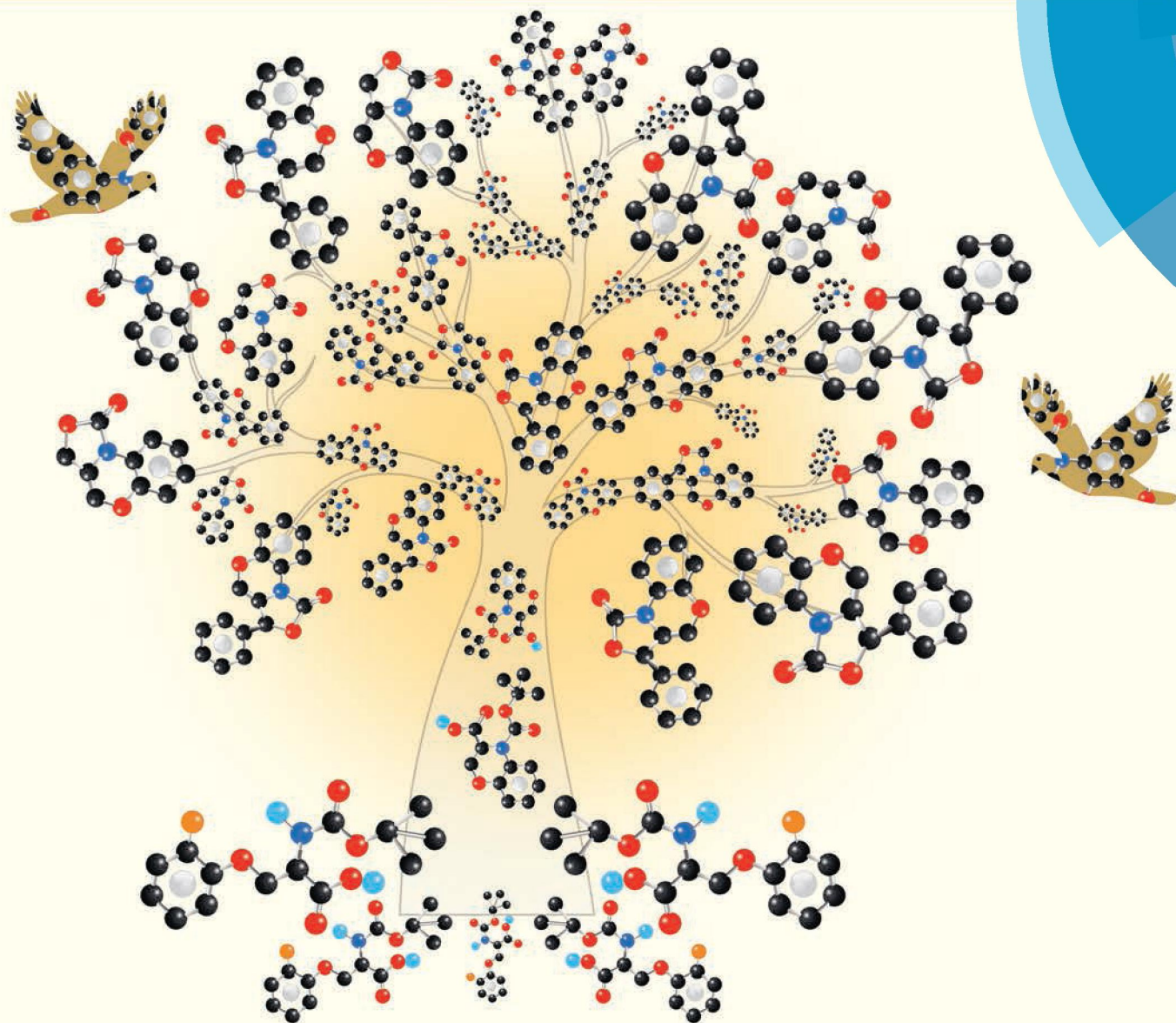


Organic & Biomolecular Chemistry

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ISSN 1477-0520



PAPER

Sourav Basu, Saumen Hajra *et al.*
Enantiopure synthesis of dihydrobenzo[1,4]-oxazine-3-carboxylic acids and
a route to benzoxazinyl oxazolidinones

Organic & Biomolecular Chemistry

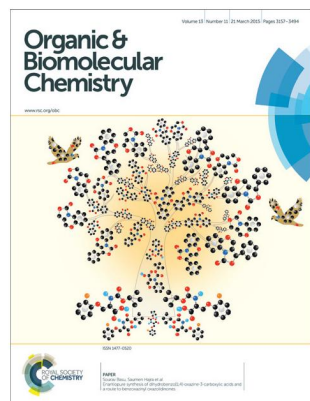
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ISSN 1477-0520 CODEN OBCRAK 13(11) 3157–3494 (2015)



Cover

See Rajesh Malhotra, Tushar K. Dey, Sourav Basu* and Saumen Hajra,* pp. 3211–3219.

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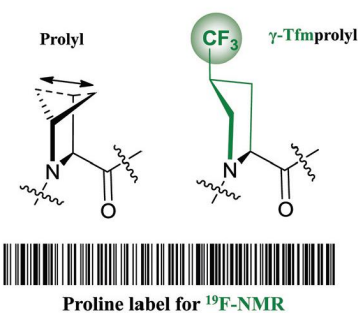
REVIEW

3171

γ -(S)-Trifluoromethyl proline: evaluation as a structural substitute of proline for solid state ^{19}F -NMR peptide studies

Vladimir Kubyskin, Sergii Afonin, Sezgin Kara, Nediljko Budisa, Pavel K. Mykhailiuk* and Anne S. Ulrich*

γ -(S)-Trifluoromethyl proline has been evaluated as a structural label for solid state ^{19}F -NMR studies of polypeptides.



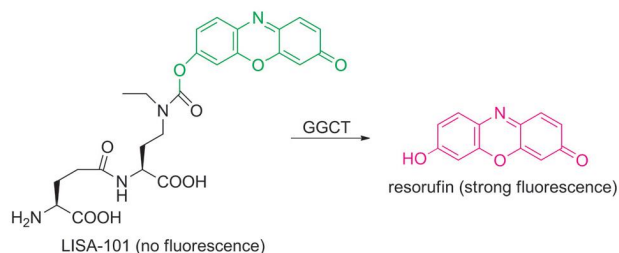
COMMUNICATIONS

3182

A GGCT fluorogenic probe: design, synthesis and application to cancer-related cells

Taku Yoshiya,* Hiromi Ii, Shugo Tsuda, Susumu Kageyama, Tatsuhiro Yoshiki and Yuji Nishiuchi*

Cancer-related γ -glutamyl cyclotransferase (GGCT) specifically decomposes γ -glutamyl amino acids. Here we report a novel GGCT fluorogenic probe "LISA-101".



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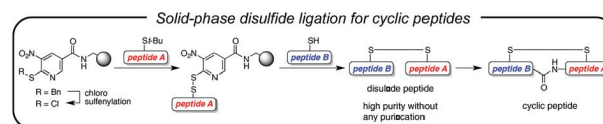
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3186

3-Nitro-2-pyridinesulfonyl-mediated solid-phase disulfide ligation in the synthesis of disulfide bond-containing cyclic peptides

Akihiro Taguchi, Kentarou Fukumoto, Yuya Asahina, Akihiro Kajiyama, Shunsuke Shimura, Keisuke Hamada, Kentaro Takayama, Fumika Yakushiji, Hironobu Hojo and Yoshio Hayashi*

A new solid-phase disulfide ligation method is developed to prepare a disulfide peptide from two types of Cys-containing peptide fragments with minimum purification steps.

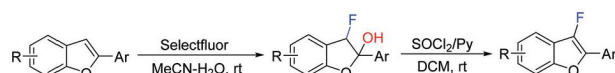


3190

Fluorination of 2-substituted benzo[*b*]furans with Selectfluor™

Mingliang Wang, Xixi Liu, Lu Zhou, Jidong Zhu* and Xun Sun*

An efficient protocol was developed to access 3-fluoro-2-hydroxy-2-substituted benzo[*b*]furans with Selectfluor™ as the fluorinating reagent in MeCN and water. By utilizing SOCl₂/Py as the dehydrating agent, the compounds above were readily converted to 3-fluorinated, 2-substituted benzo[*b*]furans in high yields.

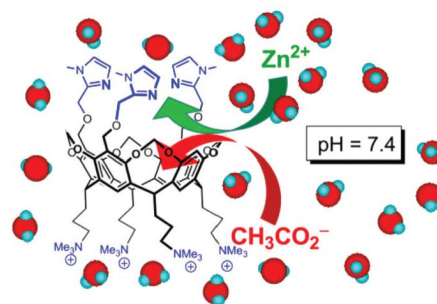


3194

The first water-soluble bowl complex: molecular recognition of acetate by the biomimetic tris(imidazole) Zn(II) system at pH 7.4

Stéphanie Rat, Jérôme Gout, Olivia Bistri and Olivia Renaud*

The bowl-shaped cavity-ligand based on resorcinarene has been successfully transposed into a water-soluble version that readily binds Zn(II) and acetate at physiological pH.

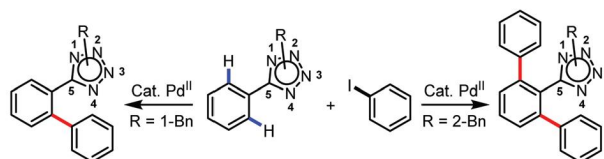


3198

Pd(II)-catalyzed, controllable C–H mono-/diarylation of aryl tetrazoles: concise synthesis of Losartan

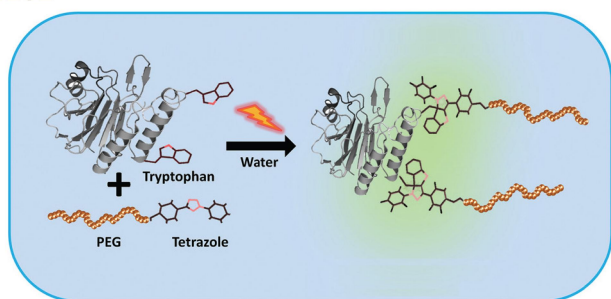
Yan-Jun Ding, Yan Li,* Sheng-Yu Dai, Quan Lan and Xi-Sheng Wang*

A palladium(II)-catalyzed C–H arylation directed by tetrazole has been developed with excellent mono-/di-selectivity through adjustment of the protecting site on the tetrazole ring.



COMMUNICATIONS

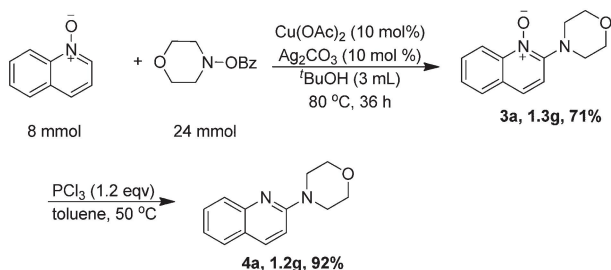
3202

**Photo-induced conjugation of tetrazoles to modified and native proteins**

Winna Siti, Amit Kumar Khan, Hans-Peter M. de Hoog, Bo Liedberg and Madhavan Nallani*

The direct addition of a water-soluble PEG-tetrazole to tryptophan residues in native proteins is demonstrated.

3207

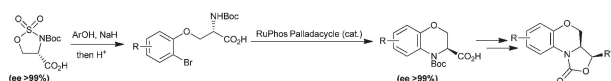
**Copper(II)-catalyzed electrophilic amination of quinoline *N*-oxides with *O*-benzoyl hydroxylamines**

Gang Li,* Chunqi Jia, Kai Sun, Yunhe Lv, Feng Zhao, Kexiao Zhou and Hankui Wu*

Copper acetate-catalyzed C–H bond functionalization amination of quinoline *N*-oxides was achieved using *O*-benzoyl hydroxylamine as an electrophilic amination reagent, thereby affording the desired products in moderate to excellent yields.

PAPERS

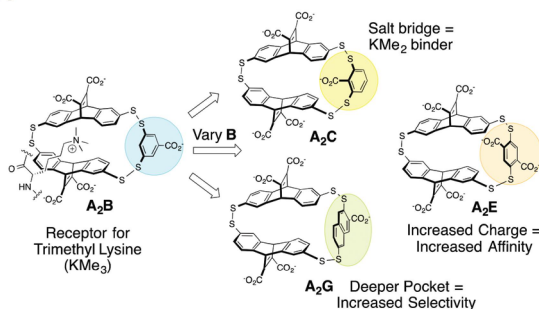
3211

**Enantiopure synthesis of dihydrobenzo[1,4]-oxazine-3-carboxylic acids and a route to benzoxazinyl oxazolidinones**

Rajesh Malhotra, Tushar K. Dey, Sourav Basu* and Saumen Hajra*

A two step protocol is developed for the enantiopure synthesis of dihydrobenzoxazine-3-carboxylic acids *via* RuPhos Palladacycle-catalyzed aminoarylation of β -(2-bromoaryloxy)amino acids.

3220

**Contributions of pocket depth and electrostatic interactions to affinity and selectivity of receptors for methylated lysine in water**

Joshua E. Beaver, Brendan C. Peacor, Julianne V. Bain, Lindsey I. James and Marcey L. Waters*

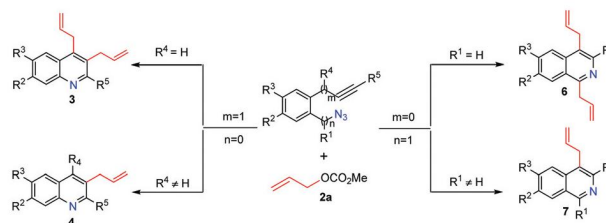
Investigation of charge and pocket depth in a series of receptors led to improved affinity and selectivity for trimethyllysine.

3227

Synthesis of allylated quinolines/isoquinolines via palladium-catalyzed cyclization–allylation of azides and allyl methyl carbonate

Jiang Luo, Zhibao Huo,* Jun Fu, Fangming Jin and Yoshinori Yamamoto

A novel and efficient strategy for one-step synthesis of allylated quinolines and isoquinolines via palladium-catalyzed cyclization–allylation of azides and allyl methyl carbonate is developed for the first time.

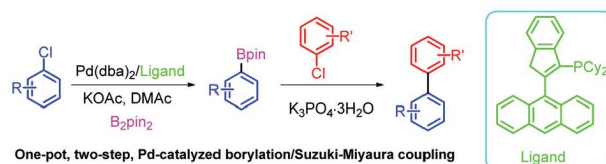


3236

Efficient phosphine ligands for the one-pot palladium-catalyzed borylation/Suzuki–Miyaura cross-coupling reaction

You Chen, Hui Peng, Yun-Xiao Pi, Tong Meng, Ze-Yu Lian, Meng-Qi Yan, Yan Liu, Sheng-Hua Liu and Guang-Ao Yu*

An air-stable 2-(anthracen-9-yl)-1*H*-inden-3-yl dicyclohexylphosphine was used in palladium-catalyzed borylation/Suzuki–Miyaura cross-coupling reaction.

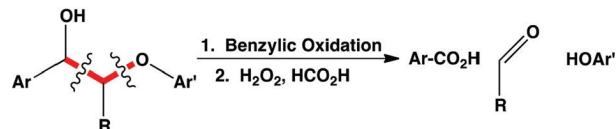


3243

Selective cleavage of the C α –C β linkage in lignin model compounds via Baeyer–Villiger oxidation

Nikhil D. Patil, Soledad G. Yao, Mark S. Meier,* Justin K. Mobley and Mark Crocker

Selective, catalytic oxidation of benzylic –OH groups followed by Baeyer–Villiger oxidation cleaves the β -O-4 linkage in lignin model compounds.

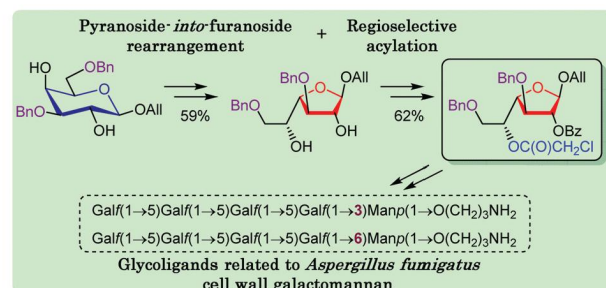


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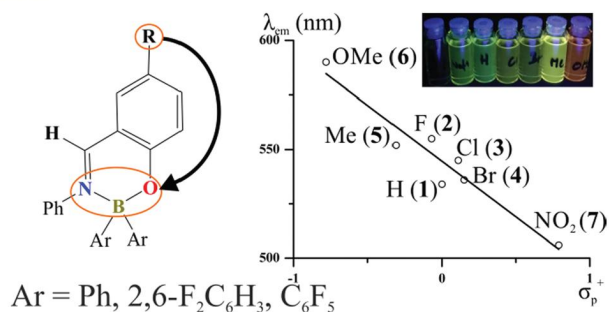
Convergent synthesis of isomeric heterosaccharides related to the fragments of galactomannan from *Aspergillus fumigatus*

D. A. Argunov, V. B. Krylov and N. E. Nifantiev*

The synthesis of heterosaccharide fragments of fungal galactomannan employing pyranoside-*into*-furanoside rearrangement.



3268

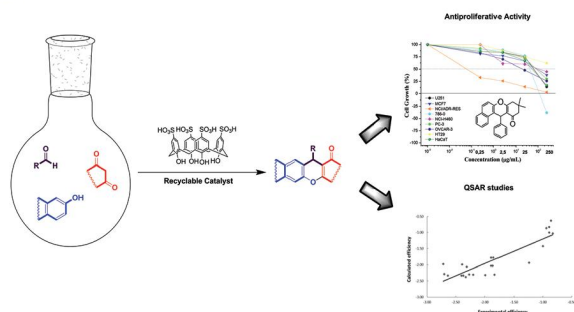


Tuning of the colour and chemical stability of model boronils: a strong effect of structural modifications

Grzegorz Wesela-Bauman,* Mateusz Urban, Sergiusz Luliński,* Janusz Serwatowski and Krzysztof Woźniak

An improved approach to luminescent diphenylborinic complexes with functionalized salicydeneaniline ligands was developed. A strong effect of structural modifications on their stability and optical properties was established.

3280

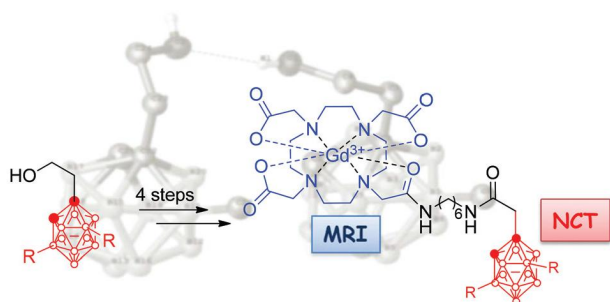


Xanthenones: calixarenes-catalyzed syntheses, anticancer activity and QSAR studies

Daniel Leite da Silva, Bruna Silva Terra, Mateus Ribeiro Lage, Ana Lúcia Tasca Góis Ruiz, Cameron Capeletti da Silva, João Ernesto de Carvalho, José Walkimar de Mesquita Carneiro, Felipe Terra Martins, Sergio Antonio Fernandes and Ângelo de Fátima*

An efficient method is proposed for obtaining tetrahydrobenzo[a]xanthene-11-ones and tetrahydro-[1,3]-dioxolo[4,5-*b*]xanthen-9-ones.

3288

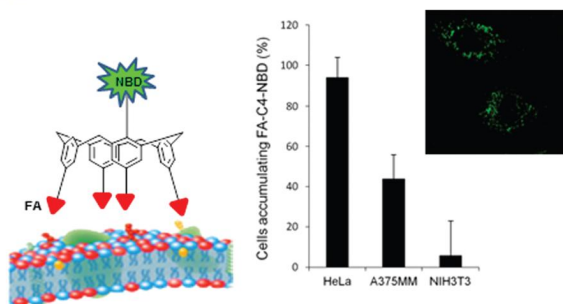


The hydroboration reaction as a key for a straightforward synthesis of new MRI-NCT agents

Paolo Boggio, Antonio Toppino, Simonetta Geninatti Crich, Diego Alberti, Domenica Marabello, Claudio Medana, Cristina Prandi, Paolo Venturello, Silvio Aime and Annamaria Deagostino*

A new lipophilic NCT/MRI agent has been synthesised in only four steps and characterised from the relaxometric point of view. This compound shows a high affinity for LDLs that can be loaded with 300 complexes per particle.

3298



Design and synthesis of a multivalent fluorescent folate-calix[4]arene conjugate: cancer cell penetration and intracellular localization

Grazia Maria Letizia Consoli,* Giuseppe Granata, Giorgia Fragassi, Mauro Grossi, Michele Sallèse* and Corrada Geraci

Fluorescent multivalent folate-calix[4]arene-NBD selectively penetrates cancer cells *via* folate receptor-mediated endocytosis and localizes in endo-lysosomes.

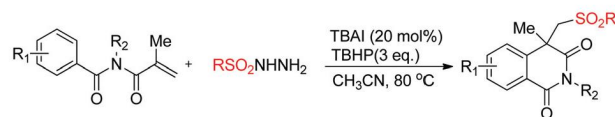
PAPERS

3308

Metal-free TBAI-catalyzed arylsulfonylation of activated alkenes with sulfonylhydrazides

Wubin Yu, Peizhu Hu, Yuanyuan Fan, Congyao Yu, Xinhuan Yan, Xiaoqing Li* and Xiangsheng Xu*

An efficient approach to isoquinoline-1,3(2*H*,4*H*)-dione derivatives through metal-free oxidative arylsulfonylation of α,β -unsaturated imides with sulfonylhydrazides is developed.

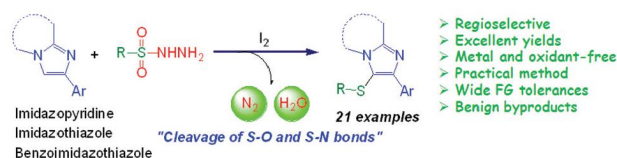


3314

Iodine-catalyzed regioselective thiolation of imidazo[1,2-*a*]pyridines using sulfonyl hydrazides as a thiol surrogate

Avik Kumar Bagdi, Shubhanjan Mitra, Monoranjan Ghosh and Alakananda Hajra*

Iodine-catalyzed regioselective sulfenylation of imidazo[1,2-*a*]pyridines via C(sp²)-H bond functionalization has been achieved using sulfonyl hydrazides as a thiol surrogate.

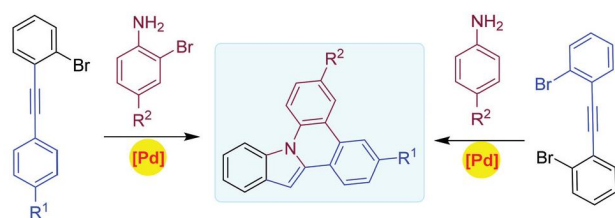


3321

Synthesis of indolo[1,2-*f*]phenanthridines by Pd-catalyzed domino C–N coupling/hydroamination/C–H arylation reactions

Thang Ngoc Ngo, Peter Ehlers, Tuan Thanh Dang, Alexander Villinger and Peter Langer*

Indolo[1,2-*f*]phenanthridines were prepared by Pd-catalyzed domino C–N coupling/hydroamination/C–H arylation reactions.

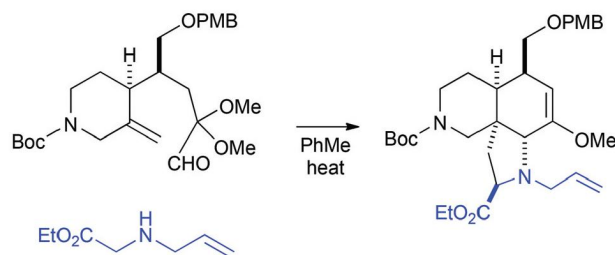


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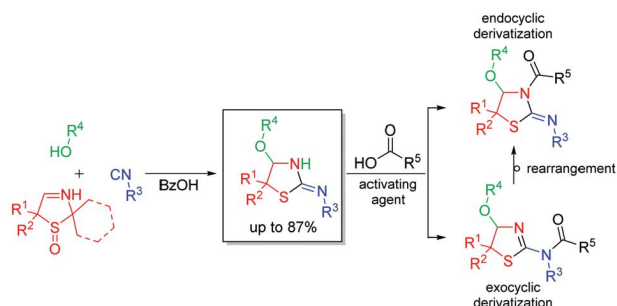
Synthesis of the tricyclic core of manzamine A

Ravindra B. Pathak, Benjamin C. Dobson, Nandita Ghosh, Khalid A. Ageel, Madeha R. Alshawish, Rungroj Saruengkhaphasit and Iain Coldham*

An approach to the ABC tricyclic ring system of the manzamine alkaloids has been achieved. A key step is the intramolecular dipolar cycloaddition of an azomethine ylide.



3341

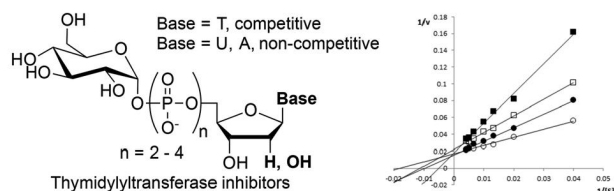


A new multicomponent reaction: unexpected formation of derivatizable cyclic α -alkoxy isothioureas

Fabian Brockmeyer, Valentin Morosow and Jürgen Martens*

With the aid of an unexpected new multicomponent reaction 2,5-dihydro-1,3-thiazole *S*-monoxides can be converted to cyclic α -alkoxy isothioureas. In addition, an observed rearrangement of subsequently acylated isothioureas is described.

3347

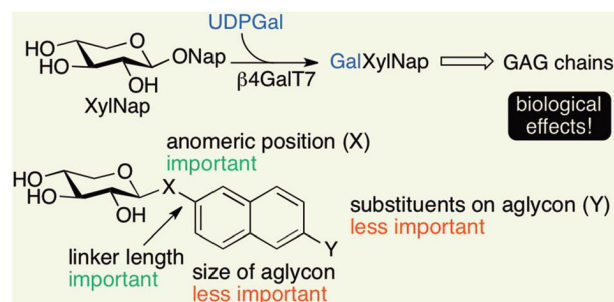


Polyphosphate-containing bisubstrate analogues as inhibitors of a bacterial cell wall thymidyltransferase

Deborah A. Smithen, Stephanie M. Forget, Nicole E. McCormick, Raymond T. Syvitski and David L. Jakeman*

The first synthesis and evaluation of bisubstrate analogues with a thymidyltransferase is reported. WaterLOGSY NMR and kinetic analyses provide insight into bisubstrate analogue binding.

3351

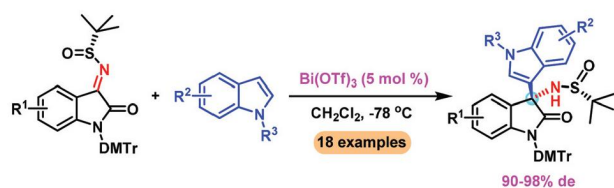


Exploration of the active site of β 4GalT7: modifications of the aglycon of aromatic xylosides

Anna Siegbahn, Karin Thorsheim, Jonas Ståhle, Sophie Manner, Christoffer Hamark, Andrea Persson, Emil Tykesson, Katrin Mani, Gunilla Westergren-Thorsson, Göran Widmalm and Ulf Ellervik*

β 4GalT7 is an essential enzyme in the biosynthesis of glycosaminoglycans. Modifications at the anomeric center of aromatic xylosides change the galactosylation efficiency significantly.

3363



A highly diastereoselective Friedel–Crafts reaction of indoles with isatin-derived *N*-sulfinyl ketimines towards the efficient synthesis of chiral tetrasubstituted 3-indolyl-3-aminooxindoles

Jian-Ping Chen, Wen-Wen Chen, Yi Li and Ming-Hua Xu*

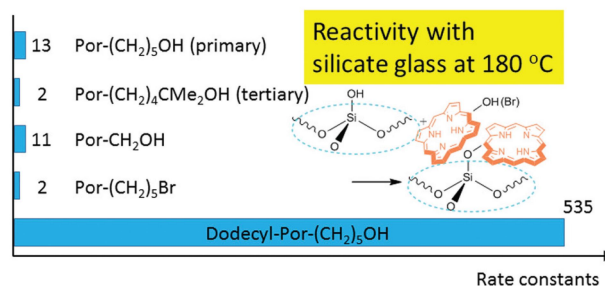
A Lewis acid promoted highly diastereoselective asymmetric Friedel–Crafts alkylation of indoles with isatin-derived *N*-*tert*-butanesulfinyl ketimines is described.

3371

Kinetics of reactions at an interface: functionalisation of silicate glass with porphyrins *via* covalent bonds

Takahiro Fujimoto, Nao Furuta and Tadashi Mizutani*

Both the reactivity of the linker and the dynamics of the interface controlled the anchoring reaction rates of porphyrin onto a silicate surface.

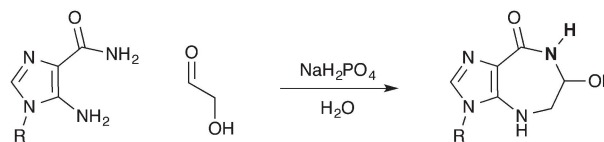


3378

One-step protecting-group-free synthesis of azepinomycin in water

Adam J. Coggins, Derek A. Tocher and Matthew W. Powner*

pH-dependent one-step multi-gram synthesis of azepinomycin in water.

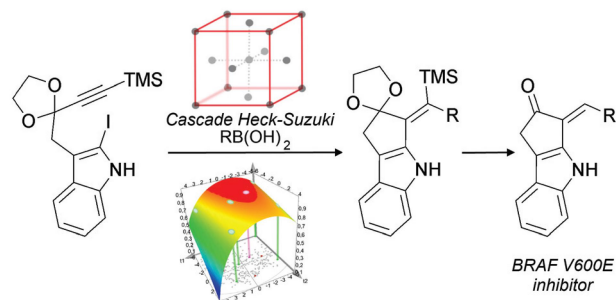


3382

Exploring a cascade Heck–Suzuki reaction based route to kinase inhibitors using design of experiments

Andreas Ekebergh, Christine Lingblom, Peter Sandin, Christine Wennerås and Jerker Mårtensson*

A fused tricyclic system with kinase inhibiting properties was assembled *via* a palladium catalyzed tandem reaction. The reaction was optimized using statistical experimental design.

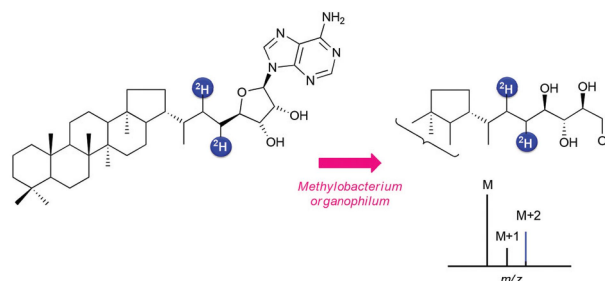


3393

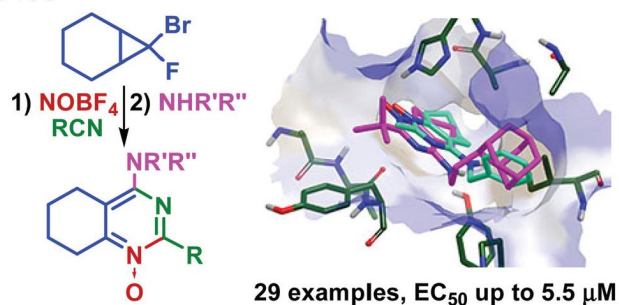
Hemisynthesis of deuteriated adenosylhopane and conversion into bacteriohopanetetrol by a cell-free system from *Methylobacterium organophilum*

Wenjun Liu, Anne Bodlenner and Michel Rohmer*

(²H₂)Adenosylhopane was synthesised *via* a reaction sequence including a cross metathesis followed by N₂²H₂ reduction of the resulting olefin.



3406

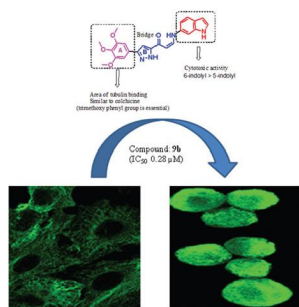


Synthesis and assessment of 4-aminotetrahydroquinazoline derivatives as tick-borne encephalitis virus reproduction inhibitors

K. N. Sedenkova, E. V. Dueva, E. B. Averina,* Y. K. Grishin, D. I. Osolodkin, L. I. Kozlovskaya, V. A. Palyulin, E. N. Savelyev, B. S. Orlinson, I. A. Novakov, G. M. Butov, T. S. Kuznetsova, G. G. Karganova* and N. S. Zefirov

A versatile synthesis of 4-aminopyrimidine *N*-oxides is developed and applied to obtain anti-TBEV agents.

3416

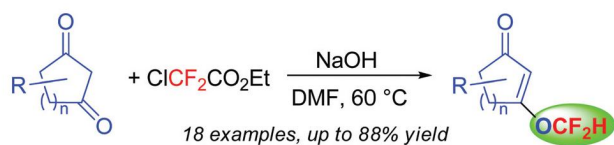


Synthesis of (Z)-(arylamino)-pyrazolyl/isoxazolyl-2-propanones as tubulin targeting anticancer agents and apoptotic inducers

Ahmed Kamal,* Vangala Santhosh Reddy, Anver Basha Shaik, G. Bharath Kumar, M. V. P. S. Vishnuvardhan, Sowjanya Polepalli and Nishant Jain

A new class of pyrazole conjugates were synthesized and evaluated for their antiproliferative activity in human cancer cell lines: **9a**, **9b** and **9f** significantly inhibited cell growth as well as tubulin polymerization.

3432

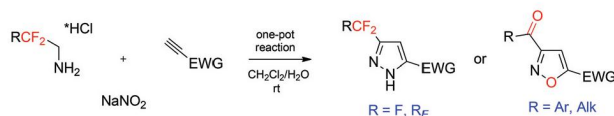


A new method for the synthesis of difluoromethyl enol ethers by *O*-difluoromethylation of 1,3-diones with $\text{ClCF}_2\text{CO}_2\text{Et}$

Xiaoxi Lin and Zhiqiang Weng*

A simple method for the synthesis of difluoromethyl enol ethers via *O*-difluoromethylation of 1,3-diones using inexpensive and commercially available $\text{ClCF}_2\text{CO}_2\text{Et}$ was developed.

3438



Three-component synthesis of fluorinated pyrazoles from fluoroalkylamines, NaNO_2 and electron-deficient alkynes

Pavel K. Mykhailiuk

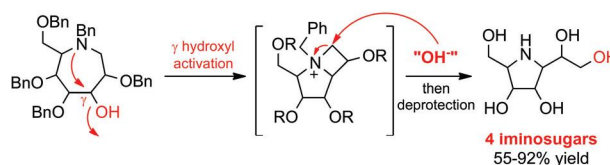
Three-component synthesis of fluorinated pyrazoles.

3446

γ -Aminoalcohol rearrangement applied to pentahydroxylated azepanes provides pyrrolidines epimeric to homoDMDP

Y. Jagadeesh, A. T. Tran, B. Luo, N. Auberger, J. Désiré, S. Nakagawa, A. Kato, Y. Zhang, M. Sollogoub and Y. Blériot*

Exploiting a γ -aminoalcohol rearrangement, pentahydroxylated azepanes are converted to pyrrolidines that are epimeric to homoDMDP, a potent glycosidase inhibitor.

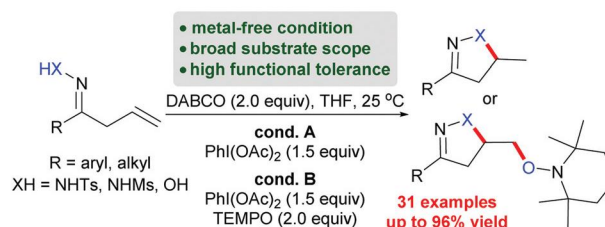


3457

PhI(OAc)₂-mediated functionalisation of unactivated alkenes for the synthesis of pyrazoline and isoxazoline derivatives

Xiao-Qiang Hu, Guoqiang Feng, Jia-Rong Chen,* Dong-Mei Yan, Quan-Qing Zhao, Qiang Wei and Wen-Jing Xiao*

A PhI(OAc)₂-promoted radical cyclization of β,γ -unsaturated hydrazones and oximes has been developed for an efficient synthesis of pyrazolines and isoxazolines.

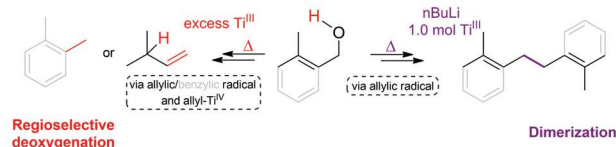


3462

Homocoupling versus reduction of radicals: an experimental and theoretical study of Ti(III)-mediated deoxygenation of activated alcohols

Consuelo Prieto, José A. González Delgado, Jesús F. Arteaga,* Martín Jaraíz,* José L. López-Pérez and Alejandro F. Barrero

A detailed study corroborates that the Ti(III)-mediated reductive deoxygenation of activated -OH proceeds via an allyl(benzyl)-radical and allyl(benzyl)-Ti, which is protonated, regioselectively in the case of allylic derivatives.

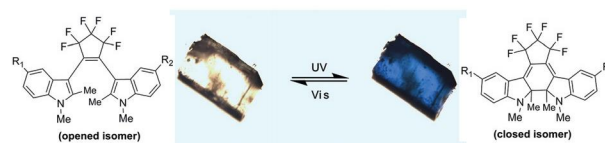


3470

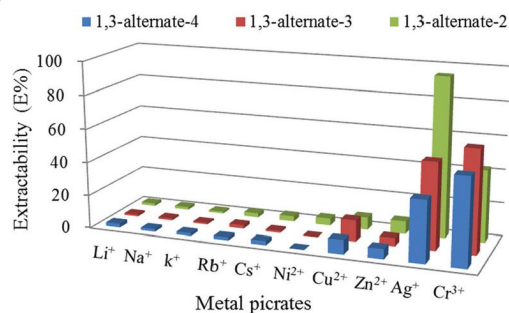
A prominent bathochromic shift effect of indole-containing diarylethene derivatives

Hong-Bo Cheng, Yao-Dong Huang, Lina Zhao, Xu Li and Hai-Chen Wu*

Bisindole diarylethene derivatives exhibit a strong bathochromic shift effect in photochromism compared with bisbenzothiophene diarylethene.



3476

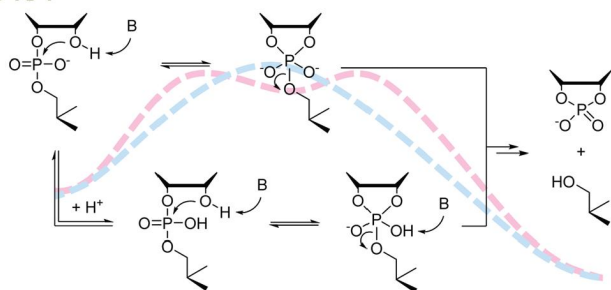


The first study about the relationship between the extractability of thiacalix[4]arene derivatives and the position of the coordination binding sites

Jiang-Lin Zhao, Hirotsugu Tomiyasu, Xin-Long Ni, Xi Zeng, Mark R. J. Elsegood, Carl Redshaw, Shofiur Rahman, Paris E. Georghiou, Simon J. Teat and Takehiko Yamato*

The extractability of thiacalix[4]arene derivatives **2–4** are largely dependent on the position of the binding sites.

3484



Buffer catalyzed cleavage of uridylyl-3',5'-uridine in aqueous DMSO: comparison to its activated analog, 2-hydroxypropyl 4-nitrophenyl phosphate

L. Lain, H. Lönnberg and T. A. Lönnberg*

In 80% aq. DMSO, buffer catalysis of the cleavage of UpU is significant but weaker than with HPNP.