

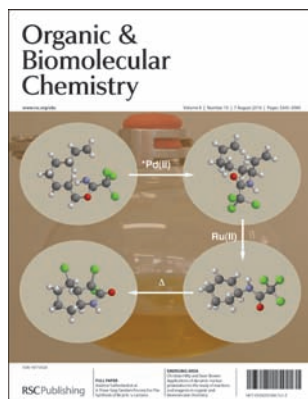
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IN THIS ISSUE

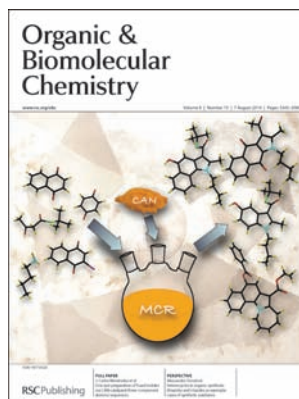
ISSN 1477-0520 CODEN OBCRAK 8(15) 3345–3580 (2010)



Cover

See Sutherland *et al.*, pp. 3418–3425.
 A one-pot, three-step tandem process involving an Overman rearrangement, a ring closing metathesis reaction and a Kharasch cyclisation has been developed for the asymmetric synthesis of bicyclic γ -lactams.

Image reproduced by permission of Fiona I. McGonagle, Lindsay Brown, Andrew Cooke and Andrew Sutherland from *Org. Biomol. Chem.*, 2010, **8**, 3418.



Inside cover

See Menéndez *et al.*, pp. 3426–3436.
 The inside cover picture shows the range of structurally diverse indole-related nitrogen heterocycles that can be synthesized on the basis of a CAN-catalyzed three-component reaction between primary amines, β -dicarbonyl compounds and (2-bromo)naphthoquinones.

Image reproduced by permission of Padmakar A. Suryavanshi, Vellaisamy Sridharan and J. Carlos Menéndez from *Org. Biomol. Chem.*, 2010, **8**, 3426.

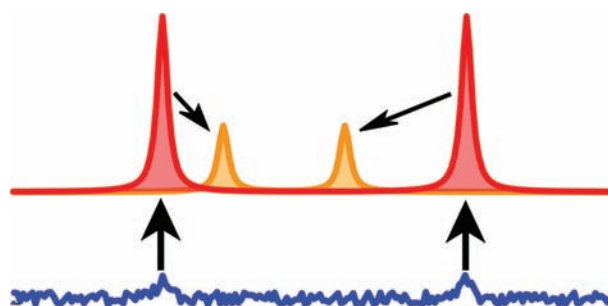
EMERGING AREA

3361

Applications of dynamic nuclear polarization to the study of reactions and reagents in organic and biomolecular chemistry

Christian Hilty* and Sean Bowen

Hyperpolarization enhances sensitivity by several orders of magnitude, enables NMR spectroscopy with minute amounts of sample, and allows the determination of kinetics and mechanisms of reactions through real time measurement.



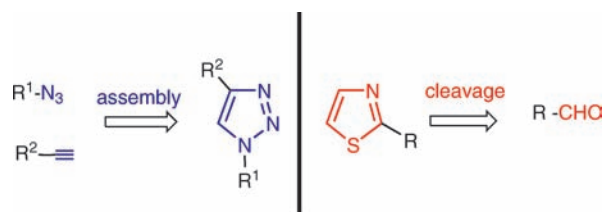
PERSPECTIVE

3366

Heterocycles in organic synthesis: thiazoles and triazoles as exemplar cases of synthetic auxiliaries

Alessandro Dondoni*

Some heterocycles are precious tools in the tool-box of synthetic organic chemists as they can serve as auxiliaries for the formation of non-heterocyclic material. This Perspective article illustrates the key role of thiazole and triazole in the work carried out in the author's laboratory over three decades and deals with the synthesis of carbohydrate-based bioactive molecules.



triazole serves as a linker while thiazole is a masked functionality

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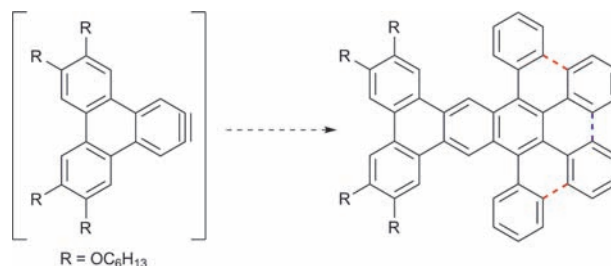
COMMUNICATIONS

3386

Aryne-mediated syntheses of structurally related acene derivatives

Diego Rodríguez-Lojo, Diego Peña,* Dolores Pérez* and Enrique Guitián

Three large substituted acene derivatives characterized by the *cata*-condensation of 5, 8 and 11 benzene rings have been obtained by cycloaddition reactions of the same aryne.

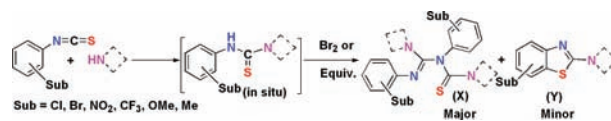


3389

Arylthioureas with bromine or its equivalents gives no 'Hugerschoff' reaction product

Ramesh Yella, Siva Murru, Abdur Rezzak Ali and Bhisma K. Patel*

The reaction of aryl-alkyl unsymmetrical thiourea with bromine or its equivalent gives product having thioamido guanidino moiety (X) and not the expected 2-aminobenzothiazole (Y). A plausible reaction mechanism has been proposed.

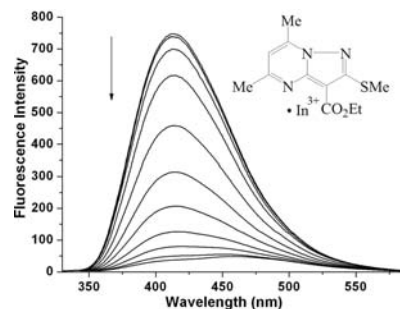


3394

A sensitive and highly selective fluorescent sensor for In³⁺

Yan-Chao Wu,* Hui-Jing Li and Hua-Zheng Yang

A simple neutral fluorescent sensor based on pyrazolo[1,5-*a*]pyrimidine exhibited a unique selectivity for indium(III) ion (In³⁺) over various other metal ions with dramatic fluorescence response in acetonitrile.

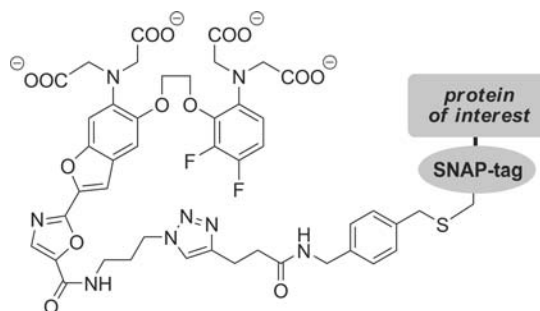


3398

Fura-2FF-based calcium indicator for protein labeling

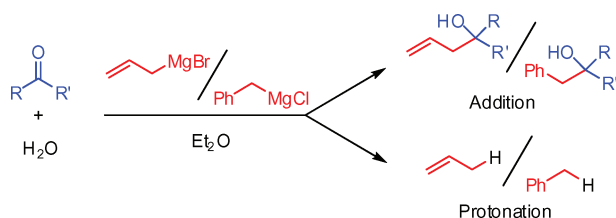
Agostina A. Ruggiu, Michael Bannwarth and Kai Johnsson*

A Fura-2FF-based fluorescent Ca²⁺ indicator that can be covalently linked to SNAP-tag fusion proteins and retains its Ca²⁺ sensing ability after coupling to protein is described.



COMMUNICATIONS

3402

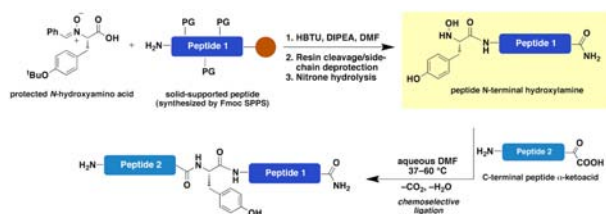
**Ultrafast Grignard addition reactions in the presence of water**

Gyorgyi Osztrovszky, Torkil Holm* and Robert Madsen*

For two highly reactive Grignard reagents (allylMgBr and benzylMgCl) the rate of carbonyl addition is comparable to the rate of protonation by water.

PAPERS

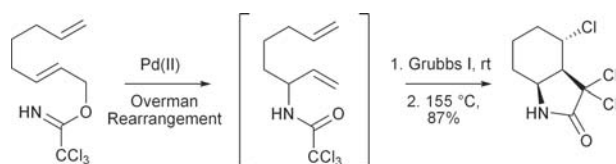
3405

**Nitrene protecting groups for enantiopure N-hydroxyamino acids: synthesis of N-terminal peptide hydroxylamines for chemoselective ligations**

S. Irene Medina, Jian Wu and Jeffrey W. Bode*

Enantiopure peptide N-terminal hydroxylamines are key substrates for a chemoselective ligation with α -ketoacids to form native peptide bonds. A robust method for their synthesis in enantiopure form *via* N-benzylidene nitrene protected intermediates is described. This procedure provides intermediates and protocols compatible with Fmoc-based solid phase peptide synthesis.

3418

**A three-step tandem process for the synthesis of bicyclic γ -lactams**

Fiona I. McGonagle, Lindsay Brown, Andrew Cooke and Andrew Sutherland*

A one-pot, three-step tandem process has been developed for the direct synthesis of functionalised bicyclic [3.3.0], [4.3.0] and [5.3.0] γ -lactams from simple allylic trichloroacetimidates.

3426

**Expedient, one-pot preparation of fused indoles *via* CAN-catalyzed three-component domino sequences and their transformation into polyheterocyclic compounds containing pyrrolo[1,2-*a*]azepine fragments**

Padmakar A. Suryavanshi, Vellaisamy Sridharan and J. Carlos Menéndez*

The CAN-catalyzed three-component reaction between primary amines, β -dicarbonyl compounds and naphthoquinones or 2-bromonaphthoquinones afforded, respectively, 5-hydroxybenzo[*g*]indoles and benzo[*f*]indole-4,9-diones. Further transformations were also studied.

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Introducing Dr Ming-Qiang Zhang

Associate Editor

Ming-Qiang Zhang is Chief Technology Officer and Vice-President for External Research at Roche R&D Centre (China). He also holds a professorship at Huaqiao University, China. He is one of the pioneers in applying novel molecular modalities such as supramolecular chemistry and synthetic biology to drug discovery and has led the discovery of multiple candidate drugs progressed to approval or clinical development for treatment of CNS, cancer, inflammatory and infectious diseases. He is a co-recipient of the RSC Malcolm Campbell Memorial Prize in Biological and Medicinal Chemistry (2007) and Nexus Life Science Award for Innovation (2008) for the discovery of sugammadex (Bridion®, Merck & Co), the first-in-class muscle relaxant reversing agent approved for clinical use. Dr Zhang received his PhD in medicinal chemistry from the University of Antwerp in Belgium (1990) and did his post-doctoral research at Vrije Universiteit Amsterdam in the Netherlands (1991). After working at the Leiden-Amsterdam Center for Drug Research (1991-1997), he joined Organon Laboratories where he was the Section Head of Medicinal Chemistry before becoming Director of Medicinal Chemistry at Shire Pharmaceuticals (2002-2004). In 2004 he joined Biotica Technology initially as Vice-President of Research and then Senior Vice-President of Research & Development (2004-2009), where he played a key role in transforming the company to drug discovery and was instrumental in establishing the two significant R&D partnerships with Wyeth Pharmaceuticals (now Pfizer) and GlaxoSmithKline, respectively. He also co-founded ViroChem Pharma Inc in 2004, which was acquired by Vertex in 2009.

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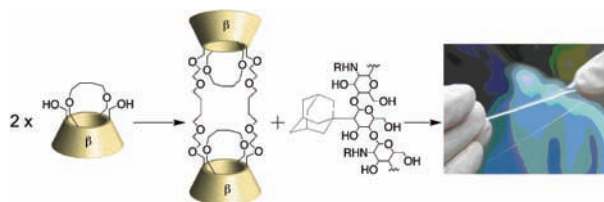
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3437

Duplex of capped-cyclodextrins, synthesis and cross-linking behaviour with a biopolymer

Olivia Bistri-Aslanoff, Yves Blériot, Rachel Auzely-Velty* and Matthieu Sollogoub*

The first doubly linked dimer of capped cyclodextrins was synthesized and studied as a cross-linking agent of adamantane-grafted chitosan.

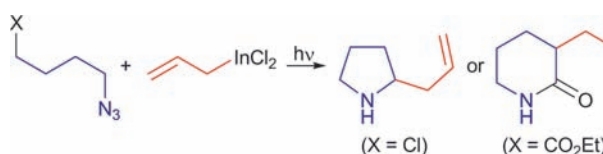


3444

Radical allylations by reaction of azides with allylindium dichloride

Giorgio Bencivenni, Tommaso Lanza, Matteo Minozzi, Daniele Nanni,* Piero Spagnolo and Giuseppe Zanardi

Allylindium dichloride is an effective reagent for converting suitable azides into allylated *N*-heterocycles through generation of indiumaminyl radicals followed by tandem 1,5-H shift and allylation of the resulting carbon radicals. Theoretical calculations showed that, compared to AlSnMe_3 , AlInCl_2 favours the overall process because of both a lower BDE of the allyl-metal bond and a considerably faster rearrangement.

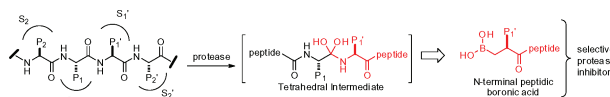


3451

N-Terminal peptidic boronic acids selectively inhibit human ClpXP

Kenneth Knott, Jennifer Fishovitz, Steven B. Thorpe, Irene Lee and Webster L. Santos*

We report the synthesis and development of *N*-terminal peptidic boronic acids as novel protease inhibitors. These boronic acids harvest the unique selectivity inherent on the P' -site of peptide substrates. Our effort provides the first selective inhibitor of hClpXP over hLon; both are ATP-dependent serine proteases present in mitochondrial matrix.

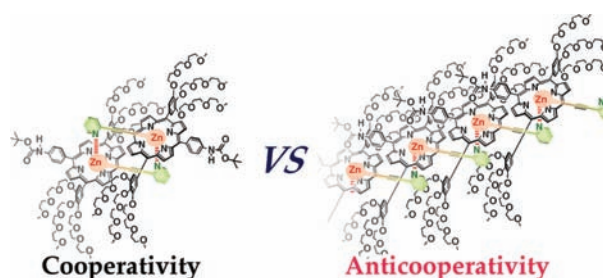


3457

Ligand-assisted J-type aggregates of zinc porphyrin: anticooperative molecular organization in self-assembled bolaamphiphile

Mitsuhiko Morisue,* Takefumi Morita and Yasuhisa Kuroda

Aqueous pyridine-appended porphyrin formed J-type aggregates *via* successive pyridyl-to-zinc coordination in self-assemblies. In contrast, an antiparallel dimer was organized *via* self-complementary coordination in non-coordinating organic solution.



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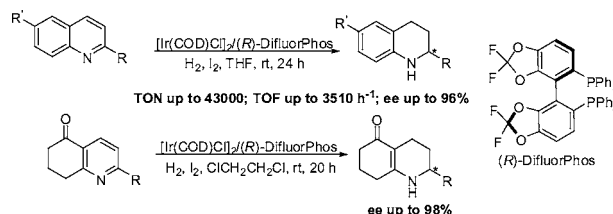
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3464

Highly efficient and enantioselective hydrogenation of quinolines and pyridines with Ir-Difluorophos catalyst

Weijun Tang, Yawei Sun, Lijin Xu,* Tianli Wang, Qinghua Fan, Kim-Hung Lam and Albert S. C. Chan

The combination of chiral bisphosphine ligand Difluorophos with $[\text{Ir}(\text{COD})\text{Cl}]_2$ resulted in a highly efficient catalyst system for asymmetric hydrogenation of quinolines and trisubstituted pyridines, affording the corresponding products with high enantioselectivities, excellent catalytic activities and productivities.

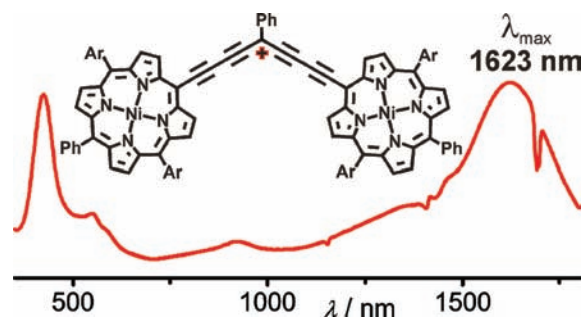


3472

Extending conjugation in porphyrin dimer carbocations

Karl J. Thorley and Harry L. Anderson*

A series of conjugated porphyrin dimer carbocations have been synthesised with varying length bridges between the porphyrin end groups and the carbocation centre. The absorption spectra of the carbocations show a strong length dependency, with intense bands deep into the near infrared.

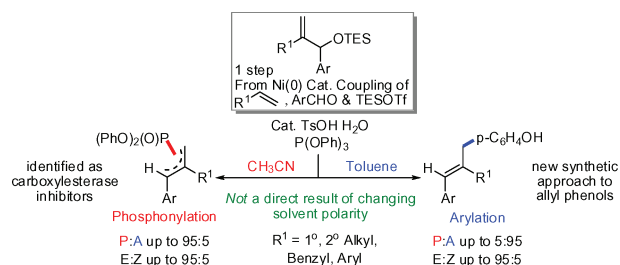


3480

Nitrile assisted, Brønsted acid catalyzed regio and stereoselective diarylphosphonylation of allyl silyl ethers

Chun-Yu Ho,* Chun-Wa Chan, Siu-Kwan Wo, Zhong Zuo* and Lai-Ying Chan

We have discovered catalytic protocols for regio- and stereoselective synthesis of trisubstituted allyl diarylphosphonates or phenolic compounds from the corresponding allyl silyl ethers. The reaction media employed can have dramatic effect on reaction outcome.

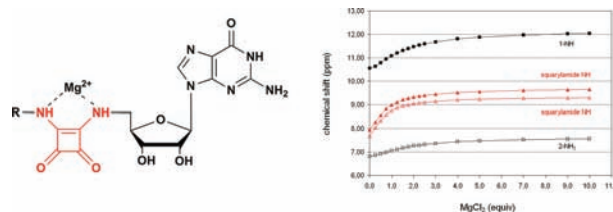


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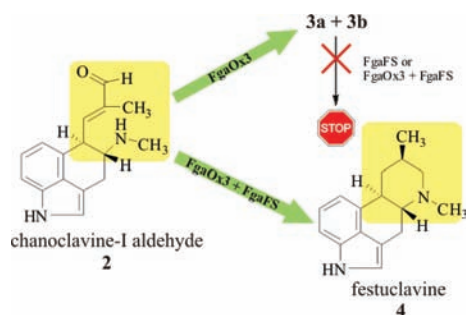
Rationally designed squaryldiamides – a novel class of sugar-nucleotide mimics?

Sven Niewiadomski, Zeenat Beebeejaun, Helen Denton, Terry K. Smith, Richard J. Morris and Gerd K. Wagner*

New GDP-sugar mimics based on a squaryldiamide fragment coordinate readily to a divalent metal, and show some inhibitory activity against a GDP-mannose-dependent mannosyltransferase.



3500

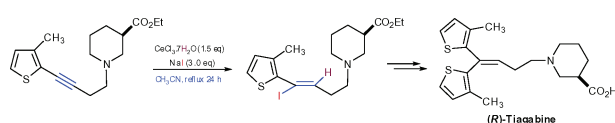


Ergot alkaloid biosynthesis in *Aspergillus fumigatus*: Conversion of chanoclavine-I aldehyde to festuclavine by the festuclavine synthase FgaFS in the presence of the old yellow enzyme FgaOx3

C. Wallwey, M. Matuschek, X.-L. Xie and S.-M. Li*

The conversion of chanoclavine-I aldehyde (**2**) to festuclavine (**4**) was demonstrated by using old yellow enzyme FgaOx3 and festuclavine synthase FgaFS. In the absence of FgaFS, **2** was converted by FgaOx3 to two shunt products **3a** and **3b**, which were not accepted by FgaFS as substrates.

3509

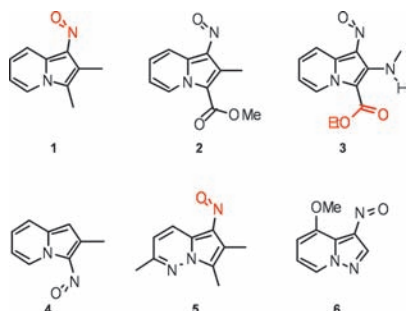


A convergent approach to (*R*)-Tiagabine by a regio- and stereocontrolled hydroiodination of alkynes

Giuseppe Bartoli, Roberto Cipolletti, Giustino Di Antonio, Riccardo Giovannini, Silvia Lanari, Mauro Marcolini and Enrico Marcantoni*

An efficient methodology for the iodofunctionalization of carbon–carbon triple bonds has efficiently been applied to the preparation of (*R*)-Tiagabine, a GABA uptake inhibitor marketed from the treatment of epilepsy.

3518

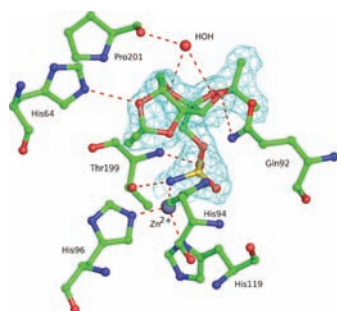


Conformational equilibria and barriers to rotation in some novel nitroso derivatives of indolizines and 3- and 5-azaindolizines – an NMR and molecular modeling study

Ion Ghiviriga,* Bahaa El-Dien M. El-Gendy, Henry Martinez, Dmytro Fedoseyenko, Eric P. Metais, Aziz Fadli and Alan R. Katritzky*

The conformational preferences of compounds **1–6** have been identified by NMR and explained by molecular modeling.

3528



The first example of a significant active site conformational rearrangement in a carbonic anhydrase-inhibitor adduct: the carbonic anhydrase I–topiramate complex

Vincenzo Alterio, Simona Maria Monti, Emanuela Truppo, Carlo Pedone, Claudiu T. Supuran* and Giuseppina De Simone*

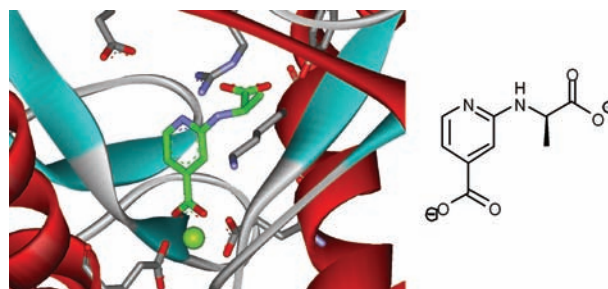
The crystallographic structure of the adduct which TPM, a widely used antiepileptic drug, forms with human Carbonic Anhydrase (CA) I, has been reported, showing for the first time a significant conformational rearrangement of the CA active site upon binding of the inhibitor.

3534

Inhibition of chorismate-utilising enzymes by 2-amino-4-carboxypyridine and 4-carboxypyridone and 5-carboxypyridone analogues

Richard J. Payne,* Esther M. M. Bulloch, Olivier Kerbarh and Chris Abell*

A number of 2-amino-4-carboxypyridine, 4- and 5-carboxypyridone-based compounds were prepared. Several compounds proved to be low micromolar inhibitors when screened against three members of the chorismate-utilising enzyme family.

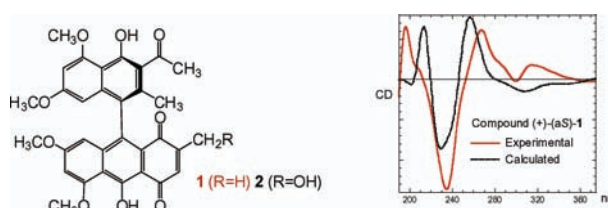


3543

Structure and absolute configuration of toxic polyketide pigments from the fruiting bodies of the fungus *Cortinarius rufo-olivaceus*

Jin-Ming Gao,* Jian-Chun Qin, Gennaro Pescitelli, Sebastiano Di Pietro, Ya-Tuan Ma and An-Ling Zhang

The structures, including the axial chirality configurations, of four polyketide pigments (**1–4**) isolated from *Cortinarius rufo-olivaceus*, were determined by spectroscopic analysis and their CD spectra and ZINDO and TDDFT calculations.

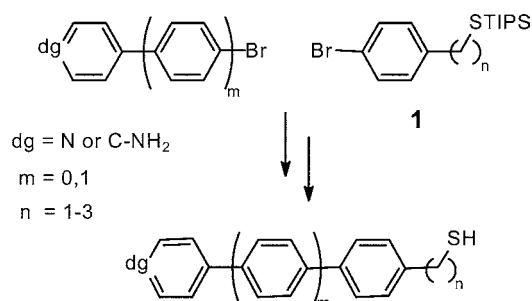


3552

A divergent synthesis of oligoarylalkane thiols with Lewis-basic N-donor termini

Björn Schüpbach and Andreas Terfort*

The gate to functional surfaces: Using a homologous series of triisopropylsilyl (TIPS) protected 4-bromophenylalkane thiols **1** as central building blocks, functional thiols for the generation of highly ordered self-assembled monolayers become accessible by short reaction sequences. The efficiency of this approach is demonstrated by the syntheses of six oligophenylalkane thiols with amino or pyridine head groups.

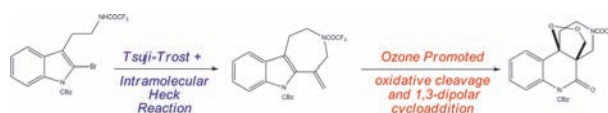


3563

Formation and reactions of azepino[4,5-*b*]indoles: an unprecedented ozone reaction in the formation of novel benzo[*c*]naphthyridinones

Scott G. Stewart,* Emilio L. Ghisalberti, Brian W. Skelton and Charles H. Heath

Herein we report the formation and interesting reactivity of several azepino[4,5-*b*]indole heterocycles bearing an additional olefin. Treatment of these ring systems with ozone results in an unprecedented secondary reaction of the Criegee intermediate, to afford a benzo[*c*]naphthyridione containing a bridging cyclic peroxide.





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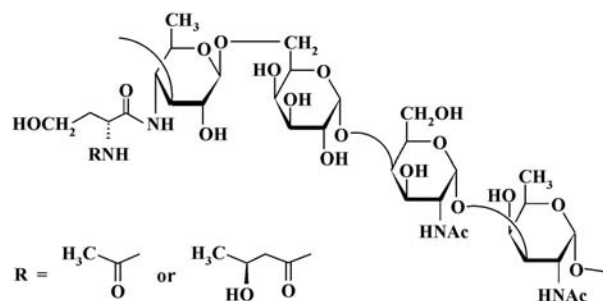
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3571

Structure of the O-antigen of *Acinetobacter lwoffii* EK30A; identification of D-homoserine, a novel non-sugar component of bacterial polysaccharides

Nikolay P. Arbatsky, Anna N. Kondakova,*
Alexander S. Shashkov, Marina S. Drutskaya,
Pavel V. Belousov, Sergei A. Nedospasov, Mayya A. Petrova
and Yuriy A. Knirel

A peculiar feature of the O-antigen of the bacterium *A. lwoffii* EK30A is the presence of D-homoserine N-substituted with either acetyl group (~50%) or with (S)-3-hydroxybutanoyl group (~50%).



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Electronic supplementary information (ESI) is available *via* the online article (see <http://www.rsc.org/esi> for general information about ESI).

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