IN THIS ISSUE...

ISSN 1477-0520 CODEN OBCRAK 3(9) 1577-1804 (2005)



Cover

See M. Nishihara, F. Perret, T. Takeuchi, S. Futaki, A. N. Lazar, A. W. Coleman, N. Sakai and S. Matile, pp. 1659–1669 Their spectacular synergism suggests that counterions may account for many 'mysterious' functions of oligoarginines in biomembranes in a general manner compelling reasons to use rather than to ignore them!

Image reproduced by permission of Stefan Matile from *Org. Biomol. Chem.*, 2005, **3**, 1659.



Inside Cover

See M. de Loos, A. Friggeri, J. van Esch, R. M. Kellogg and B. L. Feringa, pp. 1631–1639 Cyclohexane bis-urea compounds can be used for the gelation of water and aqueous solutions. These hydrogels can be tailored for a specific function or application.

Image reproduced by permission of Jan van Esch from *Org. Biomol. Chem.*, 2005, **3**, 1631.

CHEMICAL SCIENCE

C33

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Chemical Science

May 2005/Volume 2/Issue 5 www.rsc.org/chemicalscience

ESSAY

1591

Genetic alphabetic order: what came before A?

Jay S. Siegel* and Yitzhak Tor

Could a single heterocycle be responsible for the precursor to our present genetic alphabet? A "prebiotic" postulate is discussed.



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1593

Lectins: tools for the molecular understanding of the glycocode

Moira Ambrosi, Neil R. Cameron* and Benjamin G. Davis*

An overview of lectins, their interactions with carbohydrates and possible therapeutic applications is presented.

Concanavalin A bound to trimannoside (PDB ID: 1CVN, J. H. Naismith and R. A. Field)



CO₂t-Bu

94%, 95% ee

EtO₂

Boo

COMMUNICATIONS

1609

Memory of chirality in intramolecular conjugate addition of enolates: a novel access to nitrogen heterocycles with contiguous quaternary and tertiary stereocenters

Takeo Kawabata,* Swapan Majumdar, Kazunori Tsubaki and Daiki Monguchi

Nitrogen heterocycles with contiguous quaternary and tertiary stereocenters have been prepared by intramolecular conjugate addition of chiral enolates generated from α -amino acid derivatives *via* memory of chirality.

1612

Alkylation of natural endoperoxide G3-factor. Synthesis and antimalarial activity studies

Fadia Najjar, Liliane Gorrichon, Michel Baltas, Christiane André-Barrès* and Henri Vial

Alkylation of the peroxyhemiketal function is described and all synthesised endoperoxides show good antimalarial activity.

1615

Visual sensing of Ca²⁺ ion *via* photoreaction of fluorenyl ester-armed cyclen

Tomoko N. Player, Satoshi Shinoda and Hiroshi Tsukube*

The photoreaction of fluorenyl ester-armed cyclen offered naked-eye detection of Ca^{2+} ion in aqueous samples.



R = $(CH_2)_n CH_3$, Bn or $CH_2 C_5 H_5 N$ n = 0, 2, 3, 7



CO₂t-Bu

KHMDS

1617

Solvent-free mechanochemical and one-pot reductive benzylizations of malononitrile and 4-methylaniline using Hantzsch 1,4-dihydropyridine as the reductant

Ze Zhang, Jie Gao, Jing-Jing Xia and Guan-Wu Wang*

Under mechanical milling conditions, direct reductive benzylizations of malononitrile and 4-methylaniline by aromatic aldehydes were achieved using a Hantzsch 1,4-dihydropyridine as the reductant.





OMe

DABCO catalyzed addition of selenosulfonates to

In the presence of DABCO, the addition of various selenosulfonates to activated olefins proceeded smoothly to give the adducts in good yields

A new method for the synthesis of acyltitanium complexes and their application to copper-mediated acylmetallation of carbon-carbon multiple bonds in aqueous media

Zhenfu Han, Takuma Fujioka, Shin-ichi Usugi, Hideki Yorimitsu, Hiroshi Shinokubo and Koichiro Oshima*

Treatment of alkynes with an acyltitanium reagent in the presence of triethylamine and copper in aqueous THF yielded the corresponding 1,4-diketones.

ARTICLES

1624

1631

HC=CCH2OM

Cu powder, Et₃N

THF / H₂O (5 mL/3 mL)

The effect of the anion on the physical properties of trihalide-based N,N-dialkylimidazolium ionic liquids

Alessandro Bagno,* Craig Butts, Cinzia Chiappe,* Fabio D'Amico, Jason C. D. Lord, Daniela Pieraccini and Federico Rastrelli

Trihalide-based ionic liquids have a lower melting point and viscosity and a higher density and hydrophobicity when compared to other common imdazolium ionic liquids, including chlorides, bromides or iodides.

Cyclohexane bis-urea compounds for the gelation of water and aqueous solutions

Maaike de Loos, Arianna Friggeri, Jan van Esch,* Richard M. Kellogg and Ben L. Feringa*

End-group modification of a well-known organogelator yields easily accessible and efficient gelators of organic solvents and water, displaying unusual stereochemical aspects.



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 $R = OH, CH_2OH \text{ or } CH_2NH_2$

The reaction of S-nitroso-N-acetyl-D,L-penicillamine (SNAP) with the angiotensin converting enzyme inhibitor, captopril-mechanism of transnitrosation

Danielle V. Aquart and Tara P. Dasgupta*

S-Nitroso-N-acetyl-D,L-penicillamine and captopril react to yield two distinct stages, one being transnitrosation, the other releasing the nitroxyl ion in solution.

ARTICLES

1647

Correlation of bilayer membrane cation transport and biological activity in alkyl-substituted lariat ethers

W. Matthew Leevy, Michelle E. Weber, Michael R. Gokel, George B. Hughes-Strange, David D. Daranciang, Riccardo Ferdani and George W. Gokel*

Alkyl-substituted lariat ethers have been prepared and their effects on liposome cation transport and bacterial toxicity examined.

1653

Synthesis and triplex forming properties of pyrrolidino pseudoisocytidine containing oligodeoxynucleotides

Alain Mayer, Adrian Häberli and Christian J. Leumann*

Triplex forming oligodeoxynucleotides containing pyrrolidino pseudoisocytidine units were found to significantly increase the thermal stability of DNA triplexes.



Arginine magic with new counterions up the sleeve

Masamichi Nishihara, Florent Perret, Toshihide Takeuchi, Shiroh Futaki, Adina N. Lazar, Anthony W. Coleman, Naomi Sakai* and Stefan Matile*

The synergistic anion carrier activity of polyarginine-counteranion complexes is investigated with emphasis on counteranion recognition by polyarginine on the one hand and lipid bilayer membranes on the other.

1670

First enantioselective synthesis of (-)- and (+)-virgatusin, tetra-substituted tetrahydrofuran lignan

Satoshi Yamauchi,* Momotoshi Okazaki, Koichi Akiyama, Takuya Sugahara, Taro Kishida and Takehiro Kashiwagi

First highly enantioselective synthesis of (-)- and (+)-virgatusin, 2,5-diaryl-3,4-bis(methoxymethyl)tetrahydrofuran lignan, was accomplished.

1676

Proton di-ionizable *p-tert*-butylcalix[4]arene-crown-6 compounds in cone, partial-cone and 1,3-alternate conformations: synthesis and alkaline earth metal cation extraction

Hui Zhou, Kazimierz Surowiec, David W. Purkiss and Richard A. Bartsch*

To probe the influence of the positioning of ionizable groups relative to the crown cavity, di-ionizable calix[4]arene-crown-6 ligands with carboxylic acid and N-(X)sulfonyl carboxamide groups were prepared and evaluated.





Phosphate recognition

Base

🗁 Base

recognition

Į



1.3-Alternate





1685



1690



1694



1702



1708



β-Selective synthesis of 2'-deoxy-5,6-dihydro-4-thiouridine, a precursor of the unstable nucleoside product of ionising radiation damage 2'-deoxy-5,6-dihydrocytidine

Frédéric Peyrane and Pascale Clivio*

The diastereoselective synthesis of the disilyl derivative of 2'-deoxy-5,6-dihydro-4-thiouridine (5) from its 5,6-dihydrouracil counterpart (1), and its subsequent amination, is reported.

Biosynthesis of the allene (–)-marasin in *Marasmius ramealis*

David G. Davies and Philip Hodge*

Incorporation of [14 C]-labelled **8b** into (–)-marasin indicates that the diyne-allene moiety is formed by rearrangement of an alkyltriyne moiety.

On the activation of valerolactam with triflic anhydride: the synthesis of ω -trifluorosulfonamido dipeptides using a transpeptidation reaction under mild conditions

Nikolai Kuhnert,* Ian Clemens and Rodney Walsh

Valerolactam was activated with triflic anhydride to give an N-triflated derivative, which reacts with a series of nucleophiles to produce esters and amides under mild conditions.

Synthesis and evaluation of the glycosidase inhibitory activity of 5-hydroxy substituted isofagomine analogues

Mohammed M. Matin, Tarun Sharma, Sushma G. Sabharwal and Dilip D. Dhavale*

An efficient synthesis of 5-hydroxy substituted isofagomine analogues starting from D-glucose and their glycosidase inhibitory activity is presented.

Optical glucose detection across the visible spectrum using anionic fluorescent dyes and a viologen quencher in a two-component saccharide sensing system

David B. Cordes, Aaron Miller, Soya Gamsey, Zach Sharrett, Praveen Thoniyot, Ritchie Wessling and Bakthan Singaram*

Fluorescent anionic dyes are used in combination with a boronic acid-modified viologen quencher to sense glucose at pH 7.4 in buffered aqueous solution.

ARTICLES

1714

$1, N^6$ -Etheno-7-deaza-2,8-diazaadenosine: syntheses, properties and conversion to 7-deaza-2,8-diazaadenosine

Wenqing Lin, Hong Li, Xin Ming and Frank Seela*

The synthesis of the fluorescent etheno ribonucleoside **4** is described using compound **6** as educt. Nucleoside **4** was converted into 2,8-diaza-7-deazapurine nucleoside **5**.



1719

Assembly intermediates in polyketide biosynthesis: enantioselective syntheses of β-hydroxycarbonyl compounds

Christine Le Sann, Dulce M. Muñoz, Natalie Saunders, Thomas J. Simpson, David I. Smith, Florilène Soulas, Paul Watts and Christine L. Willis*

Functionalised β -hydroxycarbonyl compounds were prepared *via* aldol reactions with acylated oxazolidinone; reversal of stereoselectivity occurred using either (*R*)- or (*S*)-3-*tert*-butyldimethylsilyloxybutanal.

1729

Asymmetrized tris(hydroxymethyl)methane as a precursor of N- and O-containing 6-membered heterocycles through ring-closing metathesis

Luca Banfi, Giuseppe Guanti, Monica Paravidino and Renata Riva*

Enantiodivergent elaboration of a chemoenzymatically obtained acyclic chiral building block: an approach to iminosugars through ring-closing metathesis.





1738

Synthesis of aminocyclopentanols: α-D-galacto configured sugar mimics

Marie Bøjstrup and Inge Lundt*

Four aminocyclopentanols mimicking α-D-galactose were synthesised from carbohydrate starting materials and their inhibitory activity towards a range of glycosidases measured.



Powder-to-powder polycondensation of natural saccharides. Facile preparation of highly branched polysaccharides

Atsushi Kanazawa, Shohei Okumura and Masato Suzuki*

Natural saccharides were polymerized with a H_3PO_4 catalyst in the solid state at 110 °C, giving highly branched polysaccharides.









 $X = NR^2$

1,6-anhydride terminal unit Conv.=11-84 %, M,,=1400-19000, M,=1200-3700



1757



1768



46-93% isolated yields

1776

Solvent effects in the interaction of methyl-β-cyclodextrin with solvatochromic merocyanine dyes

Cristina de Garcia Venturini, Jürgen Andreaus, Vanderlei Gageiro Machado* and Clodoaldo Machado*

The spectroscopic behavior of dyes 1 and 2 was investigated in solutions of methyl- β -cyclodextrin in hydroxylic and dipolar non-protic solvents.

Factors affecting the selection of products from a photochemically generated singlet biradical

David A. Broyles and Barry K. Carpenter*

The chemistries of a monoradical of the ultrafast "radical-clock" type and a structurally related singlet biradical, generated by Norrish type II photochemistry, are compared. The monoradical is found to undergo the characteristic ring-opening reaction of its class while the singlet biradical shows no evidence of the analogous ring-opening reaction.

Selective synthesis of *C*-arylglycosides *via* Cp*RuClcatalyzed partially intramolecular cyclotrimerizations of *C*-alkynylglycosides

Yoshihiko Yamamoto,* Tomoaki Saigoku, Hisao Nishiyama, Takashige Ohgai and Kenji Itoh

In the presence of catalytic amounts of Cp*RuCl(cod), the partially intramolecular cyclotrimerizations of various *C*-alkynylglycosides and *C*-diynylglycosides proceeded at ambient temperature to afford *C*-arylglycosides.

Determining the $\sigma\text{-}\text{donor}$ ability of the cyclopropane C–C bond

Nathan L. Fifer and Jonathan M. White*

The strong σ -donor ability of the cyclopropane ring is demonstrated by X-ray structural analysis of derivatives of cyclopropylmethanol and dicyclopropylmethanol.

1781



Modification and structure–activity relationship of a small molecule HIV-1 inhibitor targeting the viral envelope glycoprotein gp120

Jingsong Wang, Nhut Le, Alonso Heredia, Haijing Song, Robert Redfield and Lai-Xi Wang*

This paper describes selected modification and structure–activity relationship of the small molecule HIV-1 inhibitor, 4-benzoyl-1-[(4-methoxy-1*H*-pyrrolo[2,3-*b*]pyridin-3-yl)oxoacetyl]-2-(*R*)-methylpiperazine (BMS-378806).

ARTICLES

1787

Fluorometric sensing of alkali metal and alkaline earth metal cations by novel photosensitive monoazacryptand derivatives in aqueous micellar solutions

Yoshio Nakahara, Toshiyuki Kida, Yohji Nakatsuji* and Mitsuru Akashi*

Novel monoazacryptand-type fluorescent chemosensors were found to detect alkali metal and alkaline earth metal cations with high selectivity in water in the presence of nonionic or anionic surfactant micelles.

1795

Synthesis of screening substrates for the directed evolution of sialic acid aldolase: towards tailored enzymes for the preparation of influenza A sialidase inhibitor analogues

Thomas Woodhall, Gavin Williams, Alan Berry and Adam Nelson*

Stereoselective methods for the synthesis of two diastereomeric sialic acid analogues are described. The compounds may be exploited in the directed evolution of sialic acid aldolase.





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Gageiro Machado, Vanderlei, 1751 Gamsey, Soya, 1708 Gao Jie 1617 Gokel, George W., 1647 Gokel, Michael R., 1647 Gorrichon, Liliane, 1612 Guanti, Giuseppe, 1729 Häberli, Adrian, 1653 Han, Zhenfu, 1622 Heredia, Alonso, 1781 Hodge, Philip, 1690 Hughes-Strange, George B., 1647 Itoh, Kenji, 1768 Kanazawa, Atsushi, 1746 Kashiwagi, Takehiro, 1670 Kawabata, Takeo, 1609 Kellogg, Richard M., 1631 Kida, Toshiyuki, 1787 Kishida, Taro, 1670 Kuhnert, Nikolai, 1694 Lazar, Adina N., 1659 Le Sann, Christine, 1719 Le, Nhut, 1781 Leevy, W. Matthew, 1647 Leumann, Christian J., 1653 Li, Hong, 1714 Lin, Wenqing, 1714 Lord, Jason C. D., 1624 Lundt, Inge, 1738 Machado, Clodoaldo, 1751 Majumdar, Swapan, 1609 Matile, Stefan, 1659 Matin, Mohammed M., 1702 Maver, Alain, 1653 Miller, Aaron, 1708

Ming, Xin, 1714 Monguchi, Daiki, 1609 Muñoz, Dulce M., 1719 Najjar, Fadia, 1612 Nakahara, Yoshio, 1787 Nakatsuji, Yohji, 1787 Nelson, Adam, 1795 Nishihara, Masamichi, 1659 Nishivama, Hisao, 1768 Ohgai, Takashige, 1768 Okazaki, Momotoshi, 1670 Okumura, Shohei, 1746 Oshima, Koichiro, 1622 Paravidino, Monica, 1729 Perret, Florent, 1659 Peyrane, Frédéric, 1685 Pieraccini, Daniela, 1624 Player, Tomoko N., 1615 Purkiss, David W., 1676 Rastrelli, Federico, 1624 Redfield, Robert, 1781 Riva, Renata, 1729 Sabharwal, Sushma G., 1702 Saigoku, Tomoaki, 1768 Sakai, Naomi, 1659 Saunders, Natalie, 1719 Seela, Frank, 1714 Sharma, Tarun, 1702 Sharrett, Zach, 1708 Shi, Min, 1620 Shi, Yong-Ling, 1620 Shinoda, Satoshi, 1615 Shinokubo, Hiroshi, 1622 Siegel, Jay S., 1591 Simpson, Thomas J., 1719

Singaram, Bakthan, 1708 Smith, David I., 1719 Song, Haijing, 1781 Soulas, Florilène, 1719 Sugahara, Takuya, 1670 Surowiec, Kazimierz, 1676 Suzuki, Masato, 1746 Takeuchi, Toshihide, 1659 Thonivot, Praveen, 1708 Tor, Yitzhak, 1591 Tsubaki, Kazunori, 1609 Tsukube, Hiroshi, 1615 Usugi, Shin-ichi, 1622 van Esch, Jan, 1631 Vial, Henri, 1612 Walsh, Rodney, 1694 Wang, Guan-Wu, 1617 Wang, Jingsong, 1781 Wang, Lai-Xi, 1781 Watts, Paul, 1719 Weber, Michelle E., 1647 Wessling, Ritchie, 1708 White, Jonathan M., 1776 Williams, Gavin, 1795 Willis, Christine L., 1719 Woodhall, Thomas, 1795 Xia, Jing-Jing, 1617 Yamamoto, Yoshihiko, 1768 Yamauchi, Satoshi, 1670 Yorimitsu, Hideki, 1622 Zhang, Ze, 1617 Zhou, Hui, 1676

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Facile synthesis of multisubstituted buta-1,3-dienes *via* Suzuki–Miyaura and Kumada cross-coupling strategy of **2,4-diiodo-buta-1-enes with arylboronic acids and Grignard reagents** Li-Xiong Shao and Min Shi (DOI: 10.1039/b504071j)

Model compounds for (6–4) photolyases: a comparative flavin induced cleavage study of oxetanes and thietanes Marcus G. Friedel, Michaela K. Cichon and Thomas Carell (**DOI**: 10.1039/b503205a)

Radical properties governing the hypoxia-selective cytotoxicity of antitumor 3-amino-1,2,4-benzotriazine 1,4-dioxides Robert F. Anderson, Sujata S. Shinde, Michael P. Hay, Swarna A. Gamage and William A. Denny (DOI: 10.1039/b502586a)

Deprotonation - electrophile trapping of terminal epoxides

David M. Hodgson, Eirene H. M. Kirton, Steven M. Miles, Stéphanie L. M. Norsikian, Nigel J. Reynolds and Steven J. Coote (**DOI**: 10.1039/b502888d)

 $\label{eq:prebiotic carbohydrate synthesis: zinc-proline catalyzes direct aqueous aldol reactions of α-hydroxy aldehydes and ketones$

Jacob Kofoed, Jean-Louis Reymond and Tamis Darbre (DOI: 10.1039/b501512j)

Design and synthesis of new bicyclic diketopiperazines as scaffolds for receptor probes of structurally diverse functionality Pedro Besada, Liaman Mamedova, Craig J. Thomas, Stefano Costanzi and Kenneth A. Jacobson (**DOI**: 10.1039/b416349d)

Syntheses and copper(II)-dependent DNA photocleavage by acridine and anthracene 1,10-phenanthroline conjugate systems

Lourdes Gude, María-José Fernández, Kathryn B. Grant and Antonio Lorente (DOI: 10.1039/b502485d)

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Papers

Identification of the F1F0 mitochondrial ATPase as a target for modulating skin pigmentation by screening a tagged triazine library in zebrafish

Da-Woon Jung, Darren Williams, Sonya M. Khersonsky, Tae-Wook Kang, Noushin Heidary, Young-Tae Chang and Seth J. Orlow

Protease profiling using a fluorescent domino peptide cocktail

Yang Yongzheng and Jean-Louis Reymond

Protein immunosensor using single-wall carbon nanotube forests with electrochemical detection of enzyme labels Xin Yu, Sang Nyon Kim, Fotios Papadimitrakopoulos and James F. Rusling

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