

## In this issue...

Chemical Science – a ‘snapshot’ of the latest news and developments across the chemical sciences  
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## Cover

See Anja Hoffmann-Röder and Norbert Krause, pp. 387–391

**The Golden Gate to Catalysis** is opened by the use of gold compounds in homogenous catalysis. Due to their unique ability to activate carbon–carbon double and triple bonds, as well as C–H bonds, gold salts have emerged as highly active catalysts for the formation of C–C, C–O, C–N and C–S bonds. By using chiral allenes as substrates, gold catalysts can even be applied in stereoselective synthesis.

Front cover image provided by Norbert Krause. Photograph of Golden Gate Bridge supplied by permission of by David Robertson.

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## CHEMICAL SCIENCE

C9

Drawing together the research highlights and news from all RSC publications, *Chemical Science* provides a ‘snapshot’ of the latest developments across the chemical sciences showcasing newsworthy articles, as well as the most significant scientific advances.

# Chemical Science

February 2005/Volume 2/Issue 2

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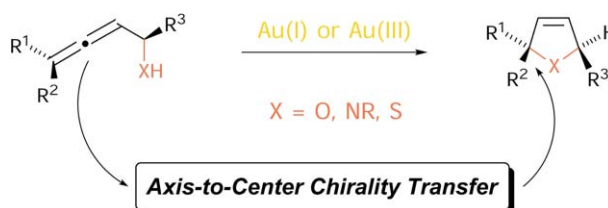
## EMERGING AREA

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## The golden gate to catalysis

Anja Hoffmann-Röder and Norbert Krause\*

Recent examples on the use of gold in homogeneous catalysis are summarised.



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Organic & Biomolecular Chemistry (print: ISSN 1477-0520; electronic: ISSN 1477-0539) is published 24 times a year by the Royal Society of Chemistry, Thomas Graham House, Science Park, Milton Road, Cambridge, UK CB4 0WF.

All orders, with cheques made payable to the Royal Society of Chemistry, should be sent to Portland Customer Services, Commerce Way, Colchester, Essex, CO2 8HP. Tel +44 (0) 1206 226050; E-mail sales@rscdistribution.org

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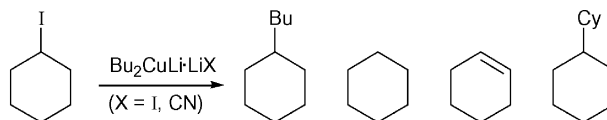
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**Subtle factors are important: radical formation and transmetallation in reactions of butyl cuprates with cyclohexyl iodide**

Steven H. Bertz,\* Jason Human, Craig A. Ogle\* and Paul Seagle

The reaction of butyl cuprates with CyI proceeds *via* radical and transmetallation pathways to a mixture of products, which depends upon subtle factors such as the surface properties of the reaction vessel, the kind of solvent still, and the lot of 'ultrapure' copper(i) salt.

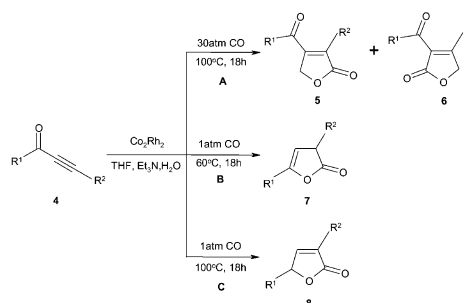


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**Cyclohydrocarbonylation of substituted alkynes and tandem cyclohydrocarbonylation–CO insertion of  $\alpha$ -keto alkynes catalyzed by immobilized Co–Rh heterobimetallic nanoparticles**

Kang Hyun Park, So Yeon Kim and Young Keun Chung\*

The use of cobalt–rhodium (Co<sub>2</sub>Rh<sub>2</sub>) heterobimetallic nanoparticles in the cyclohydrocarbonylation of substituted alkynes and tandem cyclohydrocarbonylation–CO insertion of  $\alpha$ -keto alkynes to give 2(3*H*)- or 2(5*H*)-furanones is described.

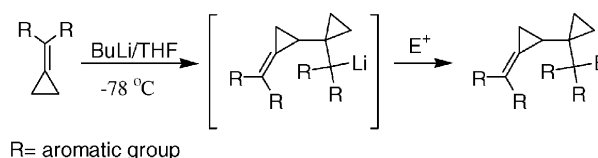


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**Carbolithiation of *gem*-aryl disubstituted methylenecyclopropanes**

Jin-Wen Huang and Min Shi\*

Novel carbolithiation of *gem*-aryl disubstituted methylenecyclopropanes was disclosed by the treatment with BuLi in THF at  $-78^\circ\text{C}$ . The corresponding addition products can be obtained in good to high yields by quenching with various electrophiles.

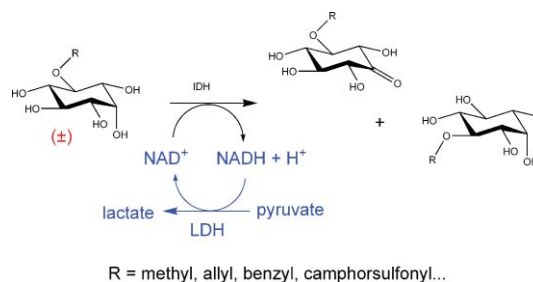


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**Stereoselective oxidation of protected inositol derivatives catalyzed by inositol dehydrogenase from *Bacillus subtilis***

Richard Daniellou, Christopher P. Phenix, Pui Hang Tam, Michael C. Laliberte and David R. J. Palmer\*

A nonpolar cavity in the active site in *Bacillus subtilis* inositol dehydrogenase allows stereoselective recognition of *myo*-inositol derivatives and disaccharides.

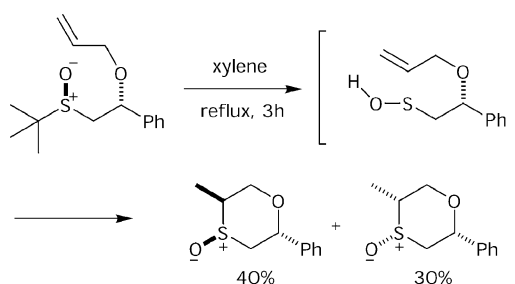


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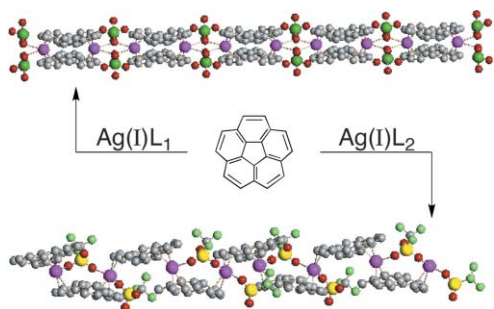
**Stereoselective synthesis of 2,5-disubstituted-1,4-oxathiane *S*-oxides**

Simon T. Bedford, Richard S. Grainger,\* Jonathan W. Steed and Patrizia Tisselli

$\beta$ -Allyloxy and  $\beta$ -propargyloxy *tert*-butyl sulfoxides undergo tandem sulfoxide elimination–intramolecular sulfenic acid addition reactions to produce 1,4-oxathiane *S*-oxides.



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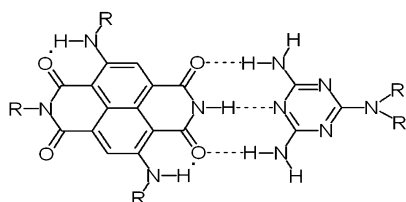


### Anion mediated structural motifs in silver(I) complexes with corannulene

Eric L. Elliott, Gerardo A. Hernández, Anthony Linden and Jay S. Siegel\*

Three silver(I)-corannulene complexes are described, each displaying different silver–corannulene connectivity and 3-D topology.

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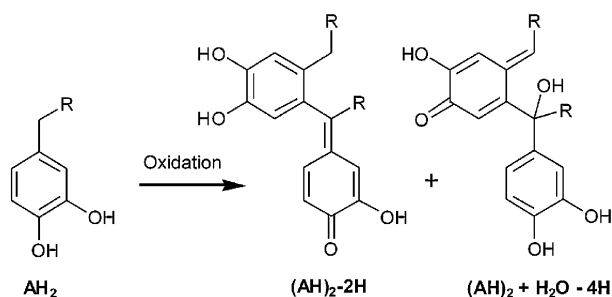


### Hydrogen bond directed self-assembly of core-substituted naphthalene bisimides with melamines in solution and at the graphite interface

Christoph Thalacker, Atsushi Miura, Steven De Feyter,\* Frans C. De Schryver and Frank Würthner\*

Core-substituted naphthalene bisimide dyes form triple hydrogen bonded arrays in solution with complementary melamine receptors.

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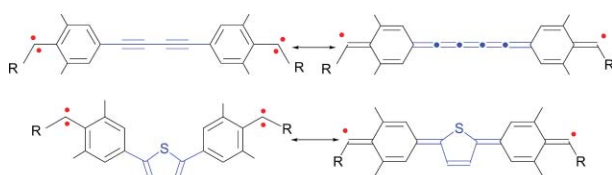


### Antioxidant activity of olive phenols: mechanistic investigation and characterization of oxidation products by mass spectrometry

Marjolaine Roche, Claire Dufour,\* Nathalie Mora and Olivier Dangles

The antioxidant activity of olive phenols was investigated using a DPPH test, a LC–MS characterization of the oxidation products and an analysis of the inhibition of lipid peroxidation in SDS micelles.

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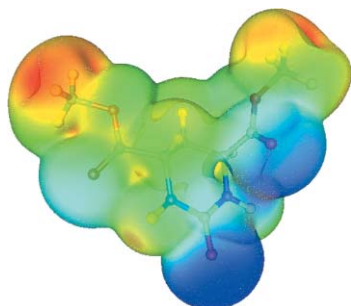


### Preparation of bis(diazo) compounds incorporated into butadiyne and thiophene units and generation and characterization of their photoproducts

Fumika Morisaki, Masakuni Kurono, Katsuyuki Hirai and Hideo Tomioka\*

Aryldiazomethyl substituents are introduced at the 1,4-positions of butadiyne and 2,5-positions of thiophene. Subsequent irradiation generates bis(carbenes) with singlet quinoidal diradical ground states with a singlet–triplet energy gap of  $<1$  kcal mol<sup>-1</sup>.

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### Experimental charge density of a potential DHO synthetase inhibitor: dimethyl-*trans*-2-oxohexahydro-pyrimidine-4,6-dicarboxylate

David E. Hibbs,\* Jacob Overgaard, Siân T. Howard and Thanh Ha Nguyen

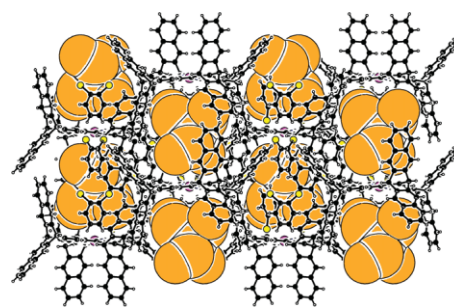
An analysis of the electrostatic properties obtained from the experimental charge density of the title compound reveals its potential interaction sites with DHO synthetase.

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### Extremely large cavity assembled by self-interlocking of distorted biconcave porphyrins

Hidemitsu Uno,\* Hikaru Watanabe, Yuko Yamashita and Noboru Ono

A large cage of *ca.* 720 Å<sup>3</sup> is formed between two tetrakis(dihydropentaceno)porphyrin molecules in the crystal.

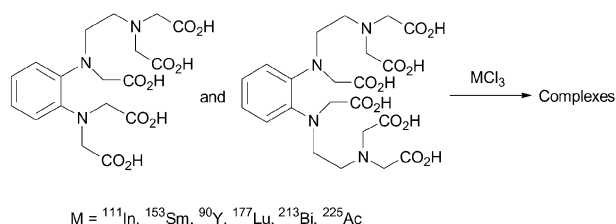


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### Synthesis and metal complexation properties of Ph-DTPA and Ph-TTHA: novel radionuclide chelating agents for use in nuclear medicine

Sébastien G. Gouin, Jean-François Gestin, Laurence Monrandeau, Fabienne Segat-Dioury, Jean Claude Meslin and David Deniaud\*

Synthesis and metal complexation properties of new radionuclide chelating agents for use in nuclear medicine.

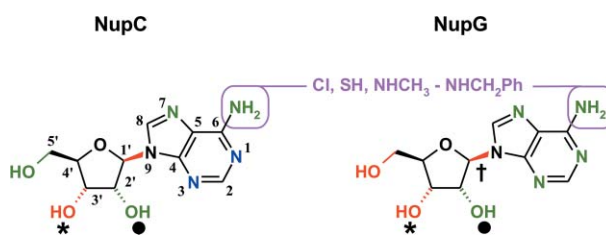


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### The nucleoside transport proteins, NupC and NupG, from *Escherichia coli*: specific structural motifs necessary for the binding of ligands

Simon G. Patching, Stephen A. Baldwin, Alexander D. Baldwin, James D. Young, Maurice P. Gallagher, Peter J. F. Henderson and Richard B. Herbert\*

A series of 46 natural nucleosides and analogues were tested as inhibitors of [<sup>14</sup>C]uridine uptake by the concentrative, H<sup>+</sup>-linked nucleoside transport proteins NupC and NupG from *Escherichia coli*.

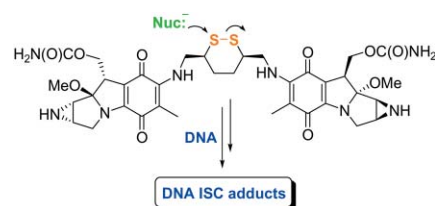


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### 7-*N*,7'-*N'*-(1'',2''-Dithianyl-3'',6''-dimethylenyl)-bismitomycin C: synthesis and nucleophilic activation of a dimeric mitomycin

Sang Hyup Lee and Harold Kohn\*

Dimeric mitomycin provides high levels of DNA interstrand cross-links (ISC) under nucleophilic activation conditions.

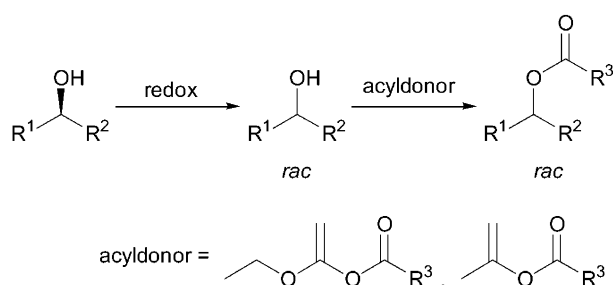


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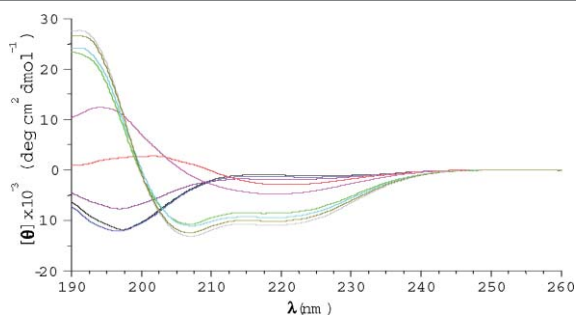
### Combined epimerisation and acylation: Meerwein–Ponndorf–Verley–Oppenauer catalysts in action

Dirk Klomp, Kristina Djanashvili, Nina Cianfanelli Svennum, Nuttanun Chantapariyavat, Chung-Sing Wong, Filipe Vilela, Thomas Maschmeyer, Joop A. Peters and Ulf Hanefeld\*

For the recovery of side-products of unselective alcohol syntheses and of kinetic resolutions a highly efficient epimerisation–acylation cascade was developed.



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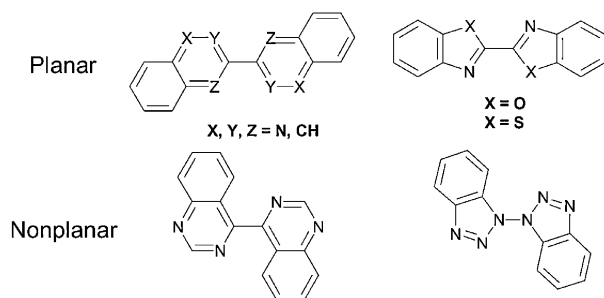


### Conformational properties of peptide fragments homologous to the 106–114 and 106–126 residues of the human prion protein: a CD and NMR spectroscopic study

Giuseppe Di Natale, Giuseppe Impellizzeri and Giuseppe Pappalardo\*

CD experiments at different sodium dodecyl sulfate concentrations revealed the presence of a conformational polymorphism in a PrP[Ac-106–126-NH<sub>2</sub>] peptide analogue.

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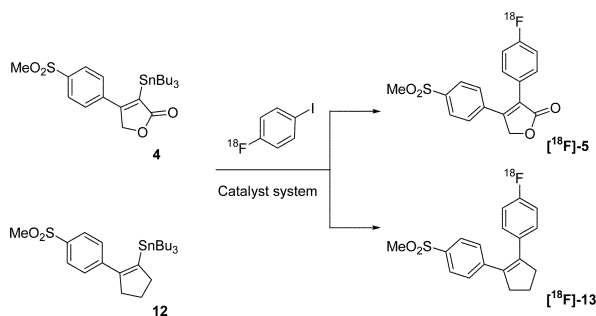


### Solid state conformations of symmetrical aromatic biheterocycles: an X-ray crystallographic investigation

Christopher M. Fitchett, Chris Richardson and Peter J. Steel\*

Accurate low temperature X-ray crystal structures are reported for seven symmetrical biheterocycles, five of which have planar centrosymmetric conformations.

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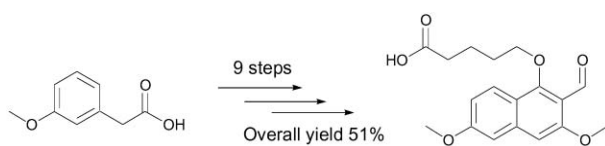


### Synthesis of <sup>18</sup>F-labelled cyclooxygenase-2 (COX-2) inhibitors *via* Stille reaction with 4-[<sup>18</sup>F]fluoroiodobenzene as radiotracers for positron emission tomography (PET)

Frank R. Wüst,\* Aileen Höhne and Peter Metz

The Stille reaction with 4-[<sup>18</sup>F]fluoroiodobenzene, a novel approach for the synthesis of radiotracers for PET monitoring of COX-2 expression, is developed. <sup>18</sup>F-Labelled COX-2 inhibitors [<sup>18</sup>F]-5 and [<sup>18</sup>F]-13 are obtained in radiochemical yields of 94% and 68%, respectively.

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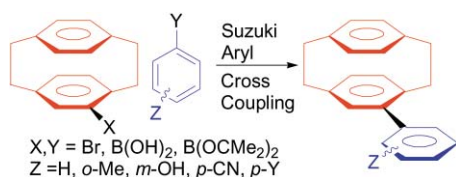


### Role of the *peri*-effect in synthesis and reactivity of highly substituted naphthaldehydes: a novel backbone amide linker for solid-phase synthesis

Michael Pittelkow, Ulrik Boas, Mikkel Jessing, Knud J. Jensen and Jørn B. Christensen\*

A convenient synthesis of a number of novel highly substituted naphthalene compounds and a facile synthetic route to the first backbone amide linker based on a trialkoxynaphthaldehyde has been developed.

515



### An exploration of Suzuki aryl cross coupling chemistry involving [2.2]paracyclophane derivatives

Alex J. Roche\* and Belgin Canturk

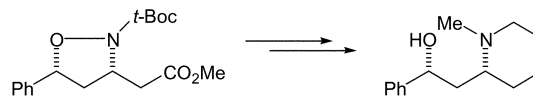
An examination into the scope and limitations of Suzuki aryl cross coupling chemistry using derivatives of [2.2]paracyclophane is described.

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**Synthesis of sedamine by tethered cyclofunctionalisation**

Roderick W. Bates\* and Jutatip Boonsombat

A synthesis of (+)-sedamine has been achieved, using an isoxazolidine intermediate.

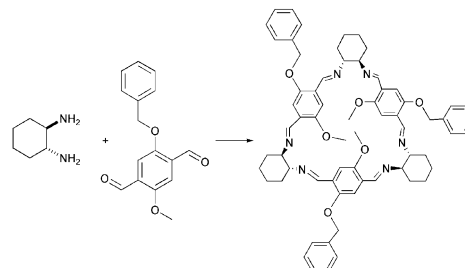


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**The synthesis and conformation of oxygenated trianglimine macrocycles**

Nikolai Kuhnert,\* Ana Lopez-Periago and Giulia M. Rossignolo

The synthesis of a number of oxygen substituted aromatic dicarboxaldehydes is described, along with their reactivity in the [3 + 3] cyclocondensation reaction to give novel trianglimine macrocycles.

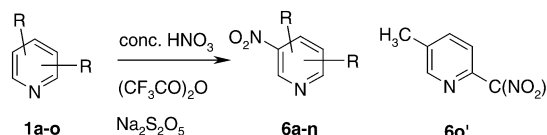


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**Preparation of nitropyridines by nitration of pyridines with nitric acid**

Alan R. Katritzky,\* Eric F. V. Scriven, Suman Majumder, Rena G. Akhmedova, Anatolii V. Vakulenko, Novruz G. Akhmedov, Ramiah Murugan and Khalil A. Abboud

Nitration of pyridines **1a–o** with nitric acid in trifluoroacetic anhydride, gave the corresponding nitropyridines **6a–o'** in yields of 10–83%. An X-ray study on **6o'** was conducted in order to confirm the structure.

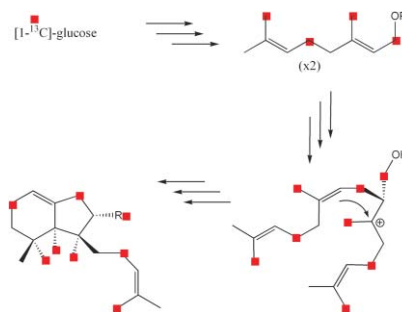


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**Biosynthesis of irregular diterpenes in *Anisotome lyallii* by head-to-head coupling of geranyl diphosphate**

John W. van Klink,\* Hans Becker and Nigel B. Perry

Biosynthesis of irregular diterpenes in *Anisotome* via the MEP pathway through head-to-head coupling of two GPP units.



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