C(H₂C)₅ (CH₂)₅CH₃

i. [NiCl₂(dppp)], THF, refluxing. ii. [Pd(PPh₃)₄], toluene/2M K_2CO_3 (3:2, V/V), refluxing



A solid-phase version of the Nozaki-Hiyama allylation of aldehydes with supported allylic bromides Klaus Breitenstein, Amadeu Llebaria and Antonio Delgado*

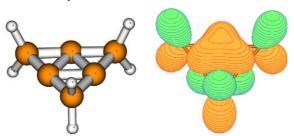
pp 1511-1513

A solid-phase version of the Nozaki–Hiyama allylation of supported allylic bromides with aldehydes is described. α -Methylene γ -butyrolactones 7 can be obtained by cyclization cleavage of the supported, intermediate homoallylic alcohols 6, which can also be isolated by proper choice of reaction conditions.

A system with three contiguous planar tetracoordinate carbons is viable: a computational study on a $C_6H_6^{2^+}$ isomer

pp 1515-1517

U. Deva Priyakumar and G. Narahari Sastry*



A novel structure, C₆H₆²⁺, with three contiguous planar tetracoordinate carbon atoms is proposed as a viable candidate.

[6+2] Cycloaddition of N-phenyltriazolinedione with cycloheptatriene derivatives mediated and stereodirected by a chiral 3-oxy substituent

pp 1519-1521

Takashi Sugimura,* Chun Young Im and Tadashi Okuyama

The PTAD addition to 7,7-dimethylcycloheptatriene resulted in a stereocontrolled [6+2] cycloaddition as a result of the rearrangement of the [4+2] adduct.

Synthesis of \mathbf{p} -rubranitrose by using a novel method for constructing functionalized branched-chain structures

pp 1523-1525

Ken-ichi Sato,* Daisuke Miyama and Shoji Akai

Synthesis of the highly potent prostanoid FP receptor agonist, AFP-168: a novel 15-deoxy-15,15-difluoroprostaglandin $F_{2\alpha}$ derivative

pp 1527-1529

Yasushi Matsumura,* Nobuaki Mori, Takashi Nakano, Hideshi Sasakura, Takeshi Matsugi, Hideaki Hara and Yoshitomi Morizawa

A novel 15-deoxy-15,15-difluoro-prostaglandin(PG) $F_{2\alpha}$ derivative **6** (AFP-168) has been synthesized from the Corey aldehyde in 6 steps.

Desymmetrizations of meso oxabicyclic compounds by asymmetric C-H insertion

pp 1531-1534

Pauline Chiu,* Xiaomei Zhang and Rebecca Y. Y. Ko

Using chiral rhodium catalysts, moderate enantioselectivities have been achieved in the desymmetrizations of *meso* oxabicyclo[3.2.1]diazoketones via intramolecular C–H insertion to give fused cyclopentanones.

Synthesis of thiiranes from oxiranes in water using polymeric cosolvents

pp 1535-1537

Bahman Tamami* and Majid Kolahdoozan

+ NH₄SCN
$$\frac{\text{Polymeric cosolvent}}{\text{H}_2\text{O}, \text{NaOH}}$$

$$\text{rt- 45°C}$$
Polymeric cosolvent: $-\text{H}_2\text{C}-\text{CH--}$

$$\text{NH}_2$$

$$\text{Or } -\text{H}_2\text{C}-\text{CH--}$$

$$\text{NH}_2$$

$$\text{Or } -\text{H}_2\text{C}-\text{CH--}$$

$$\text{NH}_2$$

Probing disaccharide selectivity with modular fluorescent sensors

pp 1539-1542

Susumu Arimori, Marcus D. Phillips and Tony D. James*

Bismuth triflate catalyzed condensation of δ -hydroxy- α , β -unsaturated aldehydes with aryl amines

pp 1543-1546

J. S. Yadav,* B. V. S. Reddy, G. Parimala and A. Krishnam Raju

$$R^2$$
 + R' OR' CHO OR' CHO OR' R^2 R^3 R^4 R^4

One-pot easy conversion of Baylis-Hillman adducts into carbamates of unsaturated \(\beta \)-amino acids

pp 1547-1550

Manouchehr Mamaghani* and Abed Badrian

R'=H, 4-Me, 4-MeO, 3-NO2, 4-NO2, 4-CI, 2-CI

An easy, one-pot convenient transformation of Baylis–Hillman adducts into carbamates of unsaturated β-amino acids, for example, 7a-g and 8a-g via reaction with the Burgess reagent is described.

Synthesis of homopropargyl alcohols via sonochemical Barbier-type reaction

pp 1551-1553

Adam Shih-Yuan Lee,* Shu-Fang Chu, Yu-Ting Chang and Shu-Huei Wang

R-CHO + = CH₂Br
$$\frac{5.0 \text{ Zn, } 1.0 \text{ ICH}_2\text{CH}_2\text{I, THF, })), 2.5\text{h;}}{2\text{M HCI (2mL/eq.)}}$$

The homopropargyl alcohols were obtained as the only product when aldehydes were reacted with 3-bromo-1-propyne under the sonochemical Barbier-type reaction condition.

$Efficient\ asymmetric\ transfer\ hydrogenation\ of\ activated\ olefins\ catalyzed\ by\ ruthenium\ amido\ complexes$

pp 1555-1558

Ying-Chun Chen, Dong Xue, Jin-Gen Deng,* Xin Cui, Jin Zhu and Yao-Zhong Jiang

NC_CN NC_CN Ph. Ph. Ph. Ph. NC_CN
$$A = CH_2$$
, O,S up to 88.5% ee

Synthesis of alkaloids from aminol derivatives by $\beta\mbox{-fragmentation}$ of primary alkoxyl radicals

pp 1559-1563

Alicia Boto,* Rosendo Hernández,* Adriana Montoya and Ernesto Suárez

$$\begin{array}{c} \text{HO} \quad \text{CO}_2\text{CMe}_3 \\ \text{N} \quad \text{One-pot} \\ \beta\text{-fragmentation-oxidation-alkylation} \\ \hline \\ \text{O} \quad \text{O} \\ \end{array} \begin{array}{c} \text{CO}_2\text{CMe}_3 \\ \text{Ph} \quad \text{N} \\ \text{O} \quad \text{O} \\ \end{array}$$

Ring opening of 2,3-epoxy phenyl ketones upon reaction with nitric oxide

pp 1565-1566

Zhongquan Liu, Rui Li, Desuo Yang and Longmin Wu*

2,3-Epoxy phenyl ketones are opened regioselectively at C-3 by nitric oxide, affording erythro- α -hydroxyl nitrates in a highly syn-selective manner.

An expedient synthesis of diversified pyrrolizines and indolizines

pp 1567-1570

George Bashiardes,* Imad Safir, Francis Barbot and Joelle Laduranty

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*Corresponding author

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