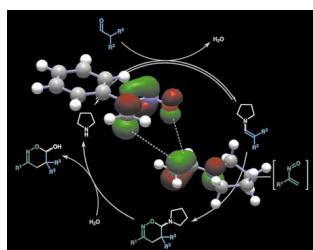


# Organic & Biomolecular Chemistry

INDEXED IN MEDLINE

Incorporating Acta Chemica Scandinavica

**Cover**

See Tobias C. Wabnitz, Steen Saaby and Karl Anker Jørgensen, page 828. The cover shows the catalytic cycle of dihydro-oxazine formation from nitroso alkenes and aldehydes in the presence of pyrrolidine. The key stage in this organocatalysed process is a hetero-Diels-Alder reaction with inverse-electron demand and the proposed frontier molecular orbital interactions of this step are indicated (molecular modelling by Mette Alstrup Lie).



Chemical biology articles published in this journal also appear in the *Chemical Biology Virtual Journal*: [www.rsc.org/chembiol](http://www.rsc.org/chembiol)

## contents

## COMMUNICATIONS

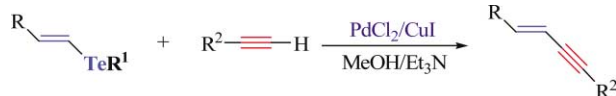


803 805

### Stereoselective preparation of conjugated *E*-enynes from *E*-vinylic tellurides and terminal alkynes *via* Sonogashira cross-coupling

Gilson Zeni, Diego Alves, Jesus M. Pena, Antonio L. Braga, Helio A. Stefani and Cristina W. Nogueira

*E*-Vinylic tellurides undergo a direct cross-coupling reaction with alkynes in the presence of palladium to give *E*-enynes in good yields.

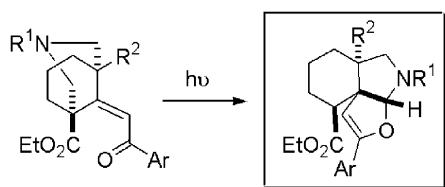


806 807

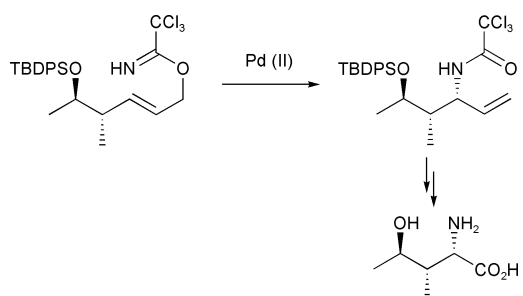
### Unprecedented photochemical induced cascading rearrangement of the 3-azabicyclo[3.3.1]nonane skeleton

Craig M. Williams, Ralf Heim, Douglas J. Brecknell and Paul V. Bernhardt

Certain 3-azabicyclo[3.3.1]nonane derivatives undergo unprecedented stereospecific skeletal cleavage when subjected to light affording a novel heterotricyclic skeleton.



808 809

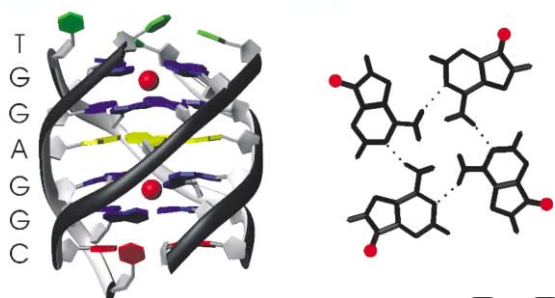


**The first enantioselective synthesis of the amino acid, (2*S*,3*S*,4*R*)- $\gamma$ -hydroxyisoleucine using a palladium(II) catalysed 3,3-sigmatropic rearrangement**

Andrew G. Jamieson, Andrew Sutherland and Christine L. Willis

A palladium catalysed Overman rearrangement which uses allylic 1,3-strain to control the stereoselectivity is the key step in the synthesis of (2*S*,3*S*,4*R*)- $\gamma$ -hydroxyisoleucine.

810 812

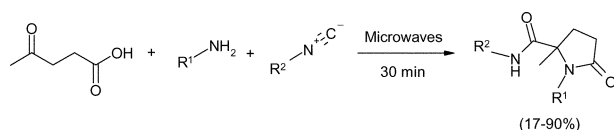


**Structure and  $K^+$  ion-dependent stability of a parallel-stranded DNA quadruplex containing a core A-tetrad**

Mark S. Searle, Huw E. L. Williams, Cathal T. Gallagher, Richard J. Grant and Malcolm F. G. Stevens

NMR studies and molecular dynamics simulations are described on intermolecular quadruplex structures containing the purine-rich GGAGG motif which assemble to form stable parallel-stranded quadruplexes containing A- and G-tetrads.

813 815



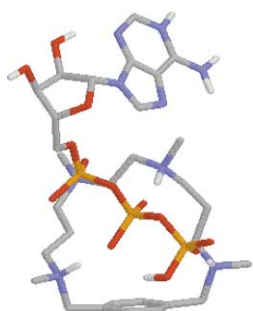
**Use of a Design of Experiments approach for the optimisation of a microwave assisted Ugi reaction**

Heather Tye and Mark Whittaker

The utility of a Design of Experiments approach for the rapid and efficient optimisation of a microwave assisted Ugi 3cc reaction of levulinic acid is demonstrated.

## ARTICLES

816 820

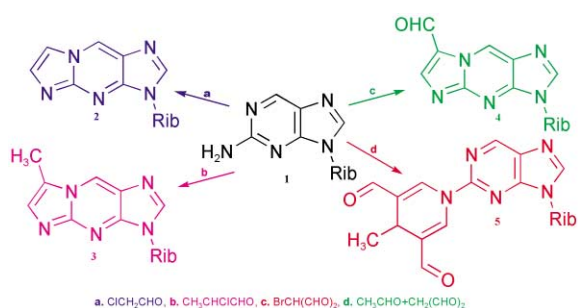


**Studies on the interaction of phosphate anions with *N*-functionalised polyaza[*n*]paracyclophanes: the role of *N*-methylation**

M Teresa Albelda, Juan C. Frías, Enrique García-España and Santiago V. Luis

*N*-methylation of polyazacyclophanes significantly increase their binding constants with ATP.

821 827



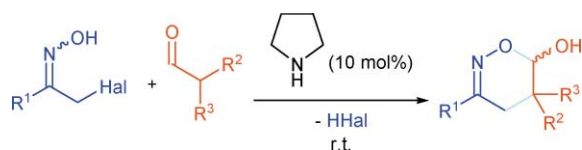
a.  $\text{ClCH}_2\text{CHO}$ , b.  $\text{CH}_3\text{CHClCHO}$ , c.  $\text{BrCH}(\text{CHO})_2$ , d.  $\text{CH}_3\text{CHO} + \text{CH}_2(\text{CHO})_2$

**New nucleoside analogs from 2-amino-9-( $\beta$ -D-ribofuranosyl)-purine**

Piritta Virta, Toni Holmström, Mattias U. Roslund, Peter Mattjus, Leif Kronberg and Rainer Sjöholm

In the current work, we report on the synthesis of four novel derivatives of 2-amino-9-( $\beta$ -D-ribofuranosyl)purine (**1**).

828 834

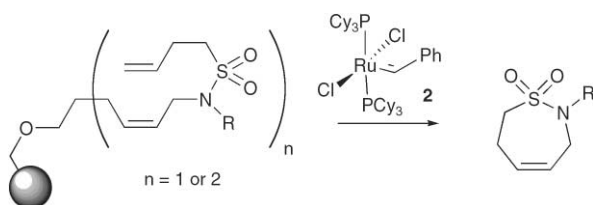


### The first catalytic inverse-electron demand hetero-Diels–Alder reaction of nitroso alkenes using pyrrolidine as an organocatalyst

Tobias C. Wabnitz, Steen Saaby and Karl Anker Jørgensen

5,6-Dihydro-4*H*-oxazines are directly accessible from aldehydes and halooximes *via* the first organocatalytic inverse-electron demand hetero-Diels–Alder reaction of transient nitroso alkenes.

835 844

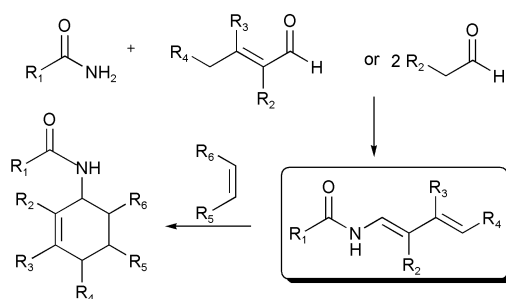


### Ring-closing metathesis: development of a cyclisation–cleavage strategy for the solid-phase synthesis of cyclic sulfonamides

Jean-Dominique Moriggi, Lynda J. Brown, José L. Castro and Richard C. D. Brown

A series of cyclic sulfonamides have been prepared using a solid-phase RCM cyclisation–cleavage strategy. The use of a novel double armed-linkage allowed efficient release of cyclic sulfonamides using as little as 1 mol% of the Grubbs' catalyst **2**.

845 851

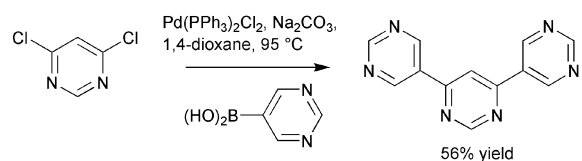


### From a spin-off to the advantageous use in Diels–Alder reactions: a combined synthetic, spectroscopic and computational approach to *N*-(dienyl)acylamines

Dirk Gördes, Axel Jacobi von Wangelin, Stefan Klaus, Helfried Neumann, Dirk Strübing, Sandra Hübner, Haijun Jiao, Wolfgang Baumann and Matthias Beller

For the first time, the synthesis of substituted 1-*N*-acylamino-1,3-butadienes has been accomplished directly in moderate yields upon telomerization of two molecules of aldehyde with one molecule of carboxamide.

852 857

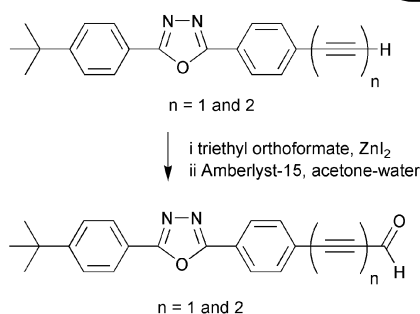


### 5-Pyrimidylboronic acid and 2-methoxy-5-pyrimidylboronic acid: new heteroarylpyrimidine derivatives *via* Suzuki cross-coupling reactions

Nezire Saygili, Andrei S. Batsanov and Martin R. Bryce

Efficient syntheses of the title boronic acids are reported, along with the crystal structure of 5-pyrimidylboronic acid, and coupling reactions to yield heteroarylpyrimidines.

858 862

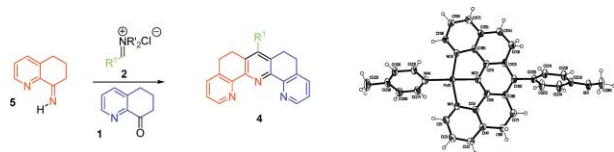


### Functionalisation reactions of 2,5-diphenyl-1,3,4-oxadiazoles bearing a terminal ethynyl or butadiynyl substituent: X-ray crystal structures of the products

David Kreher, Andrei S. Batsanov, Changsheng Wang and Martin R. Bryce

Efficient functionalisation reactions of the ethynyl and butadiynyl units of **1** and **2** are described, *e.g.* conversion to alkynyl aldehydes, and a subsequent Wittig product.

863 868

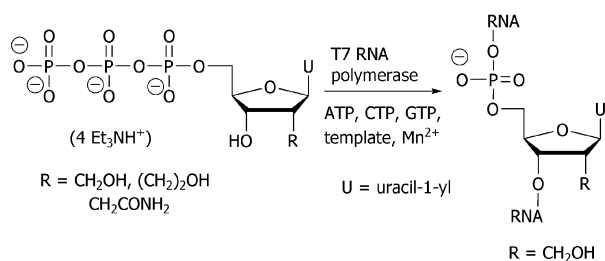


### Selective synthesis of U-shaped terpyridines. Versatile ligands for the preparation of platinum complexes

Dirk Sielemann, Andreas Winter, Ulrich Flörke and Nikolaus Risch

The development of a simple and highly selective one-pot method for the preparation of substituted U-shaped terpyridines is described. The terpyridine derivatives are used for the preparation of several platinum(II) complexes **14**. Complex **14e** is characterized by single-crystal X-ray analysis.

869 875

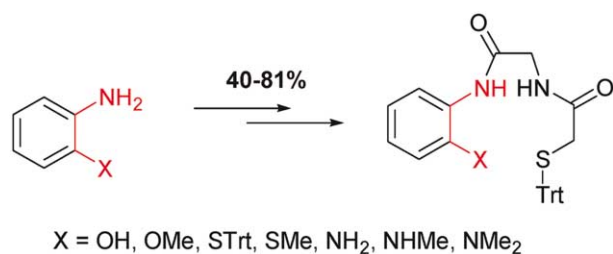


### Synthesis and transcription studies on 5'-triphosphates derived from 2'-C-branched-uridines: 2'-homouridine-5'-triphosphate is a substrate for T7 RNA polymerase

John B. J. Pavey, Anthony J. Lawrence, Ian A. O'Neil, Stefan Vortler and Richard Cosstick

The first report of a 2'-deoxy-2'- $\alpha$ -C-branched nucleoside 5'-triphosphate acting as a substrate for T7 RNA polymerase.

876 883

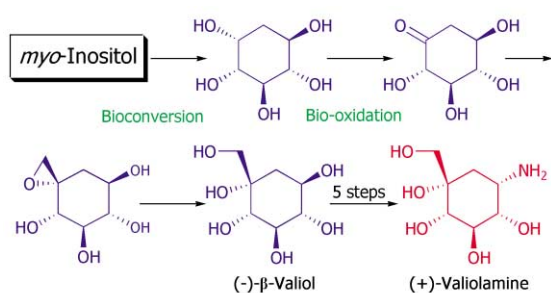


### Design and synthesis of a novel family of semi-rigid ligands: versatile compounds for the preparation of <sup>99m</sup>Tc radiopharmaceuticals

Julien Le Gal, Laure Latapie, Marie Gressier, Yvon Coulais, Michèle Dartiguenave and Eric Benoist

A novel series of semi-rigid tetradentate derivatives was prepared, characterised and used as chelate ligands for a <sup>99m</sup>TcO core.

884 889

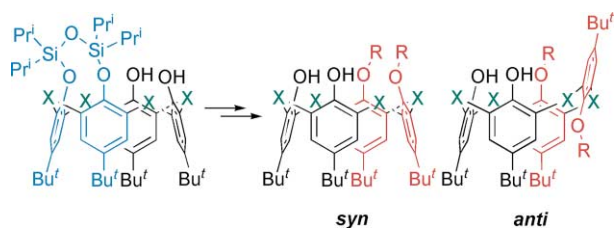


### Convenient synthesis of (+)-valiolamine and (-)-1-epi-valiolamine from (-)-vibo-quercitol

Seiichiro Ogawa, Yo Ohishi, Miwako Asada, Akihiro Tomoda, Atsushi Takahashi, Yoriko Ooki, Midori Mori, Masayoshi Itoh and Takashi Korenaga

(+)-Valiolamine and its 1-epimer were practically synthesized from 1-deoxy-L-*myo*-inositol, readily obtained by bioconversion of *myo*-inositol.

890 898

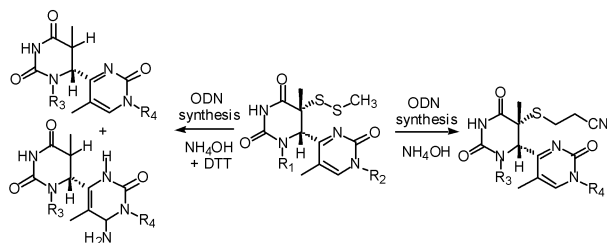


### Stereoselective dialkylation of the proximal hydroxy groups of calix- and thiocalix[4]arenes

Fumitaka Narumi, Tetsutaro Hattori, Naoya Morohashi, Nobuji Matsumura, Waka Yamabuki, Hiroshi Kameyama and Sotaro Miyano

An efficient *syn/anti*-selective proximal dialkylation has been achieved by initial *O,O'*-capping with a disiloxane-1,3-diyl bridge, subsequent dialkylation, followed by desilylation.

899 907

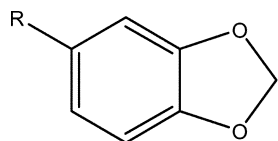


### Studies on the chemical synthesis of oligodeoxynucleotides containing the s<sup>5</sup>T(6-4)T photoproduct: side reactions derived from the methylsulfenyl thiol protection elucidated by MALDI mass spectrometry

V. Guérineau, S. K. A. Matus, F. Halgand, O. Laprèvote and P. Clivio

Incorporation of the TpT (6-4) photoproduct-C5 thio analogue phosphoramidite, *S*-protected with the CH<sub>3</sub>S group, gives rise to (2-cyanoethylthio)<sup>5</sup>T(6-4)T photoproduct-containing ODNs under standard deprotection conditions. In the presence of thiol, ODNs containing the h<sup>5</sup>T(6-4)T or its dihydropyrimidone derivative are obtained.

908 914



$$\Delta_{\text{C}}U_{\text{m}}^{\circ}, \Delta_{\text{Cr}}^{\text{g}}H_{\text{m}}^{\circ}, \Delta_{\text{f}}H_{\text{m}}^{\circ}$$

### Thermodynamic study of sesamol, piperonyl alcohol, piperonylic acid and homopiperonylic acid: a combined experimental and theoretical investigation

M. Agostinha R. Matos, Manuel J. S. Monte, Clara C. S. Sousa, Ana R. R. P. Almeida and Victor M. F. Morais

Thermodynamic study of sesamol (R = OH), piperonyl alcohol (R = CH<sub>2</sub>OH), piperonylic acid (R = COOH) and homopiperonylic acid (R = CH<sub>2</sub>COOH): a combined experimental and theoretical investigation.

915 921

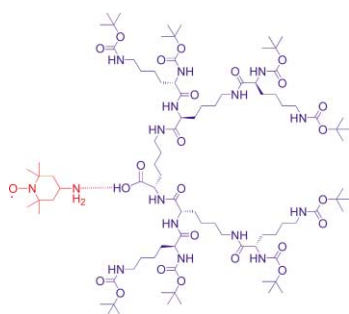
Arg-Gln-Ile-Lys-Ile-Trp-Phe-Gln-Asn-Arg-Arg-Met-Lys-Trp-Lys-Lys  
*Antp* helix 3 homeodomain

### Relative DNA binding affinity of helix 3 homeodomain analogues, major groove binders, can be rapidly screened by displacement of prebound ethidium bromide. A comparative study

Yong-Ho Shim, Paola B. Arimondo, Alain Laigle, Anna Garbesi and Solange Lavielle

Relative binding affinity of major groove dsDNA ligands can be estimated by displacement of prebound ethidium bromide.

922 926



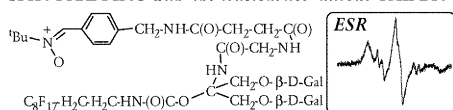
### NMR and ESR investigations of the interaction between a carboxylic acid and an amine at the focal point of L-lysine based dendritic branches

Graham M. Dykes, David K. Smith and Agneta Caragheorghopol

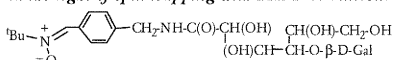
Using host-guest chemistry at the focal point of a dendritic structure is an effective way to control and modify the solution phase properties and mobility of active species such as radicals.

927 934

SPIN TRAPPING with the tensioactive nitron TAIPBN



in the light of spin trapping with LAMPBN nitron

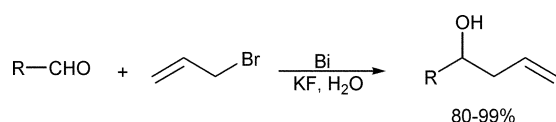


### ESR study of spin-trapping with two glycosylated analogues of PBN able to target cell membrane lectins

Florence Chalier, Olivier Ouari and Paul Tordo

The spin trapping behaviour of the two galactosylated nitrones LAMPBN and TAIPBN, were widely investigated on a variety of free radicals in aqueous media.

935 938



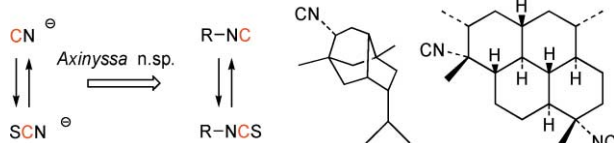
### A convenient procedure for bismuth-mediated Barbier-type allylation of aldehydes in water containing fluoride ions

Keith Smith, Sandra Lock, Gamal A. El-Hiti, Makoto Wada and Norikazu Miyoshi

Excellent yields of homoallylic alcohols were obtained from bismuth-mediated allylation reactions of aldehydes in water.



939 948



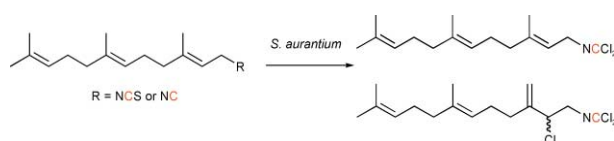
### Biosynthetic pathways to isocyanides and isothiocyanates; precursor incorporation studies on terpene metabolites in the tropical marine sponges *Amphimedon terpenensis* and *Axinyssa* n.sp.

Jamie S. Simpson and Mary J. Garson

Biosynthetic pathways leading to marine isocyanides and isothiocyanates are defined by precursor incorporation experiments in the marine sponges *A. terpenensis* and *Axinyssa* n.sp.



949 956



### Biosynthetic pathways to dichloroimines; precursor incorporation studies on terpene metabolites in the tropical marine sponge *Stylotella aurantium*

Jamie S. Simpson, Andreas Brust and Mary J. Garson

The biosynthetic pathways leading to dichloroimines are defined by precursor incorporation experiments in the marine sponge *S. aurantium*.

xi

Dates, venues and contact details of forthcoming events.