

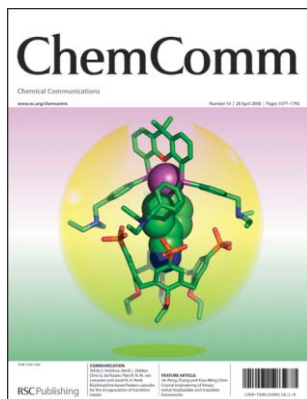
# ChemComm

Chemical Communications  
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## IN THIS ISSUE

ISSN 1359-7345 CODEN CHCOFS (16) 1677-1792 (2006)



### Cover

See Joost N. H. Reek *et al.*, page 1700. The figure shows the structure of a molecular capsule with a transition metal inside the cavity, which potentially can be used for homogeneous catalysis. Image reproduced by permission of Tehila S. Koblenz, Henk L. Dekker, Chris G. de Koster, Piet W. N. M. van Leeuwen and Joost N. H. Reek, from *Chem. Commun.*, 2006, 1700.



### Inside cover

See R. Graham Cooks *et al.*, page 1709. Rapid detection of the herbicide alachlor directly from a corn leaf using desorption electrospray ionization (DESI) coupled to a portable mass spectrometer. Cover artwork by Ryan Shreve. Image reproduced by permission of Christopher C. Mulligan, Nari Talaty and R. Graham Cooks, from *Chem. Commun.*, 2006, 1709.

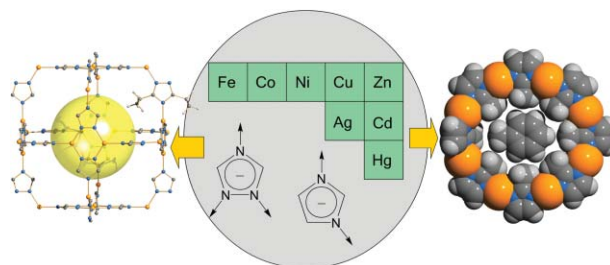
## FEATURE ARTICLE

1689

### Crystal engineering of binary metal imidazolate and triazolate frameworks

Jie-Peng Zhang and Xiao-Ming Chen\*

This article summarizes the recent advances in the crystal growth, structural control strategies and diverse structures of the binary metal imidazolate and triazolate frameworks.



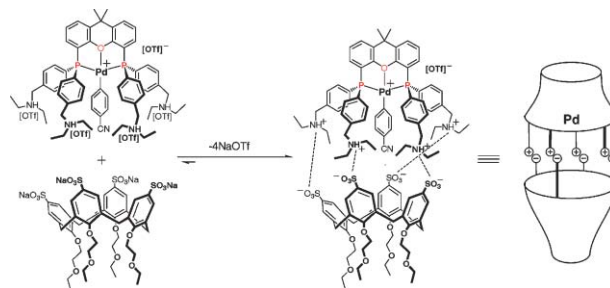
## COMMUNICATIONS

1700

### Bisphosphine based hetero-capsules for the encapsulation of transition metals

Tehila S. Koblenz, Henk L. Dekker, Chris G. de Koster, Piet W. N. M. van Leeuwen and Joost N. H. Reek\*

Just mixing the complementary building blocks provides a molecular capsule with the transition metal on the inside of the cavity.



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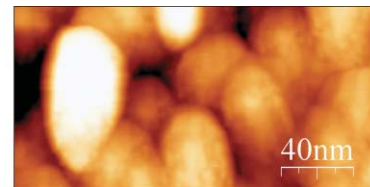
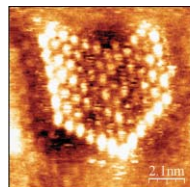
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1703

**Nanoscale electrocrystallisation of Sb and the compound semiconductor AlSb from an ionic liquid**

C. L. Aravinda and W. Freyland\*

We report the first example of nanoscale electrocrystallisation of the compound semiconductor AlSb from an ionic liquid.

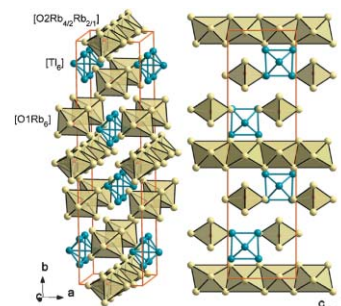


1706

**A<sub>10</sub>Tl<sub>6</sub>O<sub>2</sub> (A = K, Rb) cluster compounds combining structural features of thallium cluster anions and of alkali metal sub-oxides**

Andrey Karpov and Martin Jansen\*

New alkali metal thallideoxides, A<sub>10</sub>Tl<sub>6</sub>O<sub>2</sub> (A = K, Rb), crystallize in a unique structure consisting of hypoelectronic [Tl<sub>6</sub>]<sup>6-</sup> clusters in the shape of compressed octahedra, together with oxygen-centred alkali metal octahedra that have been identified as constitutive of alkali metal sub-oxides.

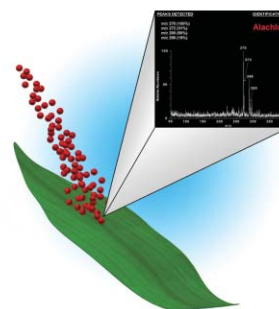


1709

**Desorption electrospray ionization with a portable mass spectrometer: *in situ* analysis of ambient surfaces**

Christopher C. Mulligan, Nari Talaty and R. Graham Cooks\*

*In situ* analysis of pharmaceuticals, alkaloids, explosives, chemical agents and agricultural chemicals is demonstrated from biological and non-biological surfaces using a portable MS. Air monitoring applications are also shown using DESI-MS.

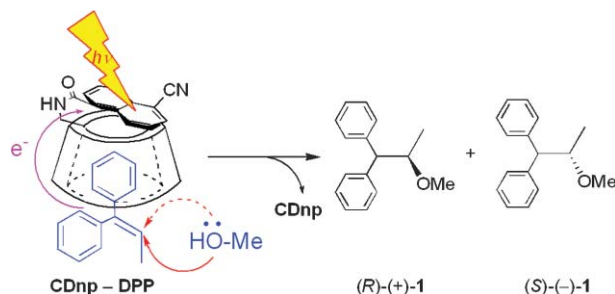


1712

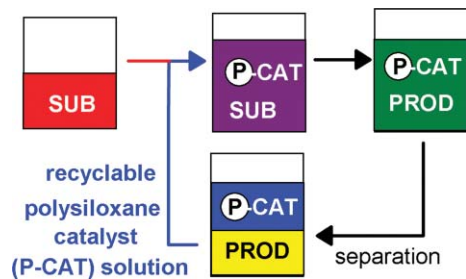
**The first supramolecular photosensitization of enantiodifferentiating bimolecular reaction: anti-Markovnikov photoaddition of methanol to 1,1-diphenylpropene sensitized by modified β-cyclodextrin**

Gaku Fukuhara, Tadashi Mori, Takehiko Wada and Yoshihisa Inoue\*

Supramolecular photoaddition of methanol to 1,1-diphenylpropene, sensitized by cyanonaphthalene-modified β-cyclodextrin, gave anti-Markovnikov adduct **1** with an unusual switching of product chirality by temperature.



1715

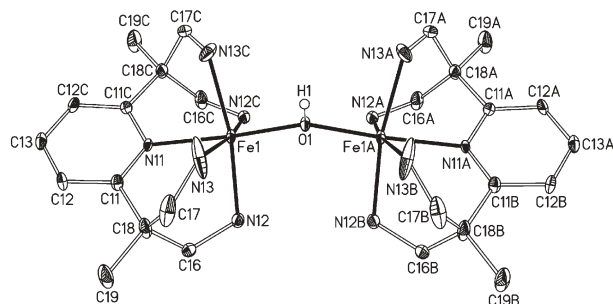


### Liquid/liquid separation of polysiloxane-supported catalysts

Melissa A. Grunlan, Katherine R. Regan and David E. Bergbreiter\*

Liquid/liquid separation after monophasic reactions is a viable way to use and recover polysiloxane-supported catalysts.

1718

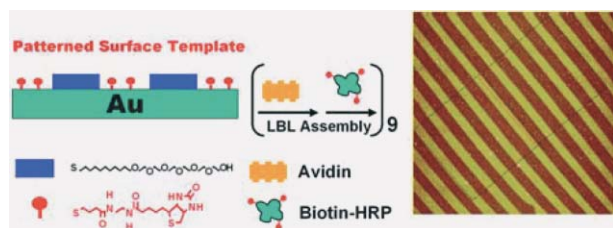


### A non-heme dinuclear iron(II) complex containing a single, unsupported hydroxo bridge

Jesús Pitarch López, Holger Kämpf, Matthias Grunert, Philipp Gülich, Frank W. Heinemann, Raju Prakash and Andreas Grohmann\*

The depicted complex contains the unprecedented diferrous fragment,  $\text{Fe}^{\text{II}}-\mu(\text{OH})-\text{Fe}^{\text{II}}$ , in a non-heme, tetraamineimine ligand environment

1721

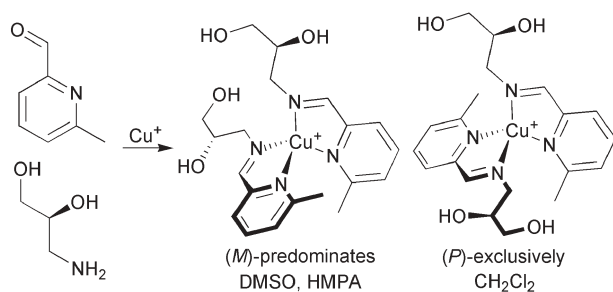


### Building three-dimensional nanostructures with active enzymes by surface templated layer-by-layer assembly

Sakandar Rauf, Dejian Zhou,\* Chris Abell, David Klenerman and Dae-Joon Kang\*

The first controlled fabrication of three-dimensional nanostructures with functional enzymes by templated layer-by-layer assembly of avidin and biotinylated horseradish peroxidase on micro-contact printing patterned surfaces is presented.

1724



### Solvent-tunable inversion of chirality transfer from carbon to copper

Marie Hutin and Jonathan Nitschke\*

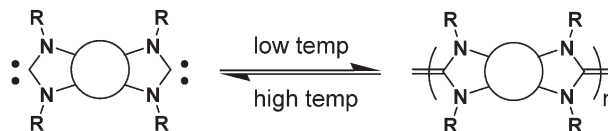
A change of solvent causes an inversion of the stereochemistry at copper of the chiral  $\text{Cu}^{\text{I}}$  complex described.

1727

### Dynamic covalent polymers based upon carbene dimerization

Justin W. Kamplain and Christopher W. Bielawski\*

Thermally-reversible covalent polymers featuring dynamic carbon-carbon double bonds and tunable molecular weights were prepared from difunctional carbenes; addition of transition metal complexes to these materials afforded the respective main-chain organometallic polymers.

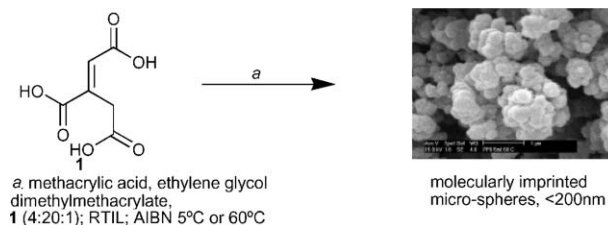


1730

### Efficient preparation and improved sensitivity of molecularly imprinted polymers using room temperature ionic liquids

Kate Booker, Michael C. Bowyer, Clovia I. Holdsworth and Adam McCluskey\*

Synthesis of *trans*-aconitic acid molecularly imprinted polymers in [bmim][BF<sub>4</sub>] and [bmim][PF<sub>6</sub>] under photochemical (5 °C, AIBN) and thermal (60 °C, AIBN) conditions gave polymer micro-spheres (<200 nm).

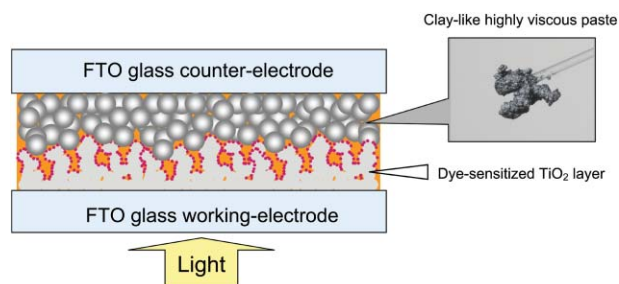


1733

### Conductive polymer-carbon-imidazolium composite: a simple means for constructing solid-state dye-sensitized solar cells

Nobuyuki Ikeda, Kenjiro Teshima and Tsutomu Miyasaka

Polymer-carbon-ionic liquid composite, a clay like electro-conductive paste, is effective for fabricating a solid-state dye-sensitized photocell. Without incorporation of iodine as a redox material, the cell gives power conversion efficiencies of 3.48% and 4.07% under 100 mW cm<sup>-2</sup> and 23 mW cm<sup>-2</sup> incident powers, respectively.

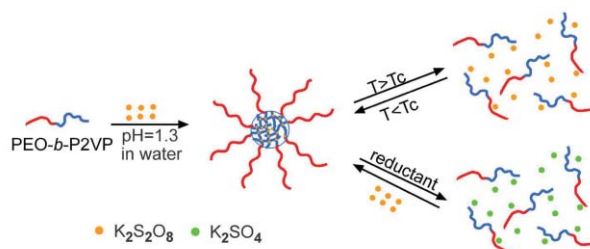


1736

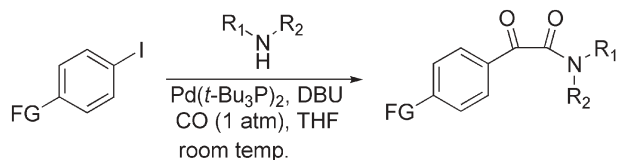
### Preparation of PEO-*b*-P2VPH<sup>+</sup>-S<sub>2</sub>O<sub>8</sub><sup>2-</sup> micelles in water and their reversible UCST and redox-responsive behavior

Xuan Jia, Daoyong Chen\* and Ming Jiang

We report here the preparation of water soluble micelles with reversible multi-responsive behavior based on PEO-*b*-P2VPH<sup>+</sup>-S<sub>2</sub>O<sub>8</sub><sup>2-</sup> complex.



1739

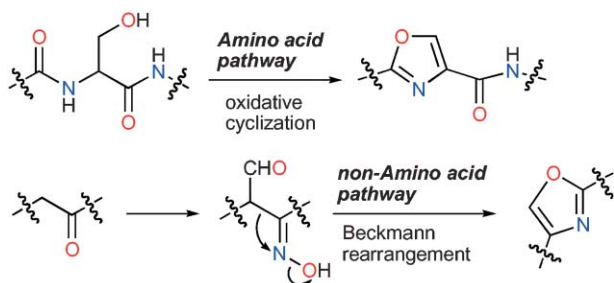


### Remarkable ligand effect on the palladium-catalyzed double carbonylation of aryl iodides

Muneaki Iizuka and Yoshinori Kondo\*

The use of *t*-Bu<sub>3</sub>P as a ligand dramatically improved the generality of the double carbonylation of aryl iodides.

1742

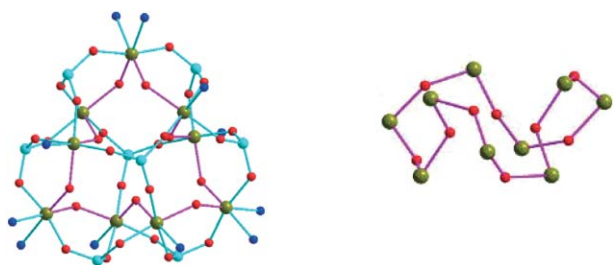


### Possibility of a non-amino acid pathway in the biosynthesis of marine-derived oxazoles

Takao Ichino, Hirokazu Arimoto\* and Daisuke Uemura\*

A novel avenue for oxazoles *via* Beckmann rearrangement of  $\alpha$ -formyl ketoxime dimethyl acetals is described that indicates the possibility of a non-amino acid biosynthetic pathway in marine natural products.

1745

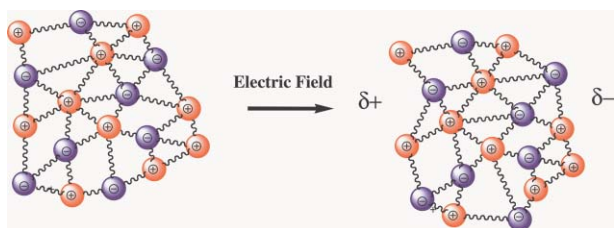


### An iron(III) phosphonate cluster containing a nonanuclear ring

Hong-Chang Yao, Jun-Jie Wang, Yun-Sheng Ma, Oliver Waldmann,\* Wen-Xin Du, You Song, Yi-Zhi Li, Li-Min Zheng,\* Silvio Decurtins\* and Xin-Quan Xin

This communication reports a novel cyclic nonanuclear iron(III) cluster  $[\text{Fe}_9(\mu\text{-OH})_7(\mu\text{-O})_2(\text{O}_3\text{PC}_6\text{H}_9)_8(\text{py})_{12}]$  which shows a quantum spin-frustration with a  $S = 1/2$  ground state.

1748



### Broadband dielectric response of the ionic liquid *N*-methyl-*N*-ethylpyrrolidinium dicyanamide

Simon Schrödle, Gary Annat, Douglas R. MacFarlane, Maria Forsyth, Richard Buchner and Glenn Hefter\*

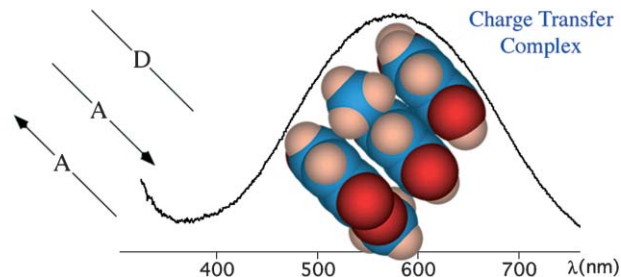
The first direct measurements of the dielectric behaviour of an ionic liquid as a function of temperature and dilution in dichloromethane reveal varying levels of ion aggregation.

1751

**A quinhydrone-type 2 : 1 acceptor–donor charge transfer complex obtained *via* a solvent-free reaction**

Marcel Bouvet,\* Bernard Malézieux and Patrick Herson

A 2 : 1 2-methoxybenzoquinone–hydroquinone complex, obtained as single crystals by a solvent-free reaction, combines dipolar and  $\pi$ – $\pi$  interactions, as well as hydrogen bonding.

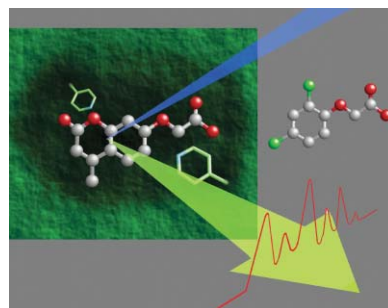


1754

**A fluorescence polarisation molecular imprint sorbent assay for 2,4-D: a non-separation pseudo-immunoassay**

Claire E. Hunt, Pamela Pasetto, Richard J. Ansell\* and Karsten Haupt\*

The first pseudo-immunoassay which employs a molecularly imprinted receptor and a fluorescent probe, and quantifies the bound analyte directly using the fluorescence anisotropy of the polymer–probe–analyte suspension, is described.

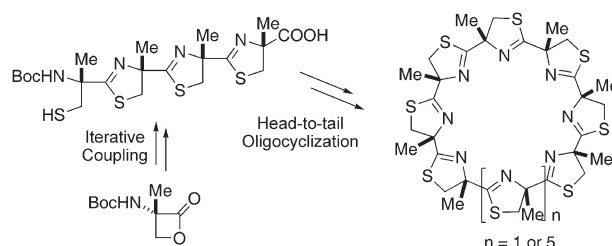


1757

**Synthesis of chiral cyclic oligothiazolines: a novel structural motif for a macrocyclic molecule**

Fu She Han, Hiroyuki Osajima, Mui Cheung, Hidetoshi Tokuyama and Tohru Fukuyama\*

24- to 36-membered chiral cyclic oligo(4- $\beta$ -methyl)thiazolines were efficiently synthesized through iterative formation of linear oligothiazolines and a head-to-tail cyclooligomerization.

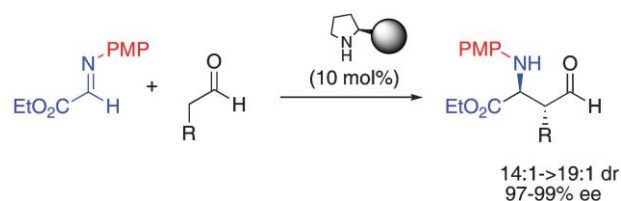


1760

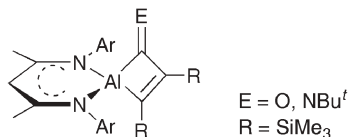
**Direct catalytic asymmetric *anti*-selective Mannich-type reactions**

Ismail Ibrahim and Armando Córdoba\*

A simple chiral pyrrolidine-catalyzed direct asymmetric *anti*-selective Mannich-type reaction is presented. The organocatalytic Mannich-type reaction was highly *anti*- and enantioselective and furnished the corresponding Mannich products in high yields with 14 : 1  $\rightarrow$  19 : 1 dr and 97–99% ee.



1763

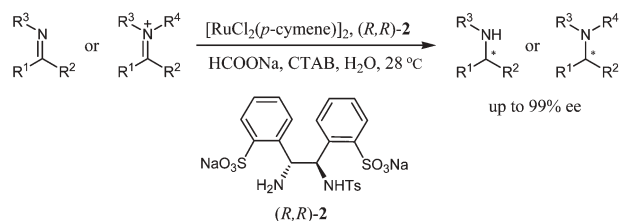


### Formation of aluminacyclobutenes *via* carbon monoxide and isocyanide insertion

Xiaofei Li, Chengbao Ni, Haibing Song and Chunming Cui\*

Novel aluminium-substituted cyclobutene analogues were obtained by the single atom insertion reactions of CO and *tert*-butyl isocyanide with  $LAl[\eta^2-(CSiMe_3)_2]$  ( $L = HC[(CMe)(NAr)]_2$ ,  $Ar = 2,6\text{-}iPr_2C_6H_3$ ).

1766

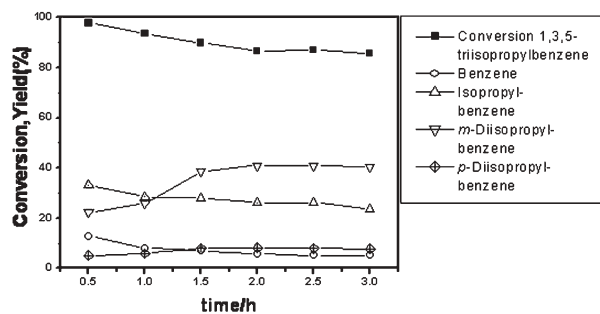


### Asymmetric transfer hydrogenation of imines and iminiums catalyzed by a water-soluble catalyst in water

Jiashou Wu, Fei Wang, Yaping Ma, Xin Cui, Linfeng Cun, Jin Zhu,\* Jingen Deng\* and Bangliang Yu

The first asymmetric transfer hydrogenation of cyclic imines and iminiums in water was successfully performed in high yields and enantioselectivities (up to 99% ee) catalyzed by a water-soluble and recyclable ruthenium(II) complex of the ligand  $(R,R)\text{-}2$ .

1769

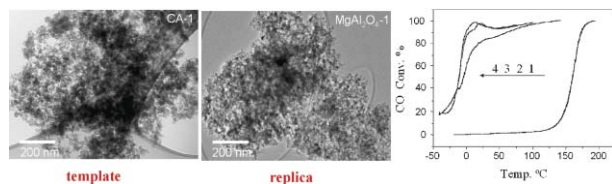


### Catalytic cracking of large molecules over hierarchical zeolites

Qian Lei, Tianbo Zhao,\* Fengyan Li, Lingling Zhang and Yue Wang

A hierarchical zeolite catalyst was synthesized by transforming the skeletons of a bimodal pore silica gel into a zeolite through a steam-assisted conversion method, and shows high catalytic activity for catalytic cracking of large molecules.

1772



### Nanocasting mesoporous $MgAl_2O_4$ spinel monoliths as support for highly active gold CO oxidation catalyst

Wen-Cui Li, Massimiliano Comotti, An-Hui Lu and Ferdi Schüth\*

Mesoporous  $MgAl_2O_4$  spinel monolith was synthesized by the nanocasting pathway. High activity in CO oxidation was observed over gold catalysts based on such monoliths as support.

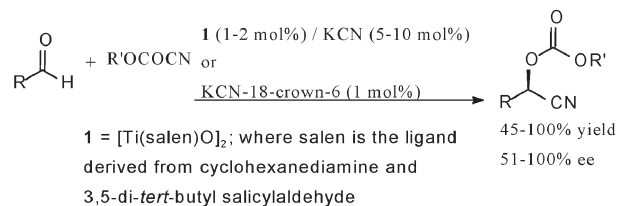


1775

### Cyanide ion cocatalysis in Ti(salen) catalysed asymmetric cyanohydrin carbonate synthesis

Yuri N. Belokon, Eisuke Ishibashi, Hiroshi Nomura and Michael North\*

Both achiral and chiral cyanoformates can be used. In the case of chiral cyanoformates, the reaction exhibits double asymmetric induction with the stereochemistry of catalyst **1** being the dominant factor in determining the stereochemistry of the product.

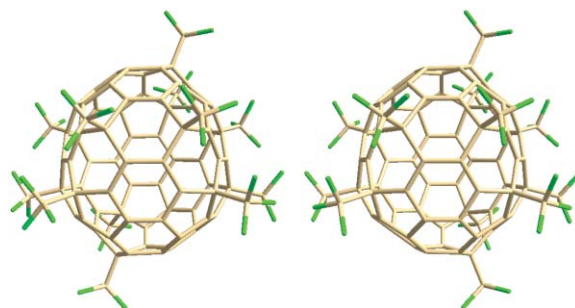


1778

### Preparation, crystallographic characterization and theoretical study of two isomers of C<sub>70</sub>(CF<sub>3</sub>)<sub>12</sub>

Daria V. Ignat'eva, Alexey A. Goryunkov, Nadezhda B. Tamm, Ilya N. Ioffe, Stanislav M. Avdoshenko, Lev N. Sidorov, Anton Dimitrov, Erhard Kemnitz and Sergey I. Troyanov\*

Two isomers of C<sub>70</sub>(CF<sub>3</sub>)<sub>12</sub> obtained by trifluoromethylation of C<sub>70</sub> with CF<sub>3</sub>I have been isolated and characterized by X-ray crystallography.

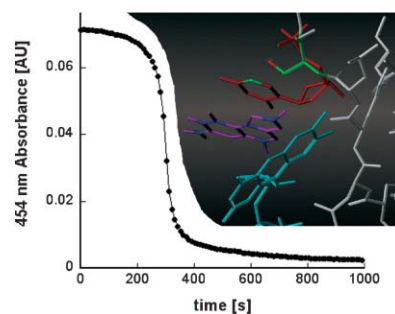


1781

### A lag-phase in the reduction of flavin dependent thymidylate synthase (FDTS) revealed a mechanistic missing link

Aaron Mason, Nitish Agrawal, M. Todd Washington, Scott A. Lesley and Amnon Kohen\*

A lag-phase was found in the single turnover reduction of FDTS bound flavin. The lag-phase's substrate-dependency sheds light on the molecular mechanism of this alternative thymidylate synthase.

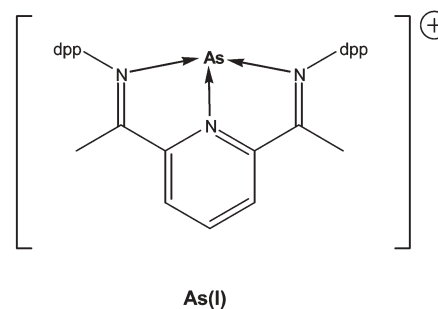


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### Controlling the oxidation state of arsenic in cyclic arsenic cations

Gregor Reeske and Alan H. Cowley\*

Reaction of "AsCl" or AsI<sub>3</sub> with a diiminopyridine ligand produces an arsenic(I) cation.




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