

Organic & Biomolecular Chemistry

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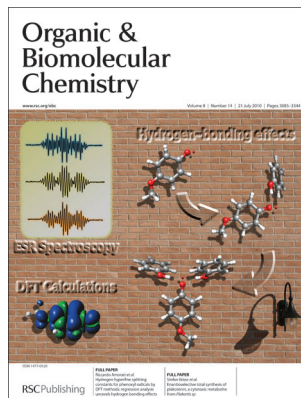
ISSN 1477-0520 CODEN OBCRAK 8(14) 3085–3344 (2010)



Cover

See Lucile Fischer and Gilles Guichard, pp. 3101–3117. Just like their oligoamide congeners, aromatic and aliphatic urea-based oligomers show high propensity to fold and to self-assemble. Ultimately, structural knowledge is providing a basis for *function*

Image reproduced by permission of Gilles Guichard from *Org. Biomol. Chem.*, 2010, **8**, 3101.



Inside cover

See Riccardo Amorati *et al.*, pp. 3136–3141. Joint ESR spectroscopic measures and DFT theoretical calculations shed new light on hydrogen bonds formation between phenols and biologically relevant phenoxyl radicals

Image reproduced by permission of Riccardo Amorati from *Org. Biomol. Chem.*, 2010, **8**, 3136.

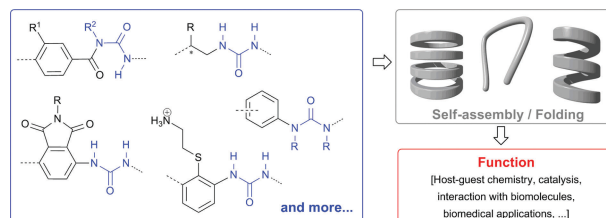
PERSPECTIVE

3101

Folding and self-assembly of aromatic and aliphatic urea oligomers: Towards connecting structure and function

Lucile Fischer and Gilles Guichard*

Aromatic and aliphatic urea-based oligomers have been designed to fold and/or self-assemble. Structural insight provides a basis for the elaboration of molecules with function.



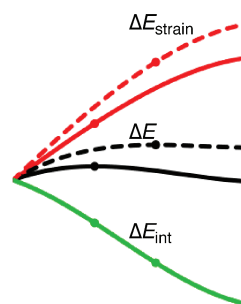
EMERGING AREA

3118

The activation strain model of chemical reactivity

Willem-Jan van Zeist and F. Matthias Bickelhaupt*

We provide an account of the activation strain model and how this model yields insight into the origin of reaction barriers and trends therein, by decomposing the reaction's energy profile (ΔE) into strain energy in the reactants (ΔE_{strain}) plus interaction between the reactants (ΔE_{int}).



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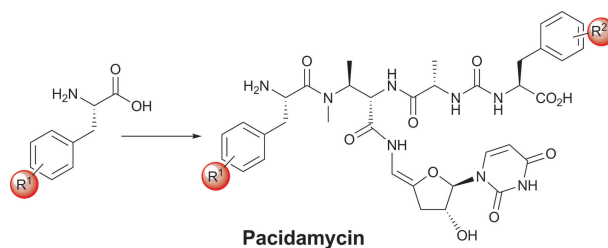
COMMUNICATIONS

3128

New pacidamycins biosynthetically: probing N- and C-terminal substrate specificity

Amany E. Ragab, Sabine Grüschow, Emma J. Rackham and Rebecca J. M. Goss*

Feeding phenylalanine analogues to *Streptomyces coeruleorubidus* reveals the remarkable steric and electronic flexibility of this biosynthetic pathway and leads to the generation of a series of new halopacidamycins.

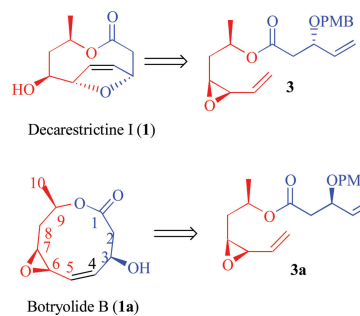


3130

Total synthesis of decarestrictine I and botrylide B via RCM protocol

Palakodety Radha Krishna* and T. Jagannadha Rao

A convergent stereoselective total synthesis of decarestrictine I (**1**) and botrylide B (**1a**) invoking a common synthetic strategy is reported.

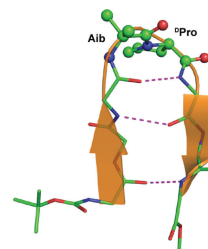


3133

Peptide hairpin nucleation with the obligatory Type I' β -turn Aib-^DPro segment

Upadhyayula Surya Raghavender, Subrayashastry Aravinda, Rajkishor Rai, Narayanaswamy Shamala* and Padmanabhan Balaram*

β -Hairpin nucleated by an Aib-^DPro Type I' β -turn segment



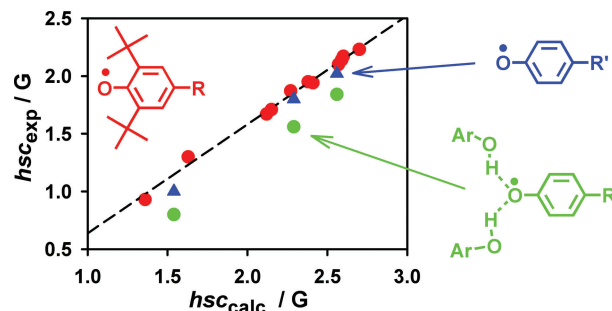
PAPERS

3136

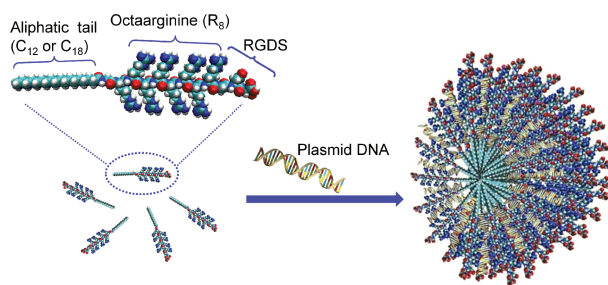
Hydrogen hyperfine splitting constants for phenoxy radicals by DFT methods: regression analysis unravels hydrogen bonding effects

Riccardo Amorati,* Gian Franco Pedulli and Maurizio Guerra

DFT calculations provide, after scaling the results, quantitative estimates of hydrogen hyperfine splitting constants in phenoxy radicals. The deviations observed for unhindered phenoxy radicals are explained in terms of H-bond formation with their parent phenols.



3142

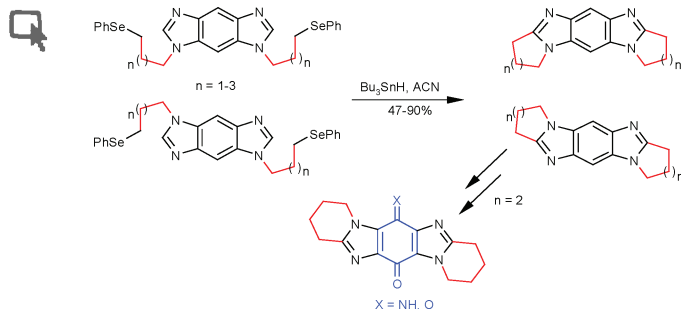


Amphiphilic cationic lipopeptides with RGD sequences as gene vectors

Jing-Xiao Chen, Hui-Yuan Wang, Chang-Yun Quan, Xiao-Ding Xu, Xian-Zheng Zhang* and Ren-Xi Zhuo

Two kinds of arginine-rich amphiphilic lipopeptides with hydrophobic aliphatic tails were designed and synthesized as functional gene vectors. Due to the incorporation of RGD sequences, these two amphiphilic lipopeptides exhibited improved transfection efficiency in HeLa cells.

3149

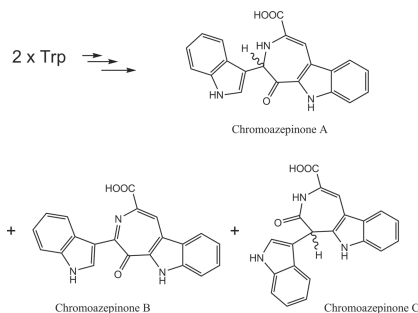


One-pot double intramolecular homolytic aromatic substitution routes to dialicyclic ring fused imidazobenzimidazolequinones and preliminary analysis of anticancer activity

Vincent Fagan, Sarah Bonham, Michael P. Carty and Fawaz Aldabbagh*

New quinone and iminoquinone anticancer agents that show specificity towards cervical and prostate cancer cell lines were prepared *via* double alkyl radical cyclizations onto imidazobenzimidazoles.

3157

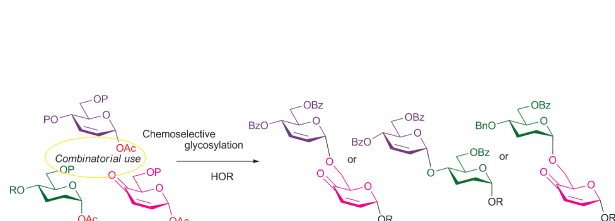


Novel tryptophan metabolites, chromoazepinone A, B and C, produced by a blocked mutant of *Chromobacterium violaceum*, the biosynthetic implications and the biological activity of chromoazepinone A and B

Takaaki Mizuoka, Kazufumi Toume, Masami Ishibashi and Tsutomu Hoshino*

The chemical mutagenesis of *Chromobacterium violaceum* afforded novel tryptophan metabolites, named chromoazepinone A–C. The biosynthetic pathways for the novel compounds are proposed and the inhibition effect of Wnt signal transcriptional activity is reported.

3164



Effective and chemoselective glycosylations using 2,3-unsaturated sugars

Shunichi Kusumi, Kaname Sasaki, Sainan Wang, Tatsuya Watanabe, Daisuke Takahashi and Kazunobu Toshima*

The 2,3-unsaturated glycosyl donors were found to exhibit high reactivity, while the corresponding 2,3-unsaturated-4-keto glycosyl donors showed low reactivity, under several conditions. These findings make it possible to realize chemoselective glycosylations *via* combinatorial uses of 2,3-unsaturated, 2,3-unsaturated-4-keto, and 2,3-dideoxy glycosyl donors providing various types of deoxyoligosaccharides in short-steps.

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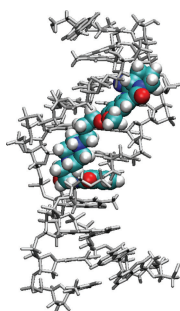
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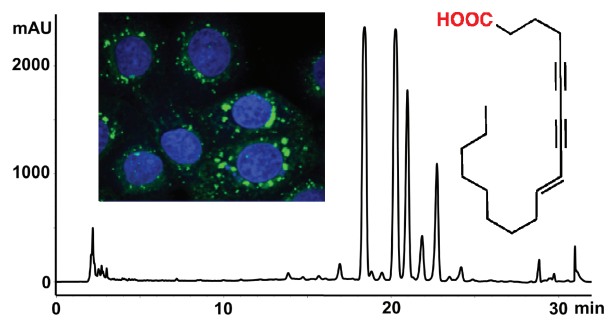


NMR structural studies on the covalent DNA binding of a pyrrolobenzodiazepine–naphthalimide conjugate

Michael Rettig, Walter Langel, Ahmed Kamal and Klaus Weisz*

The solution structure of a DNA adduct with a dual alkylating/intercalating PBD–naphthalimide conjugate has been determined by NMR and molecular dynamics simulations.

3188

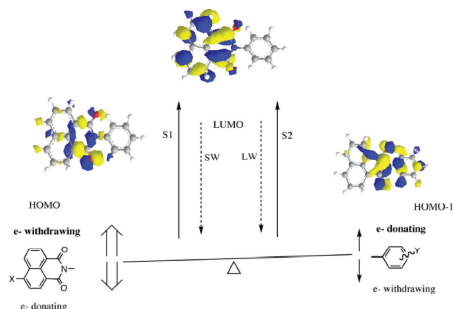


Heterofibrins: inhibitors of lipid droplet formation from a deep-water southern Australian marine sponge, *Spongia (Heterofibria) sp.*

Angela A. Salim, James Rae, Frank Fontaine, Melissa M. Conte, Zeinab Khalil, Sally Martin, Robert G. Parton and Robert J. Capon*

A southern Australian marine sponge, *Spongia (Heterofibria) sp.*, yielded new diene-ene fatty acids, which inhibit lipid droplet formation.

3195

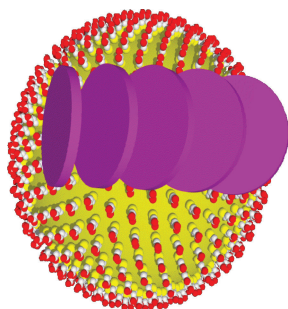


Frontier molecular orbital analysis of dual fluorescent dyes: predicting two-color emission in *N*-Aryl -1,8-naphthalimides

Premchendar Nandhikonda, Michael P. Begaye, Zhi Cao and Michael D. Heagy*

A predictive tool for the photoexcited states of *N*-phenyl-1,8-naphthalimides is proposed as a seesaw balanced photophysical model. A synthetic matrix of nine dyes demonstrates that this model serves as a guide to optimizing dual fluorescence emission.

3202



Modified porphyrin–brucine conjugated to gold nanoparticles and their application in photodynamic therapy

Kamil Záruba, Jarmila Králová, Pavel Řezanka, Pavla Poučková, Lenka Veverková and Vladimír Král*

Two porphyrin photosensitizers were immobilized on gold nanoparticles and their suitability for both *in vitro* and *in vivo* photodynamic therapy was tested.

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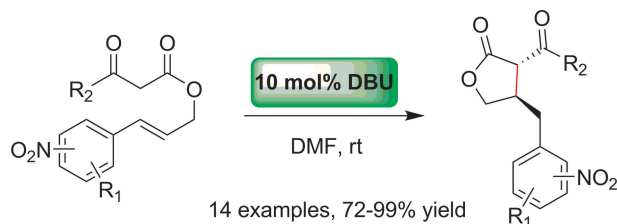
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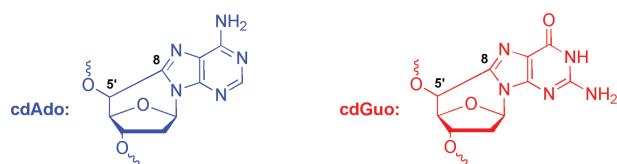


Intramolecular Michael addition reaction for the synthesis of benzylbutyrolactones

Hu He, Li-Xin Dai and Shu-Li You*

A Michael addition reaction of nitro-substituted aryl allyl β -ketocarboxylates has been developed. The reaction provides an efficient method for the preparation of γ -butyrolactone derivatives in good to excellent yields under mild conditions in the presence of catalytic amounts of DBU.

3211

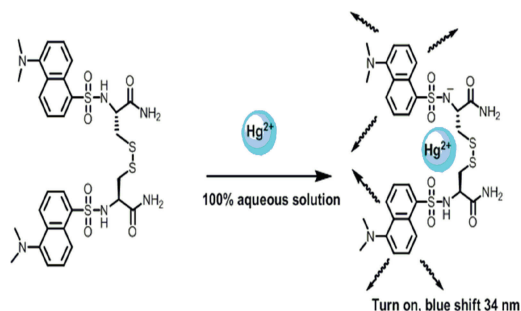


Radiation-induced formation of purine 5',8-cyclonucleosides in isolated and cellular DNA: high stereospecificity and modulating effect of oxygen

Noureddine Belmadoui, Fabien Boussicault, Maurizio Guerra, Jean-Luc Ravanat, Chrystostomos Chatgililoglu* and Jean Cadet*

The radiation-induced formation of cdAdo and cdGuo in isolated and cellular DNA has been reevaluated. Their levels were found to decrease steadily with the increase of O_2 concentration in isolated DNA, the 5'*R* diastereomers being predominant.

3220

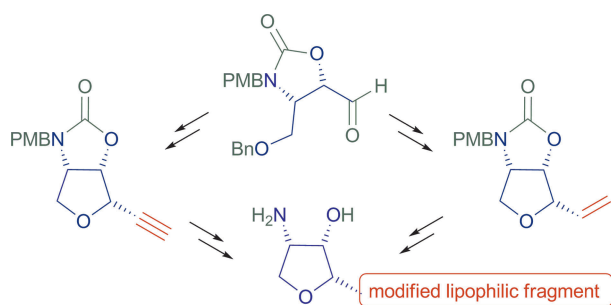


A highly sensitive and selective detection of Hg(II) in 100% aqueous solution with fluorescent labeled dimerized Cys residues

Bishnu Prasad Joshi, Chuda Raj Lohani and Keun-Hyeung Lee*

A highly sensitive sensor ($K_d = 41$ nM) for detecting Hg(II) ion in 100% aqueous solution, based on the dimerized L-Cys residues with two dansyl fluorophores was satisfactory for monitoring the maximum allowable level (2 ppb) of mercury ion in drinking water demanded by EPA.

3227



Flexible and enantioselective access to jaspine B and biologically active chain-modified analogues thereof

Yahya Salma, Stéphanie Ballereau, Carine Maaliki, Sonia Ladeira, Nathalie Andrieu-Abadie and Yves Génisson*

Jaspine B as well as five chain-modified analogues thereof were prepared allowing identification of a series of potent and cytotoxic inhibitors of sphingomyelin production in murine melanoma cells.

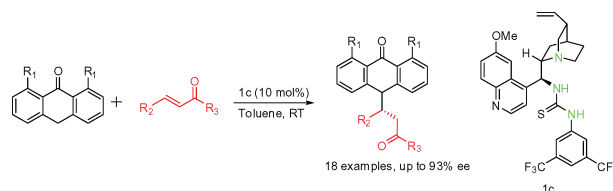
PAPERS

3244

Asymmetric organocatalytic Michael addition of anthrone to enone

Chunlin Wu, Wenjun Li, Juanjuan Yang, Xinmiao Liang* and Jinxing Ye*

The enantioselective organocatalytic Michael addition of anthrones to α,β -unsaturated ketones catalyzed by a bifunctional organocatalyst in toluene can afford the desired Michael adducts in high yields and excellent enantioselectivities.

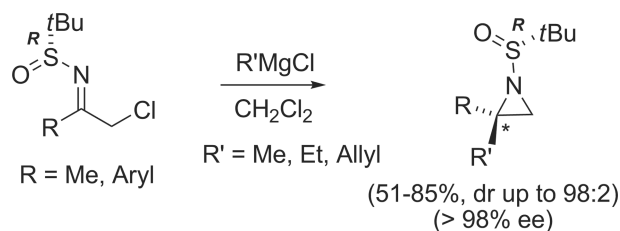


3251

Asymmetric synthesis of new chiral *N*-sulfinyl 2,2-disubstituted aziridines by Grignard additions across α -chloro *N*-sulfinyl ketimines

Filip Colpaert, Sven Mangelinckx, Erika Leemans, Bram Denolf and Norbert De Kimpe*

The reaction of chiral α -chloro *N*-*tert*-butanesulfinyl ketimines, derived from α -chloro ketones, with various Grignard reagents was developed as an attractive general two-step stereoselective pathway to new chiral *N*-sulfinyl 2,2-disubstituted aziridines.

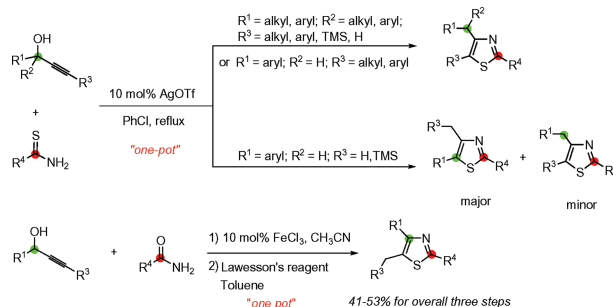


3259

Facile one-pot synthesis of three different substituted thiazoles from propargylic alcohols

Xun Gao, Ying-ming Pan, Min Lin, Li Chen and Zhuang-ping Zhan*

Three different substituted thiazoles have been successfully synthesized from readily available propargylic alcohols. Various secondary propargylic alcohols or tertiary propargylic alcohols participated well in the reaction, providing the desired products in good yields. This method provides a flexible and rapid route to substituted thiazoles.

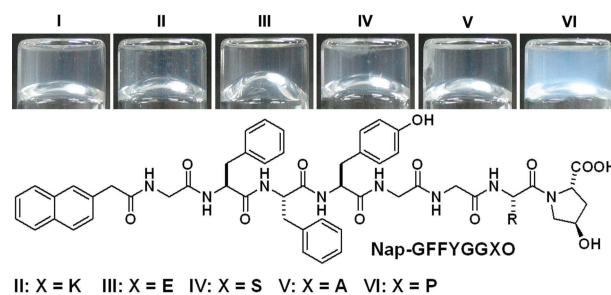


3267

Supramolecular hydrogels inspired by collagen for tissue engineering

Yuehan Hu, Huaimin Wang, Jingyu Wang, Sibing Wang, Wang Liao, Yonggang Yang, Yongjun Zhang, Deling Kong* and Zhimou Yang*

A small library of small molecules inspired by collagen were designed and synthesized, whose possibility for cell culture in 2D environments was studied in detail.



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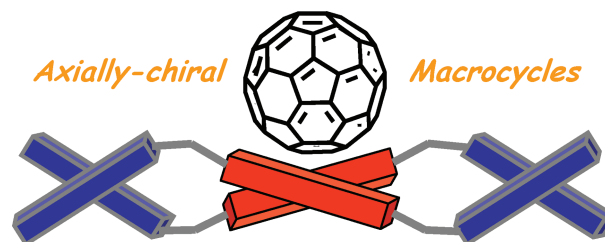
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3272

Nesting complexation of C₆₀ with large, rigid D₂ symmetrical macrocycles

Marco Caricato, Carmine Coluccini, Daniele Dondi, Douglas A. Vander Griend and Dario Pasini*

Chiral macrocycles, incorporating enantiopure binaphthyl units and rigid, conjugated spacing moieties, possess the right ensemble of suitable functionalities for the formation of nesting complexes with C₆₀.

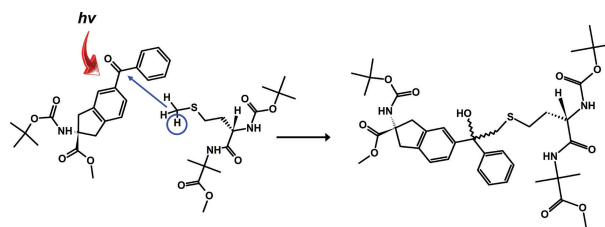


3281

A new tool for photoaffinity labeling studies: a partially constrained, benzophenone based, α -amino acid

Karen Wright,* Alessandro Moretto, Marco Crisma, Michel Wakselman, Jean-Paul Mazaleyrat, Fernando Formaggio and Claudio Toniolo*

The novel α -amino acid BpAib, a partially conformationally constrained analogue of the 3-(4-benzoylphenyl)alanine (Bpa) photoaffinity label, was synthesized, optically resolved and fully characterized. An intermolecular photocrosslinking experiment highlighted its regioselective reactivity, which is closely comparable to that of Bpa.

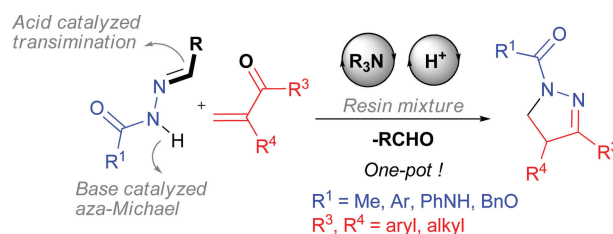


3287

Synthesis of pyrazolines by a site isolated resin-bound reagents methodology

Vincent Gembus, Jean-Jacques Bonnet, François Janin, Pierre Bohn, Vincent Levacher and Jean-François Brière*

Toward the elaboration of biologically important 3,4-substituted pyrazolines, an organocatalyzed aza-Michael/transimination domino sequence between hydrazones and enones was achieved by a mixture of heterogeneous resin-bound acid/base reagents, allowing the simultaneous use of otherwise destructive reactive functionalities using the site isolation concept.

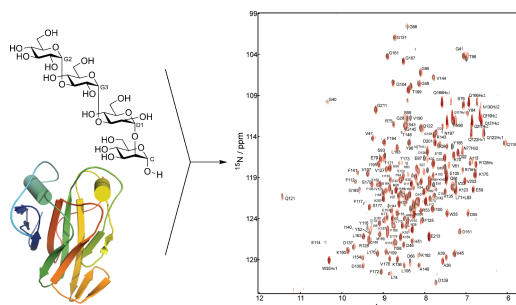


3294

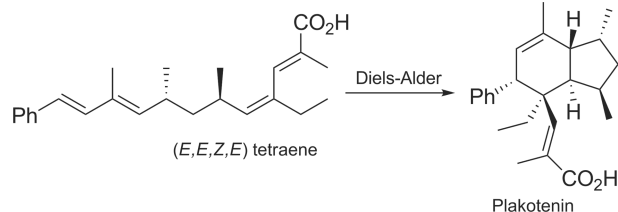
The Glc₂Man₂-fragment of the N-glycan precursor – a novel ligand for the glycan-binding protein *malectin*?

Lisa N. Müller, Claudia Muhle-Goll and Moritz B. Biskup*

The G2-G3-D1-C region of the tetrasaccharidic N-glycan precursor was synthesized and its interaction with the carbohydrate binding protein *malectin* was investigated. The protein *malectin* is involved in early stage processing of N-glycosylated proteins.



3300

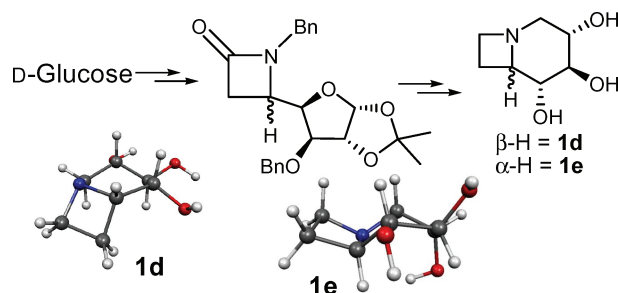


Enantioselective total synthesis of plakotenin, a cytotoxic metabolite from *Plakortis sp*

Stephanie Arzt, Emmanuel Bourcet, Thierry Muller and Stefan Bräse*

The first total enantioselective synthesis of plakotenin is described, proving the relative and absolute stereochemistry of natural plakotenin. A biomimetic intramolecular Diels–Alder reaction served as a key step in the total synthesis.

3307

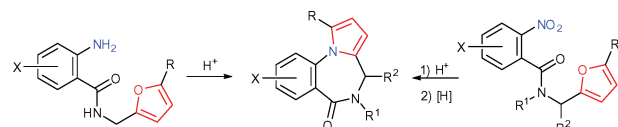


Synthesis, computational study and glycosidase inhibitory activity of polyhydroxylated conidine alkaloids—a bicyclic iminosugar

Shrihari P. Sanap, Sougata Ghosh, Amit M. Jabgunde, Rahul V. Pinjari, Shridhar P. Gejji, Shailza Singh, Balu A. Chopade and Dilip D. Dhavale*

A short and efficient synthesis of two hitherto unreported conidine iminosugars, one of which is selective inhibitor of α -mannosidase, and their conformational study by ^1H NMR, DFT calculations and molecular docking is described.

3316

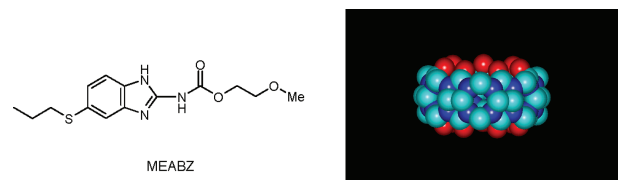


Furan ring opening–pyrrole ring closure: a new synthetic route to aryl(heteroaryl)-annulated pyrrolo[1,2-*a*][1,4]diazepines

Alexander V. Butin,* Tatyana A. Nevolina, Vitaly A. Shcherbinin, Igor V. Trushkov, Dmitry A. Cheshkov and Gennady D. Krapivin

Two new routes to pyrrolo[1,2-*a*][1,4]diazepines are proposed which are based on intramolecular furan cyclization.

3328



Enhanced cytotoxicity of benzimidazole carbamate derivatives and solubilisation by encapsulation in cucurbit[*n*]uril

Yunjie Zhao, Mohammad H. Pourgholami, David L. Morris, J. Grant Collins* and Anthony I. Day*

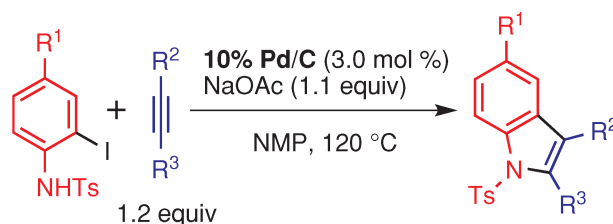
A new highly cytotoxic benzimidazole carbamate drug (MEABZ) has been synthesized, and encapsulation of the drug in the macrocycle cucurbit[*n*]uril (picture) significantly increased its water solubility.

3338

Palladium on carbon-catalyzed synthesis of 2- and 2,3-substituted indoles under heterogeneous conditions


Yasunari Monguchi, Shigeki Mori, Satoka Aoyagi, Azusa Tsutsui, Tomohiro Maegawa and Hironao Sajiki*

A mild, efficient and LiCl-free synthetic method for indole derivatives based on the heteroannulation of alkynes with 2-iodoanilines was achieved using palladium on carbon and NaOAc in heated NMP.



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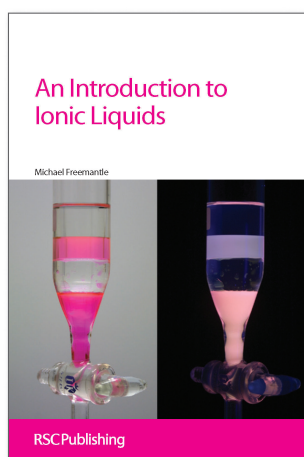
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