

# Organic & Biomolecular Chemistry

An international journal of synthetic, physical and biomolecular organic chemistry

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## IN THIS ISSUE

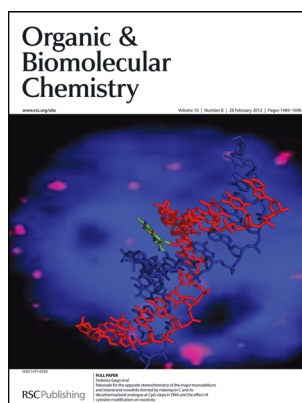
ISSN 1477-0520 CODEN OBCRAK 10(8) 1469–1696 (2012)



### Cover

See Joan G. Schellinger *et al.*, pp. 1521–1526.

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### Inside cover

See Juan A. Bueren-Calabuig *et al.*, pp. 1543–1552.

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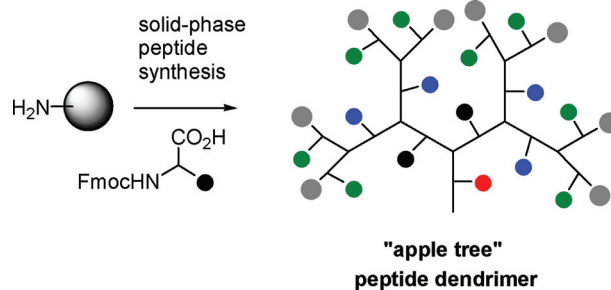
## PERSPECTIVE

1483

### Peptide and glycopeptide dendrimer apple trees as enzyme models and for biomedical applications

Jean-Louis Reymond\* and Tamis Darbre

Peptide dendrimers were selected from combinatorial libraries as esterase and aldolase models, drug delivery and antimicrobial agents, ligands for lectins, metals and vitamin B<sub>12</sub>.



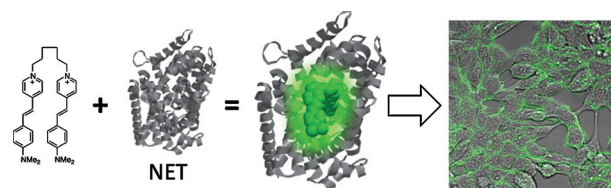
## COMMUNICATIONS

1493

### Probing the functional limits of the norepinephrine transporter with self-reporting, fluorescent stilbazolium dimers

Erika L. Smith, Adrienne S. Brown, Edward Adjaye-Mensah and James N. Wilson\*

A series of stilbazolium dimers were synthesized and investigated as sterically demanding ligands targeting the norepinephrine transporter.



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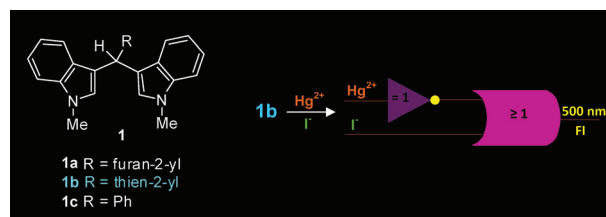
## COMMUNICATIONS

1497

**Bis(*N*-methylindolyl)methane-based chemical probes for Hg<sup>2+</sup> and Cu<sup>2+</sup> and molecular IMPLICATION gate operating in fluorescence mode**

Paramjit Kaur,\* Sandeep Kaur and Kamaljit Singh\*

Bis(*N*-methylindolyl)methane chemosensors bind Hg<sup>2+</sup> and Cu<sup>2+</sup> and signal absorption and emission changes which also correspond to rare IMPLICATION logic gate.

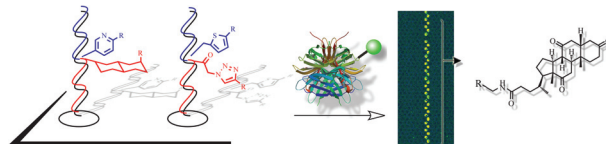


1502

**Discovery of an entropically-driven small molecule streptavidin binder from nucleic acid-encoded libraries**

Jean-Pierre Dagher, Mihai Ciobanu, Sofia Barluenga and Nicolas Winssinger\*

Dehydrocholic acid was identified as a selective streptavidin binder from a PNA-tagged library. Peptides tagged with dehydrocholic acid can be captured on a streptavidin resin and released under thermal conditions.

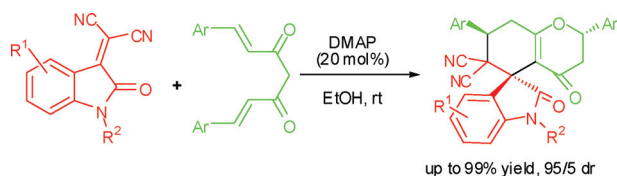


1506

**Efficient synthesis of multicyclic spirooxindoles via a cascade Michael/Michael/oxa-Michael reaction of curcumins and isatylidene malononitriles**

Xiao-Gang Yin, Xin-Yun Liu, Zhi-Peng Hu and Ming Yan\*

Cascade Michael/Michael/oxa-Michael reaction of curcumins and isatylidene malononitriles provided multicyclic spirooxindoles in excellent yields and diastereoselectivities.

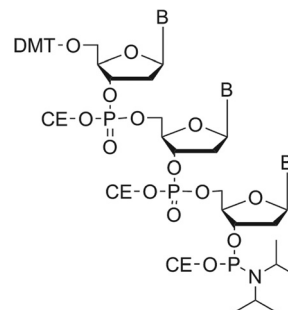


1510

**A new and convenient approach for the preparation of β-cyanoethyl protected trinucleotide phosphoramidites**

Matthäus Janczyk, Bettina Appel, Danilo Springstube, Hans-Joachim Fritz and Sabine Müller\*

A convenient approach for the preparation of fully protected trinucleotide synthons to be used for the synthesis of gene libraries is reported.



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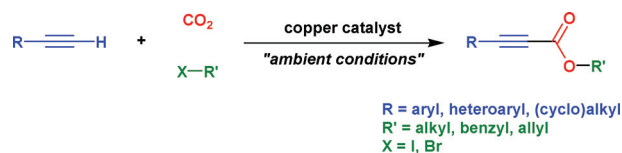
## COMMUNICATIONS

1514

**A copper-based catalytic system for carboxylation of terminal alkynes: synthesis of alkyl 2-alkynoates**

Kiyofumi Inamoto,\* Narumi Asano, Koji Kobayashi, Misato Yonemoto and Yoshinori Kondo\*

An efficient coupling of terminal alkynes and CO<sub>2</sub> in the presence of alkyl halides can be achieved under ambient conditions using a copper/phosphine catalyst system, providing facile access to a variety of functionalised alkyl 2-alkynoates.

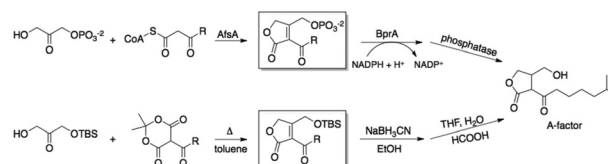


1517

**Replication of biosynthetic reactions enables efficient synthesis of A-factor, a  $\gamma$ -butyrolactone autoinducer from *Streptomyces griseus***

Jesse B. Morin, Katherine L. Adams and Jason K. Sello\*

We report a concise synthesis of A-factor, the prototypical  $\gamma$ -butyrolactone signalling compound of *Streptomyces* bacteria. In analogy to enzymatic reactions in A-factor biosynthesis, our synthesis features a tandem esterification–Knoevenagel condensation yielding a 2-acyl butenolide and a surprising, chemoselective conjugate reduction of this compound with sodium cyanoborohydride.



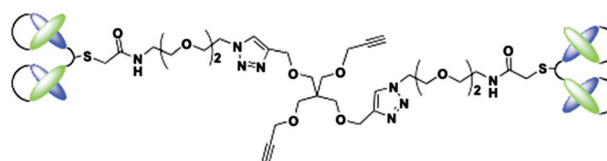
## PAPERS

1521

**A general chemical synthesis platform for crosslinking multivalent single chain variable fragments**

Joan G. Schellinger, Avinash Kudupudi, Arutselvan Natarajan, Wenjun Du, Sally J. DeNardo and Jacquelyn Gervay-Hague

A versatile chemical crosslinking strategy for the formation of multivalent scFv has been developed using CuAAC with high ligation yields and increased tumor binding affinity.

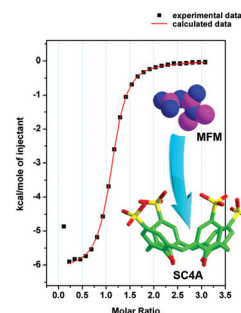


1527

**Thermodynamic origins of selective binding affinity between *p*-sulfonatocalix[4,5]arenes with biguanidiniums**

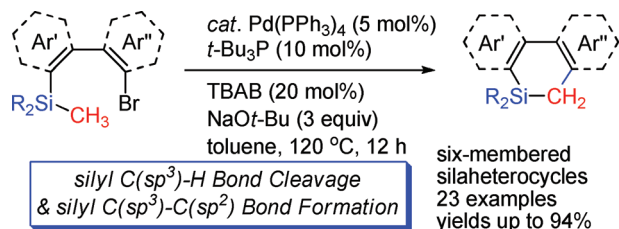
Dong-Sheng Guo, Hong-Qing Zhang, Fei Ding and Yu Liu\*

*p*-Sulfonatocalix[*n*]arenes show medium and strong binding abilities to biguanidinium guests with desired host/guest selectivities.



## PAPERS

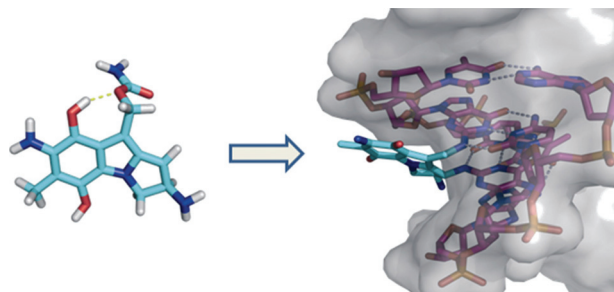
1537

**Palladium-catalyzed silyl C(sp<sup>3</sup>)-H bond activation**

Yun Liang, Weizhi Geng, Junnian Wei, Kunbing Ouyang and Zhenfeng Xi\*

The first transition-metal-catalyzed activation of silyl C(sp<sup>3</sup>)-H bond was realized and synthetically applied for the synthesis of six-membered silacycles. The adjacent Si atom played an essential role for the activation of the C(sp<sup>3</sup>)-H bond in the SiMe<sub>3</sub> group.

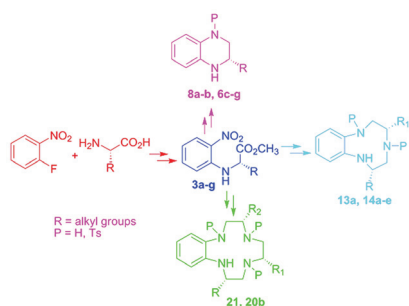
1543

**Rationale for the opposite stereochemistry of the major monoadducts and interstrand crosslinks formed by mitomycin C and its decarbamoylated analogue at CpG steps in DNA and the effect of cytosine modification on reactivity**

Juan A. Bueren-Calabuig, Ana Negri, Antonio Morreale and Federico Gago\*

Stepwise simulation of mitomycin C reactivity with DNA using molecular dynamics simulations and quantum mechanics.

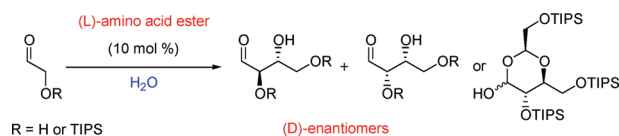
1553

**Inter- and intramolecular Mitsunobu reaction and metal complexation study: synthesis of *S*-amino acids derived chiral 1,2,3,4-tetrahydroquinoxaline, benzo-annulated [9]-N<sub>3</sub> peraza, [12]-N<sub>4</sub> peraza-macrocycles**

Krishnananda Samanta, Nitin Srivastava, Satyen Saha and Gautam Panda\*

Unsymmetrical chiral peraza-macrocycles were synthesized from an amino acid derived common synthetic intermediate.

1565

**Asymmetric organocatalytic formation of protected and unprotected tetroses under potentially prebiotic conditions**

Laurence Burroughs, Paul A. Clarke,\* Henrietta Forintos, James A. R. Gilks, Christopher J. Hayes,\* Matthew E. Vale, William Wade and Myriam Zbytniewski

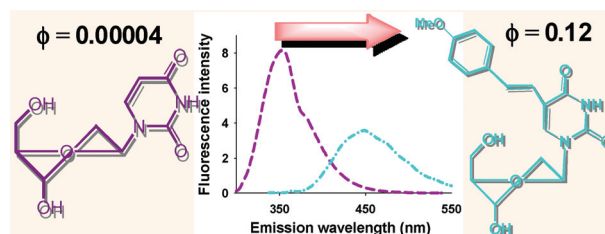
Esters of proteinogenic acyclic (L)-amino acids catalyse the formation of (D)-erythrose and (D)-threose under aqueous and potentially prebiotic conditions in the highest yields and enantioselectivities yet reported. This offers the potential to account for the link between natural (L)-amino acids and natural (D)-sugars.

1571

### Analogues of uracil nucleosides with intrinsic fluorescence (NIF-analogues): synthesis and photophysical properties

Meirav Segal and Bilha Fischer\*

5-((4-Methoxy-phenyl)-*trans*-vinyl)-2'-deoxy-uridine exhibits dramatically improved fluorescence compared to uridine, adopts the *anti* conformation and *S* sugar puckering favored by B-DNA, and is therefore suggested as a new diagnostic tool.

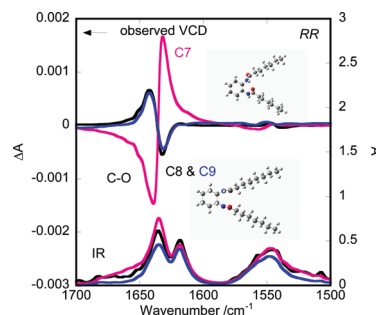


1581

### Critical effects of alkyl chain length on fibril structures in benzene-*trans*(*RR*)- or (*SS*)-*N,N'*-alkanoyl-1,2-diaminocyclohexane gels

Hisako Sato,\* Takahiro Nakae, Kazuya Morimoto and Kenji Tamura

Vibrational circular dichroism (VCD) spectra were recorded on the benzene-*d*<sup>6</sup> gels of *trans*(*RR*)- or *trans*(*SS*)-*N,N'*-alkanoyl-1,2-diaminocyclohexane. The signs of the coupled peaks assigned to the symmetric and asymmetric C=O stretching depended on the alkyl chain length critically.

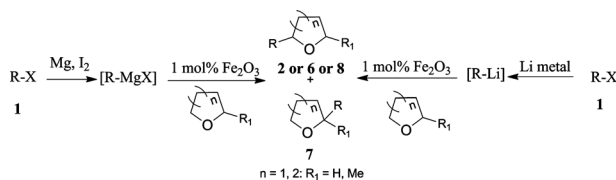


1587

### New method for C–H arylation/alkylation at $\alpha$ -position of cyclic aliphatic ethers by iron-oxide mediated reaction

Parvinder Pal Singh,\* Satish Gudup, Hariprasad Aruri, Umed Singh, Srinivas Ambala, Mahipal Yadav, Sanghapal D. Sawant and Ram A. Vishwakarma\*

Iron oxide mediated direct C–C bond formation without expensive or toxic ligands.

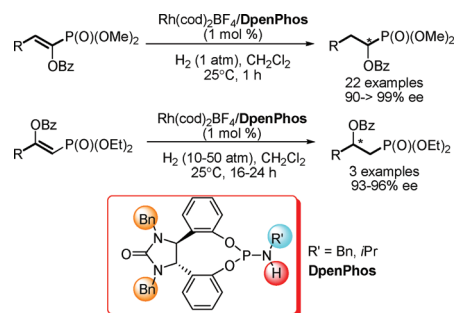


1598

### Asymmetric hydrogenation of $\alpha$ - or $\beta$ -acyloxy $\alpha,\beta$ -unsaturated phosphonates catalyzed by a Rh(I) complex of monodentate phosphoramidite

Jinzhu Zhang, Kaiwu Dong, Zheng Wang and Kuiling Ding\*

The enantioselective hydrogenation of  $\alpha$ - or  $\beta$ -acyloxy  $\alpha,\beta$ -unsaturated phosphonates, was realized under the catalysis of a Rh(I) complex of a monodentate phosphoramidite bearing a primary amine moiety (DpenPhos), affording the corresponding chiral  $\alpha$ - or  $\beta$ -hydroxy phosphonic acid derivatives with excellent enantioselectivity (90–99% ee).



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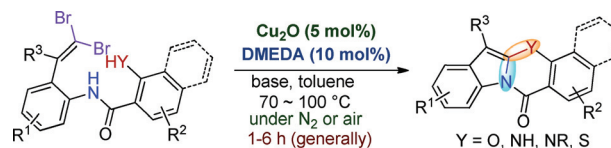


1602

### Copper-catalyzed domino intramolecular cyclization: a facile and efficient approach to polycyclic indole derivatives

Ziming Xia, Kuo Wang, Jiening Zheng, Zheyong Ma, Zhanguo Jiang, Xiaoxia Wang\* and Xin Lv\*

A mild and efficient Cu<sub>2</sub>O-catalyzed domino intramolecular C–N coupling/C–Y (Y = O, S, N) bond formation process has been developed. This has been applied to the assembly of novel fused heterocyclic indole derivatives.

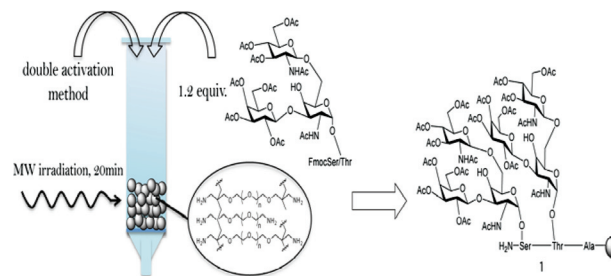


1612

### An efficient protocol for the solid-phase synthesis of glycopeptides under microwave irradiation

Fayna Garcia-Martin, Hiroshi Hinou, Takahiko Matsushita, Shun Hayakawa and Shin-Ichiro Nishimura\*

An efficient protocol for glycopeptides was established by a double coupling method under microwave irradiation.

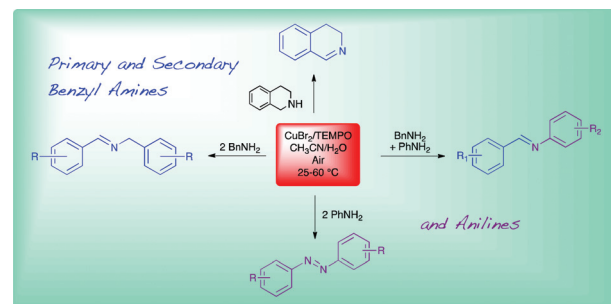


1618

### Simple copper/TEMPO catalyzed aerobic dehydrogenation of benzylic amines and anilines

Zhenzhong Hu and Francesca M. Kerton\*

CuBr<sub>2</sub> with 2,2,6,6-tetramethylpiperidyl-1-oxy (TEMPO) has been successfully employed for the aerobic oxidation of primary and secondary benzylic amines in aqueous acetonitrile.

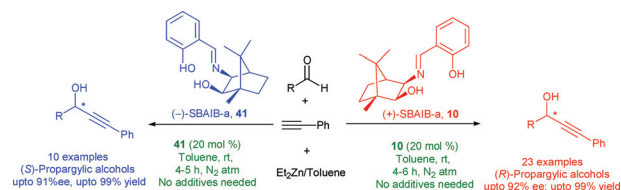


1625

### Camphor-based Schiff base ligand SBAIB: an enantioselective catalyst for addition of phenylacetylene to aldehydes

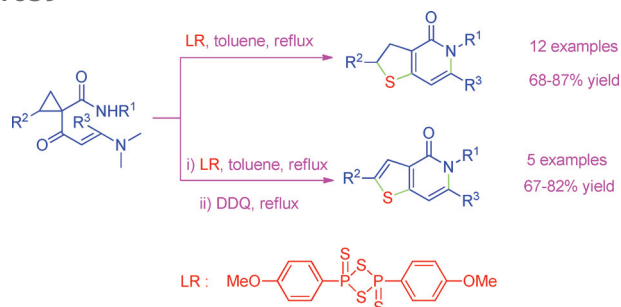
Ramalingam Boobalan, Chinpiao Chen\* and Gene-Hsian Lee

The synthesis of tridentate camphor-based Schiff base ligands (SBAIB) and their application in enantioselective phenylacetylene addition to aldehydes are described.



## PAPERS

1639

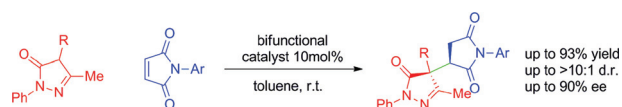


### Lawesson's reagent-initiated domino reaction of aminopropenoyl cyclopropanes: synthesis of thieno[3,2-c]pyridinones

Peng Huang, Rui Zhang, Yongjiu Liang and Dwen Dong\*

Domino reactions of dimethylaminopropenoyl cyclopropanes initiated by Lawesson's reagent provides one-pot access to 2,3-dihydrothieno[3,2-c]pyridin-4(5*H*)-ones and thieno[3,2-c]pyridin-4(5*H*)-ones.

1645

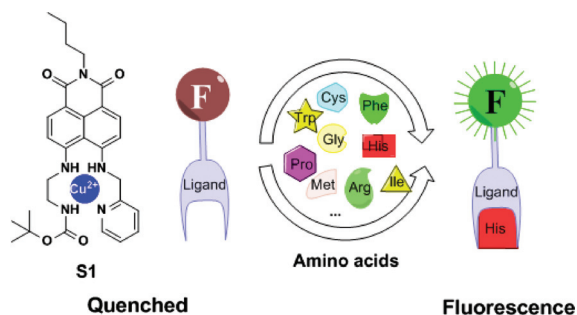


### Organocatalytic enantioselective pyrazol-3-one addition to maleimides: Reactivity and stereochemical course

Andrea Mazzanti, Teresa Calbet, Merce Font-Bardia, Albert Moyano and Ramon Rios\*

First asymmetric addition of pyrazolones to maleimides catalyzed by bifunctional thiourea catalysts.

1653

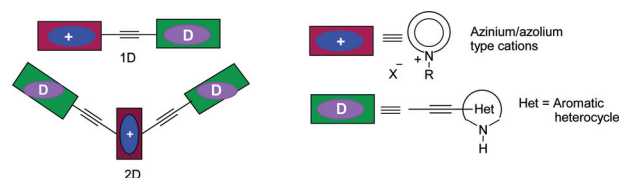


### Highly selective, naked-eye and fluorescent “off-on” probe for detection of histidine/histidine-rich proteins and its application in living cell imaging

Shenyi Zhang, Chunmei Yang, Weiping Zhu, Bubing Zeng, Youjun Yang, Yufang Xu\* and Xuhong Qian\*

A novel fluorescent probe (S1) for the colorimetric and switch-on fluorescent detection of histidine and histidine-rich proteins was designed and synthesized. The probe S1 shows good selectivity for histidine over other  $\alpha$ -amino acids, and can be used for histidine detection and imaging in living cells.

1659



### Donor-( $\pi$ -bridge)-azinium as $D-\pi-A^+$ one-dimensional and $D-\pi-A^+-\pi-D$ multidimensional V-shaped chromophores

Marco Antonio Ramírez, Ana M. Cuadro,\* Julio Alvarez-Builla, Obis Castaño, Jose L. Andrés, Francisco Mendicuti, Koen Clays, Inge Asselberghs and Juan J. Vaquero\*

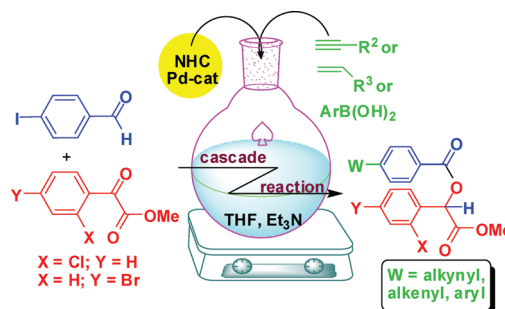
Linear 1D ( $D-\pi-A^+$ ) and 2D V-shaped ( $D-\pi-A^+-\pi-D$ ) charged chromophores were synthesized by Sonogashira reaction in good yields. The hyperpolarizabilities  $\beta$  of both chromophores were determined by hyper-Rayleigh scattering experiments and *ab-initio* quantum chemical methods.

1670

### *N*-heterocyclic carbene-mediated hydroacylation–Sonogashira/Heck/Suzuki coupling in a single pot: A new cascade reaction

M. Sreenivasulu, K. Siva Kumar, P. Rajender Kumar, K. B. Chandrasekhar and Manojit Pal\*

A dually NHC-catalyzed reaction cascade comprising an initial hydroacylation and subsequent Sonogashira/Heck/Suzuki coupling in the same pot is reported.

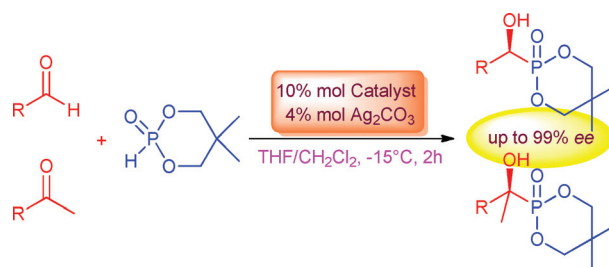


1680

### The asymmetric synthesis of chiral cyclic $\alpha$ -hydroxy phosphonates and quaternary cyclic $\alpha$ -hydroxy phosphonates

Chubei Wang, Chao Xu, Xiaosong Tan,\* Hao Peng and Hongwu He\*

An efficient method for the synthesis of chiral cyclic  $\alpha$ -hydroxy phosphonates and quaternary cyclic  $\alpha$ -hydroxy phosphonates was developed, affording the corresponding products in good yields with excellent enantioselectivity (up to 99% ee).

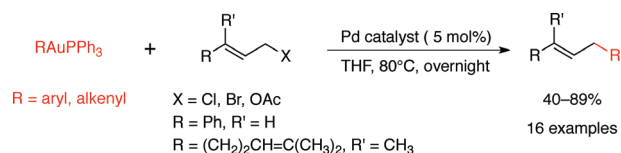


1686

### Palladium-catalyzed cross-coupling reactions of organogold(I) phosphanes with allylic electrophiles

Miguel Peña-López, Miguel Ayán-Varela, Luis A. Sarandeses\* and José Pérez Sestelo\*

Aryl and alkenylgold(I) phosphanes react regioselectively with allylic electrophiles under palladium catalysis in THF at  $80^\circ C$  to afford the  $\alpha$ -substitution product with moderate to high yields.



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