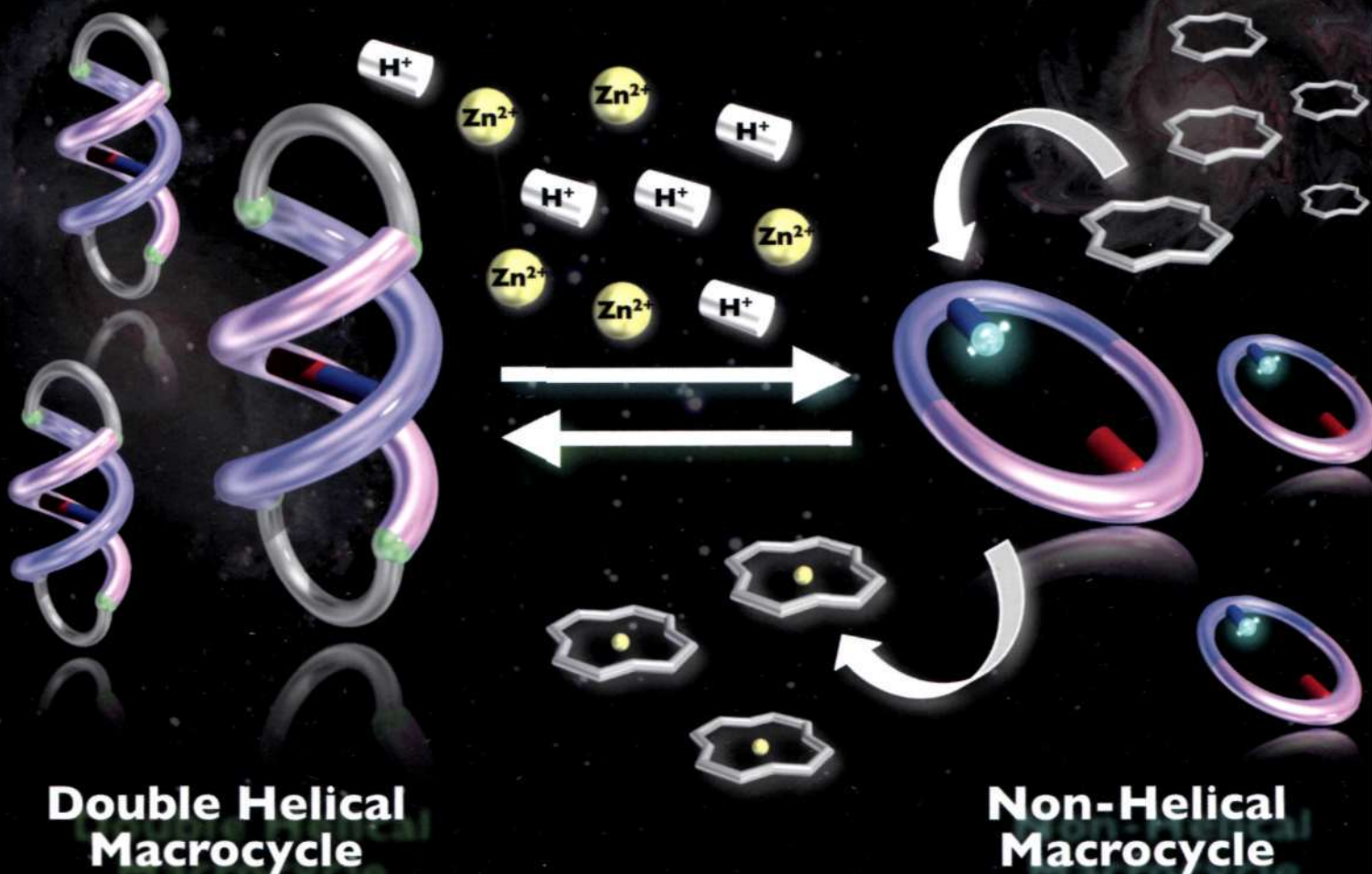


# Organic & Biomolecular Chemistry

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## Helically Twisted [1+1]Macrocycles



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PAPER

Eiji Yashima *et al.*

Synthesis of helically twisted [1 + 1]macrocycles assisted by amidinium-carboxylate salt bridges and control of their chiroptical properties



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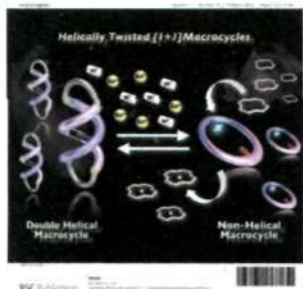
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### Organic & Biomolecular Chemistry



#### Cover

See Eiji Yashima *et al.*, pp. 1614–1623.

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### Organic & Biomolecular Chemistry



#### Inside cover

See Marie E. Krafft, Igor V. Alabugin *et al.*, pp. 1624–1630.

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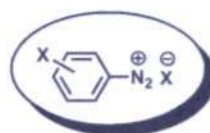
## PERSPECTIVE

1582

### Recent applications of arene diazonium salts in organic synthesis

Fanyang Mo, Guangbin Dong,\* Yan Zhang and Jianbo Wang\*

Recent advances involving arene diazonium salts as starting materials or active intermediates for various synthetically useful applications are summarized.



- ⇒ Nucleophilic substitutions
- ⇒ Radical reactions
- ⇒ Cross-coupling reactions

## COMMUNICATIONS

1594

### Ligations of *N*-acyl tryptophan units to give native peptides via 7-, 10-, 11- and 12-membered cyclic transition states

Vadim Popov, Siva S. Panda and Alan R. Katritzky\*

A series of *N*-acyl peptides containing terminal tryptophan residues has been synthesized and acyl migration of these *N*-acyl peptides via 7-, 10-, 11-, and 12-membered cyclic transition states has been studied.

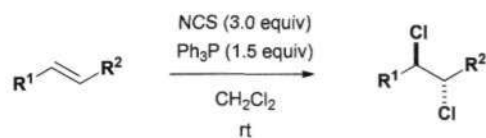


1598

**Dichlorination of olefins with NCS/Ph<sub>3</sub>P**

Yasumasa Kamada, Yuta Kitamura, Tetsuaki Tanaka and Takehiko Yoshimitsu\*

A 2 : 1 mixture of NCS and Ph<sub>3</sub>P successfully promoted the *anti*-dichlorination of olefins to provide corresponding dichlorides, serving as a molecular chlorine surrogate generated *in situ*.

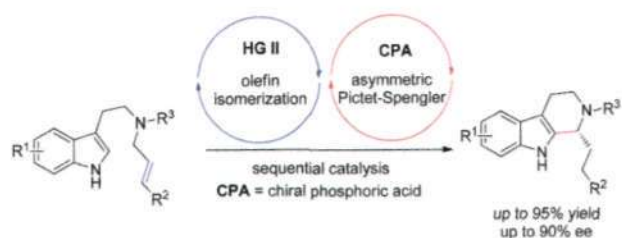


1602

**An olefin isomerization/asymmetric Pictet–Spengler cascade via sequential catalysis of ruthenium alkylidene and chiral phosphoric acid**

Quan Cai, Xiao-Wei Liang, Shou-Guo Wang and Shu-Li You\*

Chiral phosphoric acid works together with Hoveyda–Grubbs II catalyst enabling highly efficient synthesis of enantioenriched tetrahydro- $\beta$ -carboline (up to 95% yield, 90% ee) through an olefin isomerization/Pictet–Spengler cascade reaction *via* sequential catalysis.

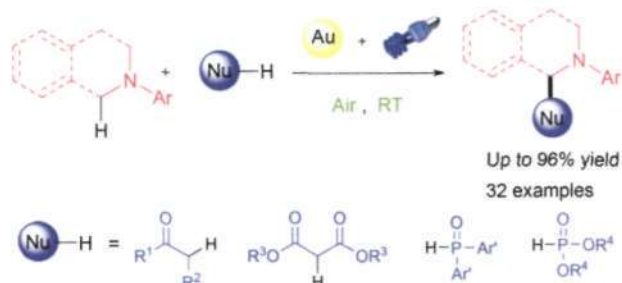


1606

**Highly efficient visible-light-induced aerobic oxidative C–C, C–P coupling from C–H bonds catalyzed by a gold(III)-complex**

Qicai Xue, Jin Xie, Hongming Jin, Yixiang Cheng and Chengjian Zhu\*

A novel and highly efficient visible-light-induced aerobic oxidative  $\alpha$ -C–H functionalization of amines catalyzed by a gold complex is reported.

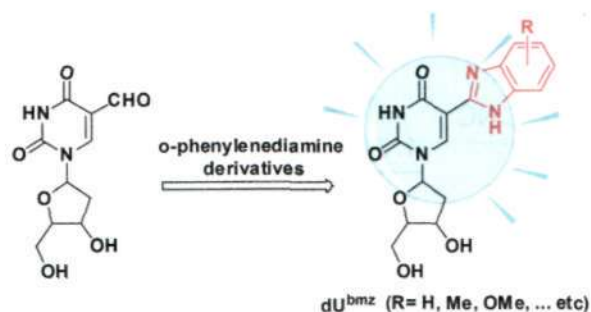


1610

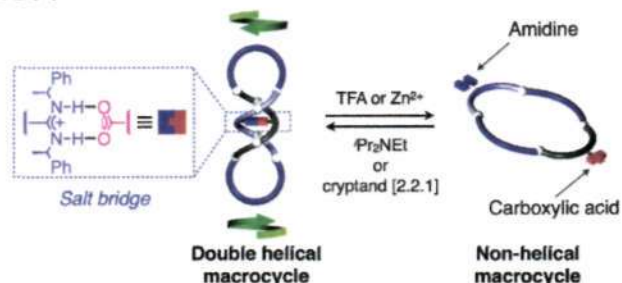
**Synthesis and spectroscopic properties of fluorescent 5-benzimidazolyl-2'-deoxyuridines 5-fdU probes obtained from *o*-phenylenediamine derivatives**

P. Guo, X. Xu, X. Qiu, Y. Zhou, S. Yan, C. Wang, C. Lu, W. Ma, X. Weng, X. Zhang and X. Zhou\*

*o*-Phenylenediamine was used for detecting 5-fdU among natural nucleosides to produce fluorescent nucleosides which are sensitive to pH and solvents.



1614

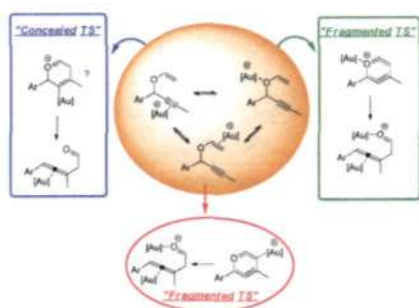


### Synthesis of helically twisted [1 + 1] macrocycles assisted by amidinium–carboxylate salt bridges and control of their chiroptical properties

Yuji Nakatani, Yoshio Furusho and Eiji Yashima\*

Novel twisted [1 + 1] macrocycles were prepared through amidinium–carboxylate salt bridges, whose chirality could be controlled by acid–base interactions and zinc coordination.

1624

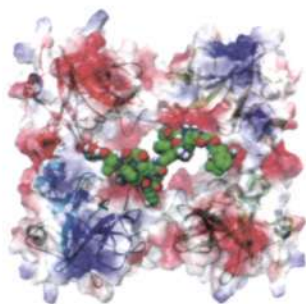


### Overriding the alkynophilicity of gold: catalytic pathways from higher energy Au(i)–substrate complexes and reactant deactivation via unproductive complexation in the gold(i)-catalyzed propargyl Claisen rearrangement

Dinesh V. Vidhani, John W. Cran, Marie E. Krafft\* and Igor V. Alabugin\*

The competition between catalytically important mechanistic pathways in the Au-promoted propargyl Claisen rearrangement depends on a variety of electronic effects unrelated to Au–alkyne coordination.

1631



### A new approach to inhibit human $\beta$ -tryptase by protein surface binding of four-armed peptide ligands with two different sets of arms

Qian-Qian Jiang, Lina Bartsch, Wilhelm Sicking, Peter R. Wich, Dominik Heider, Daniel Hoffmann and Carsten Schmuck\*

Four-armed peptide ligands with two different sets of arms containing an artificial arginine analog efficiently inhibit the enzyme by protein surface binding.

1640



### First diastereoselective [3 + 2] cycloaddition reaction of diethyl isocyanomethylphosphonate and maleimides

Carlos Arróniz, Juan Molina, Sonia Abás, Elies Molins, Josep M. Campanera, F. Javier Luque and Carmen Escolano\*

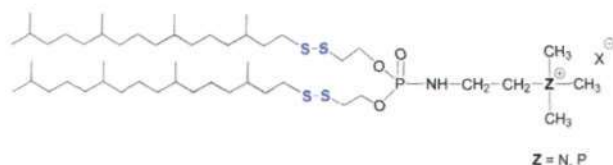
The first silver-catalysed [3 + 2] cycloaddition reaction of diethyl isocyanomethylphosphonate and *N*-substituted maleimides furnished bicyclic  $\alpha$ -iminophosphonates with complete diastereoselectivity.

1650

### Cationic lipophosphoramidates with two disulfide motifs: synthesis, behaviour in reductive media and gene transfection activity

Aurore Fraix, Tony Le Gall, Mathieu Berchel, Caroline Denis, Pierre Lehn, Tristan Montier\* and Paul-Alain Jaffrès\*

The synthesis of cationic lipids with two disulfide motifs in the hydrophobic domain is reported. They compact pDNA and release it rapidly in the presence of DTT as a reductive agent.



1659

### Cu-catalyzed *in situ* generation of thiol using xanthate as a thiol surrogate for the one-pot synthesis of benzothiazoles and benzothiophenes

D. J. C. Prasad and G. Sekar\*

A new copper-catalyzed *in situ* generation of aryl thiolates strategy was developed for the one-pot synthesis of substituted benzothiazoles from 2-iodoanilides using xanthate as thiol precursor. This one-pot protocol was successfully utilized for the synthesis of a potent antitumor agent PMX 610 and substituted benzothiophenes.

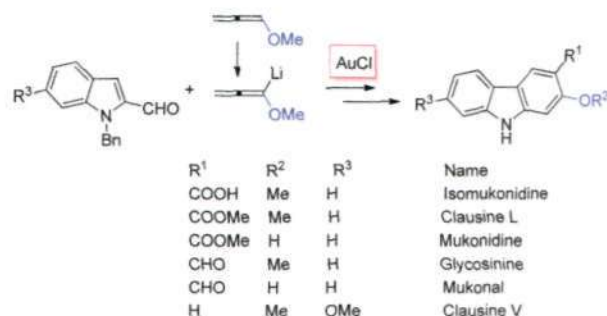


1666

### An efficient Au-catalyzed synthesis of isomukonidine, clausine L, mukonidine, glycosinine, mukonal, and clausine V from propadienyl methyl ether

Youai Qiu, Dengke Ma, Chunling Fu\* and Shengming Ma\*

Naturally occurring carbazole alkaloids such as isomukonidine, clausine-L, mukonidine, glycosinine, mukonal, and clausine V were synthesized through a gold-catalyzed cyclization reaction of 1-(indol-2-yl)-2-methoxy-2,3-allenols.

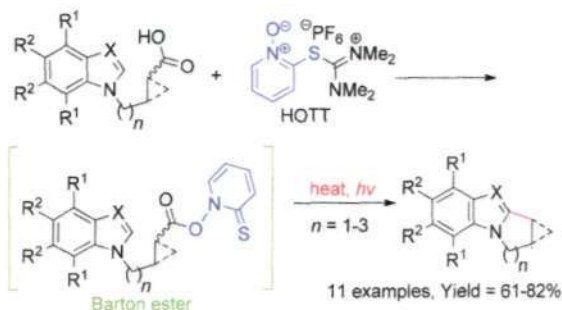


1672

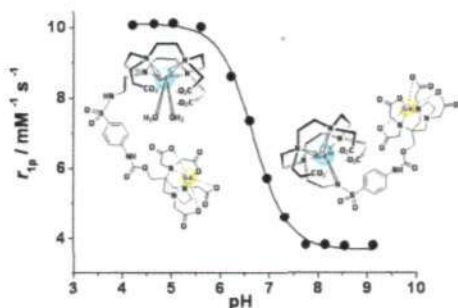
### Barton esters for initiator-free radical cyclisation with heteroaromatic substitution

Robert Coyle, Karen Fahey and Fawaz Aldabbagh\*

The first synthetically useful radical cyclisations with (hetero)aromatic substitution using Barton esters with reactions of cyclopropyl and alkyl radicals presented.



1683

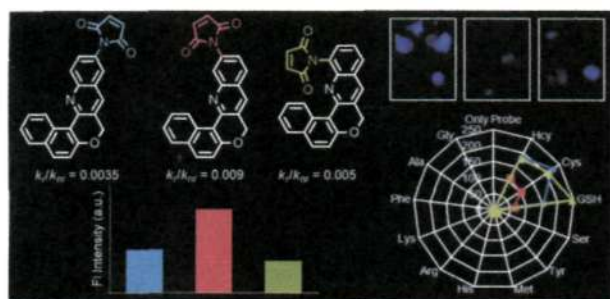


### Orthogonal synthesis of a heterodimeric ligand for the development of the Gd<sup>III</sup>–Ga<sup>III</sup> ditopic complex as a potential pH-sensitive MRI/PET probe

Nikolay Vologdin, Gabriele A. Rolla, Mauro Botta and Lorenzo Tei\*

A heterodimeric DO3A-sulfonamide–AAZTA ligand and its Gd<sup>III</sup>–Ga<sup>III</sup> heteroditopic complex were synthesised *via* an orthogonal route aiming to build a platform for the preparation of an efficient smart MRI/PET probe sensitive to pH.

1691

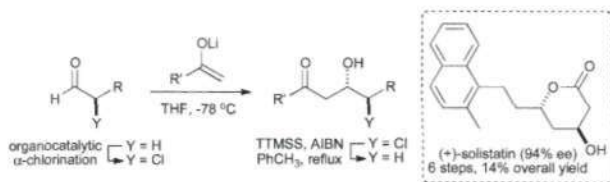


### Chromenoquinoline-based thiol probes: a study on the quencher position for controlling fluorescent Off–On characteristics

Dnyaneshwar Kand, Arunasree Marasanapalli Kalle and Pinaki Talukdar\*

A comparative study on chromenoquinoline-based fluorescent thiol probes is reported which features a higher Off–On response for hindered thiol exhibited by the probe with maleimide at the groove position of the fluorophore.

1702



### Chlorine, an atom economical auxiliary for asymmetric aldol reactions

Shira D. Halperin and Robert Britton\*

Asymmetric, auxiliary-controlled reactions of aldehydes have been developed that exploit a chlorine atom as a readily introduced and removed auxiliary.

1706



### Design and synthesis of stable $\alpha$ -diazo- $\beta$ -oxo sulfoxides

Stuart G. Collins,\* Orlagh C. M. O'Sullivan, Patrick G. Kelleher and Anita R. Maguire\*

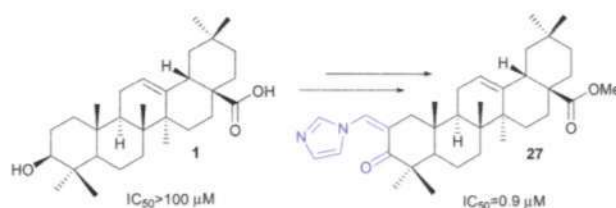
Diazo transfer adjacent to a sulfoxide moiety to provide stable, isolable  $\alpha$ -diazo- $\beta$ -oxo sulfoxides has been achieved. Use of monocyclic and bicyclic sulfoxide precursors is critical in enabling isolation of stable derivatives, through introduction of conformational constraint, while acyclic  $\alpha$ -diazo- $\beta$ -oxo sulfoxides are too labile to isolate.

1726

### Synthesis of novel heterocyclic oleanolic acid derivatives with improved antiproliferative activity in solid tumor cells

Ana S. Leal, Rui Wang, Jorge A. R. Salvador\* and Yongkui Jing\*

A series of new oleanolic acid derivatives with an imidazole ring in several positions of the oleanane backbone were synthesised, fully characterized and evaluated for their antiproliferative activity in AsPC-1 cells. Compound **27** was further tested in other cell lines.

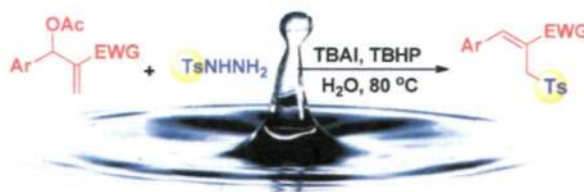


1739

### Tetrabutylammonium iodide catalyzed allylic sulfonation of Baylis–Hillman acetates with sulfonylhydrazides in water

Xiaoqing Li,\* Xiangsheng Xu\* and Yucai Tang

A tetrabutylammonium iodide catalyzed method for the synthesis of allyl aryl sulfone derivatives with Baylis–Hillman acetates and sulfonylhydrazides using *tert*-butyl hydroperoxide as an oxidation agent in water has been developed. In this process, the group eliminated from the sulfonyl precursor is molecular nitrogen.



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