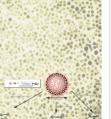
# ChemComm

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**C65** 





Cover (far left) Structure of carbamazepine-saccharin against a background photograph of the co-crystals (pp. 1889–1896).

#### Inside cover (left)

TEM image of surface-functionalised nanobeads which, along with standard polymeric supports, have been elaborated into solid-phase "triflate" equivalents for traceless linking and cross-coupling release strategies (pp. 1914–1915).

Chemical Science

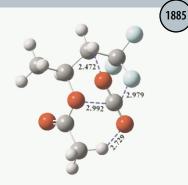
**1)]][**]]

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September 2004/Volume 1/Issue 9 www.rsc.org/chemicalscience 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.

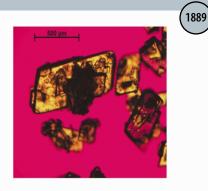


## FOCUS ARTICLI

A challenge for green chemistry: designing molecules that readily dissolve in carbon dioxide

## E. J. Beckman

Carbon dioxide is a green yet feeble solvent whose full potential won't be realized until we develop a more thorough understanding of its solvent behavior at the molecular level. Fortunately, advances in molecular modeling coupled with experiments are rapidly improving our understanding of CO<sub>2</sub>'s behavior, permitting design of new, more sustainable "CO<sub>2</sub>-philes".



## EATURE ARTICLE

Crystal engineering of the composition of pharmaceutical phases. Do pharmaceutical co-crystals represent a new path to improved medicines?

Örn Almarsson\* and Michael J. Zaworotko\*

The current and potential impact of crystal engineering on how the pharmaceutical industry evolves its approach towards formulation of active pharmaceutical ingredients (API's) is featured in the context of polymorphs and co-crystals. Carbamazepine, an API that exemplifies the issues and challenges raised by polymorphs and co-crystals, forms co-crystals with saccharin as illustrated.

i

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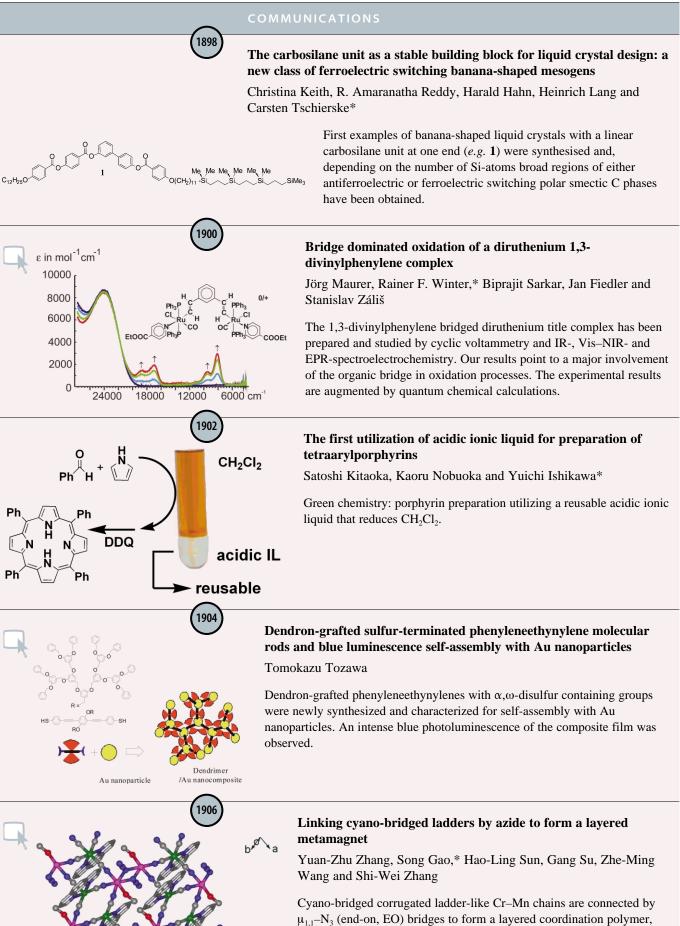
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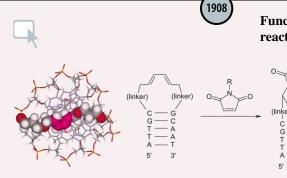
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 $\mu_{1,1}$ -N<sub>3</sub> (end-on, EO) bridges to form a layered coordination polymer, which is the first mixed cyano–azide bridged coordination polymer, and shows metamagnetism below 21.8 K.

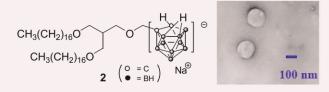


## Functionalisation of a diene-modified hairpin mimic *via* the Diels-Alder reaction

Rolf Tona and Robert Häner\*

A highly stable 1,3-butadiene-derived DNA hairpin mimic and its derivatisation *via* the Diels–Alder reaction with various dienophiles are described.

## Synthesis and vesicle formation of a nido-carborane cluster lipid for boron neutron capture therapy



 $hv > E_{bg}$ 

TiO<sub>2</sub>

 $H_2O$ 

TiO

1910

1913

mild quenching

SED

TiO<sub>2</sub>\*

Oxidised SED

Traceless

Cross-coupling

release

Hiroyuki Nakamura,\* Yusuke Miyajima, Toshiaki Takei, Satoshi Kasaoka and Kazuo Maruyama

The nido-carborane lipid **2**, which consists of an ionic boron cluster as a hydrophilic part and a double-tailed moiety, was synthesized for the first time. It was shown by transmission electron microscopy analysis that the lipid **2** formed a stable vesicle and was highly accumulated into DSPC liposomes.

## An intelligence ink for oxygen

Soo-Keun Lee, Andrew Mills\* and Anne Lepre

A novel generic irreversible, UV-activated, re-useable, colorimetric intelligence ink for oxygen comprises semiconductor photocatalyst particles, a brightlycoloured redox dye, a mild reducing agent, a polymer and a solvent.

## A novel solid-phase equivalent to the triflate group and its application to traceless linking and cross-coupling-release strategies

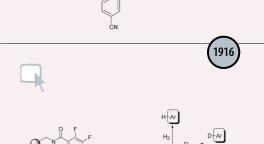
Andrew N. Cammidge\* and Zainab Ngaini

A novel polymer solid-phase equivalent to the triflate group has been developed based on polyfluorophenyl sulfonates. The supports can be employed for traceless linking and cross-coupling release strategies.

## A 'triflate-like' tetrafluoroarylsulfonate linker for multifunctional solidphase organic synthesis

Jefferson D. Revell and A. Ganesan

A new tetrafluoroarylsulfonate solid-phase linker that is readily prepared from pentafluorobenzoic acid is described. The linker functions as a solid-phase 'triflate' equivalent, enabling multifunctional compound cleavage by palladium(0) catalyzed reactions.





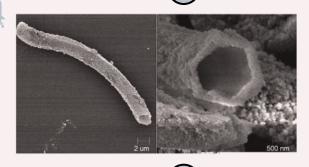
i v



## Brookite-supported highly stable gold catalytic system for CO oxidation

Wenfu Yan, Bei Chen, Shannon M. Mahurin, Sheng Dai\* and Steven H. Overbury

A significant enhancement of the gold catalysis stability against sintering has been achieved using brookite as a catalytic support.



**′**1922

1918

## Hexagonal, hollow, aluminium-containing ZSM-5 tubes prepared from mesoporous silica templates

W. Song, R. Kanthasamy, V. H. Grassian and S. C. Larsen\*

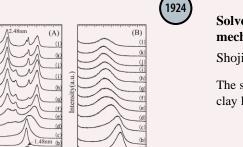
Hexagonal hollow ZSM-5 tubes were synthesized using mesoporous silica with a worm-like morphology as the template. A new method for aluminium incorporation during the hydrothermal synthesis step was developed.

## A simple and highly efficient P,O-type ligand for Suzuki–Miyaura crosscoupling of aryl halides

Fuk Yee Kwong,\* Wai Har Lam, Chi Hung Yeung,\* Kin Shing Chan and Albert S. C. Chan\*

$\begin{array}{c} \text{ArCl} + \text{R-B(OH)}_2 \xrightarrow{\text{K}_3\text{PO}_4 \text{ hydrate}} \text{Ar-R} \\ \text{R = aryl, alkyl} & 100 ^{\circ}\text{C} \end{array}$	Pt-Bu <sub>2</sub> 3 t-Bu-Bphos	<b>5</b> 1 1	toluene Ar-R	
--	---------------------------------------	--------------	--------------	--

Simple and highly efficient hemilabile P,O-type ligands were prepared in one step from inexpensive benzamide. The active Bphos ligands showed relatively high turnover numbers in Suzuki–Miyaura coupling of aryl chlorides.



3 4 5 6 7 8 9 10 2θ/° CuKα

1926

4 5 6 7 8 2θ/°CuKα

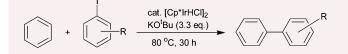
## Solvent free synthesis of polyaniline–clay nanocomposites from mechanochemically intercalated anilinium fluoride

Shoji Yoshimoto,\* Fumihiko Ohashi, Yasushi Ohnishi and Toru Nonami

The synthesis of nanocomposites that contain much conducting polyaniline in the clay layers was achieved by the mechanochemical intercalation method.

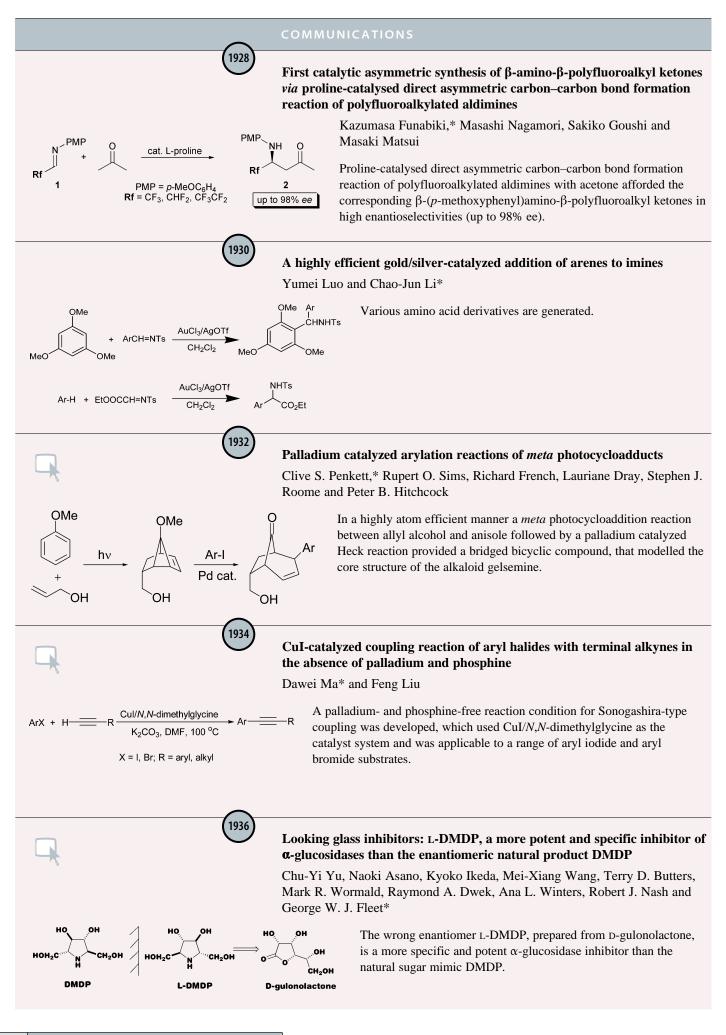
## Direct arylation of aromatic C–H bonds catalyzed by Cp\*Ir complexes

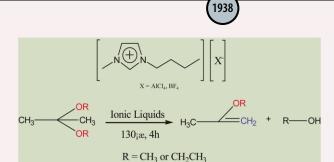
Ken-ichi Fujita,\* Mitsuru Nonogawa and Ryohei Yamaguchi\*



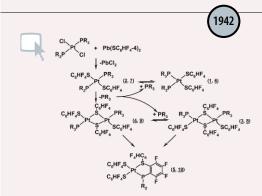
The C–H bond of benzene was directly arylated by reaction with aryl iodides in the presence of a catalytic amount of a pentamethylcyclopentadienyliridium complex and potassium *tert*-butoxide.

v





# 



# Ionic liquids as catalytic green solvents for cracking reactions

Yong Wang, Haoran Li,\* Congmin Wang and Hui Jiang

Room-temperature ionic liquids were used as catalysts for the cracking reaction of alkoxypropanes. The elimination reactions of alcohols to give alkenes in ionic liquids are investigated for the first time.

Template-assisted self assembly of two lipophilic polyion aggregates derived from sodium tetraphenyl imidodiphosphinate-complexes containing sodium ions in four different coordination environments

Perla Román-Bravo, Marcela López-Cardoso, Patricia García y García, Herbert Höpfl and Raymundo Cea-Olivares\*

The structural organization of alkaline metal ions in ion channels can be simulated by their combination with ligands that mimic the spatial distribution of carbonyl groups in peptides.

# Conversion of $[Pt(SRf)_2(PPh_{2-n}(C_6F_5)_{n+1})_2]$ (n = 0 or 1, Rf=C<sub>6</sub>HF<sub>4</sub>-4) through carbon–fluorine bond activation to $[Pt(SRf)_2(1,2-C_6F_4(SRf)-(PPh_2))]$ and chiral $[Pt(SRf)_2(1,2-C_6F_4(SRf)(PPh(C_6F_5)))]$

Luis Villanueva, Maribel Arroyo, Sylvain Bernès and Hugo Torrens\*

Rare examples of metal promoted C–F activation from  $PPh_{3-n}(C_6F_5)_n$  involving polyfluorothiolate ligands to afford 1-thiolate-2-phosphine–tetrafluorophenyl complexes.

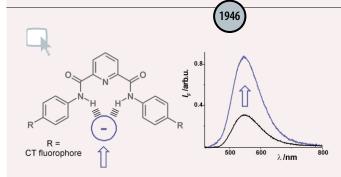
## Ion/molecule reactions of the protonated serine octamer

Scott Gronert,\* Richard A. J. O'Hair\* and Adelaide E. Fagin

(Ser)<sub>8</sub>H<sup>+</sup>

 $R_3N$ 

The protonated serine octamer and its derivatives react with amines in the gas phase to give substitution products.



1944

(Ser)<sub>7</sub>(R<sub>3</sub>N)H<sup>+</sup> + Ser

## A charge transfer-type fluorescent molecular sensor that "lights up" in the visible upon hydrogen bond-assisted complexation of anions

Anton Kovalchuk, Julia L. Bricks, Günter Reck, Knut Rurack,\* Burkhard Schulz, Agnieszka Szumna and Hardy Weißhoff

A molecular charge transfer fluorosensor consisting of a bisamidopyridine receptor and two styryl base chromophores shows  $H_2PO_4^-$  and acetate-enhanced fluorescence *via* conversion of weak intramolecular into strong intracomplex hydrogen bonds.

1948

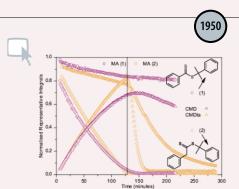
1952

 $O^-H_2 N^+ Pr_2^i$ 

## Direct oxidation of methane to acetic acid catalyzed by Pd<sup>2+</sup> and Cu<sup>2+</sup> in the presence of molecular oxygen

Mark Zerella, Sudip Mukhopadhyay and Alexis T. Bell\*

Methane is catalytically converted primarily to acetic acid in concentrated sulfuric acid using a combination of Pd<sup>2+</sup> and Cu<sup>2+</sup> in the presence of oxygen. The presence of Cu<sup>2+</sup> and O<sub>2</sub> enhances the formation of acetic acid and suppresses the reduction of Pd<sup>2+</sup> to Pd black.



Pd<sup>2+</sup>/Cu<sup>2+</sup>/O<sub>2</sub> → CH<sub>3</sub>COOH

# The slow "propagation" of the initiating and leaving group radicals during the

early part of methyl acrylate RAFT-mediated polymerisation has characteristics similar to inhibition.

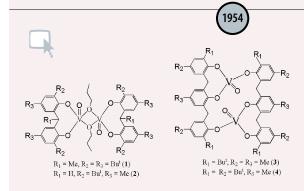
Initialisation in RAFT-mediated polymerisation of methyl acrylate J. B. McLeary, J. M. McKenzie, M. P. Tonge,\* R. D. Sanderson and B.

## Phosphoramidic acid monoesters as phosphorylating agents: steric effects and reluctance to form monomeric metaphosphate intermediates

Martin J. P. Harger

Klumperman\*

Phosphoramidic acid monoesters cannot readily form monomeric metaphosphate intermediates in the way that their P=S analogues do so they have to react with alcohols by a sterically-sensitive  $S_N 2(P)$  mechanism.



ROH S<sub>N</sub>2(P)

 $- NPr_2^i$ 

 $RO - P_{N}^{\prime \prime O}$ 

Î ОН

## Vanadyl complexes bearing bi- and triphenolate chelate ligands: highly active ethylene polymerisation procatalysts

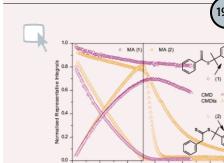
Carl Redshaw,\* Lee Warford, Sophie H. Dale and Mark R. J. Elsegood

The procatalysts 1-4 show high activity, in combination with dimethylaluminium chloride, for the conversion of ethylene to high molecular weight polyethylene.

## Bis(benzimidazole)amine vanadium catalysts for olefin polymerisation and co-polymerisation: thermally robust, single-site catalysts activated by simple alkylaluminium reagents

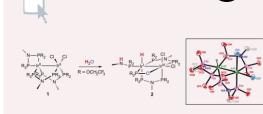
Atanas K. Tomov, Vernon C. Gibson,\* Damien Zaher, Mark R. J. Elsegood and Sophie H. Dale

Vanadium complexes containing bis(benzimidazole)amine ligands, when activated by simple alkylaluminium reagents, are found to give highly active, thermally robust, single-site catalysts for olefin polymerisation and co-polymerisation.



 $H_2SO_4$ 

 $CH_4$ 



Water addition to a two-electron mixed-valence bimetallic center

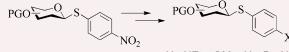
Adam S. Veige and Daniel G. Nocera\*

Water reacts at two-electron mixed-valence  $Ir^{0,u}_2$  cores ligated by diphosphazane ligands to produce hydride and an unusual dimetal hydroxy phosphite bridge.

# One-pot oligosaccharide synthesis: reactivity tuning by post-synthetic modification of aglycon

which enables one-pot oligosaccharide syntheses.

Lijun Huang, Zhen Wang and Xuefei Huang\*



X =NEt<sub>2</sub>, OMe, N<sub>3</sub>, Br, NPhth

1958

1960

80% yield (d.s. >95:5)

196

## Radical addition of nitrones to acrylates mediated by SmI<sub>2</sub>: asymmetric synthesis of γ-amino acids employing carbohydrate-based chiral auxiliaries

Post-synthetic modification of aglycons produces glycosyl building blocks with multiple levels of anomeric reactivities divergently,

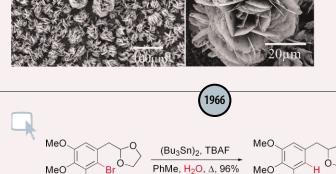
Sine A. Johannesen, Silvia Albu, Rita G. Hazell and Troels Skrydstrup\*

An asymmetric version of the samarium diiodide promoted radical addition of aliphatic nitrones to acrylates is disclosed providing examples for the synthesis of  $\gamma$ -amino acid derivatives with high diastereoselectivities (>95 : 5).

## Super-hydrophobic tin oxide nanoflowers

Aicheng Chen,\* Xinsheng Peng, Kallum Koczkur and Brad Miller

Super-hydrophobic 3D  $\text{SnO}_2$  nanoflowers were fabricated from Sn nanoflowers formed by thermal-pyrolysis of a Sn precursor. The nanoporous  $\text{SnO}_2$  flowers possess a large surface area, which supports the potential usefulness of these materials in gas sensor design.



OMe OMe (Bu<sub>3</sub>Sn)<sub>2</sub>, TBAF PhMe, D<sub>2</sub>O, Δ, 93% OMe

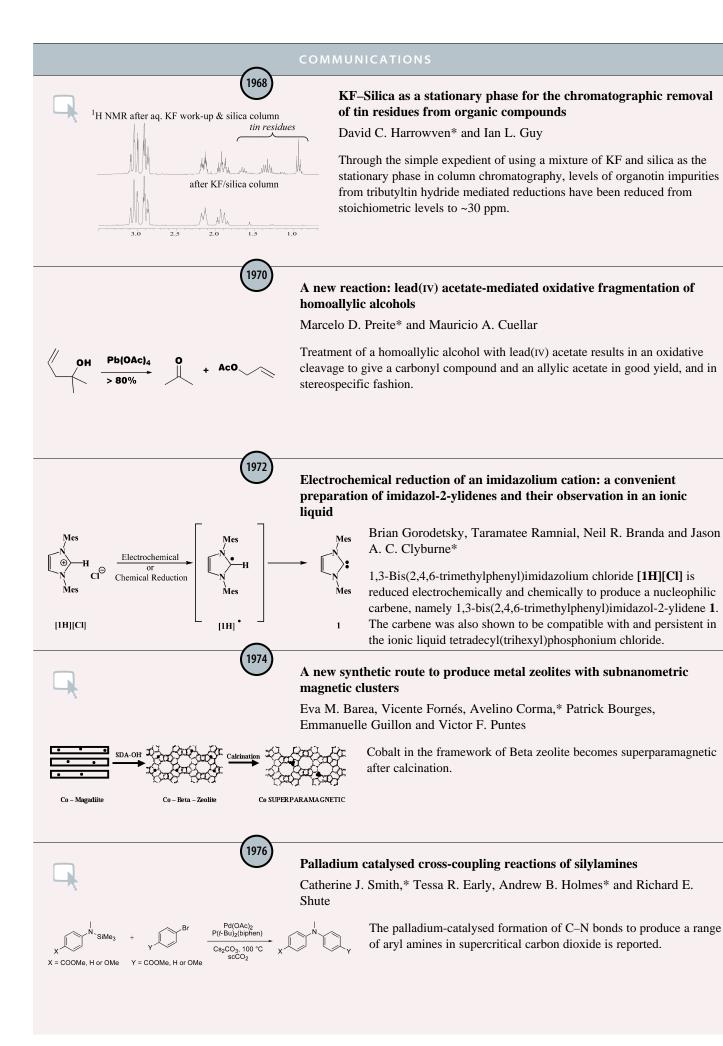
# (Bu<sub>3</sub>Sn)<sub>2</sub>–TBAF: a new combination reagent for the reduction and deuteration of aryl bromides and iodides

David C. Harrowven,\* Ian L. Guy and Michael I. T. Nunn

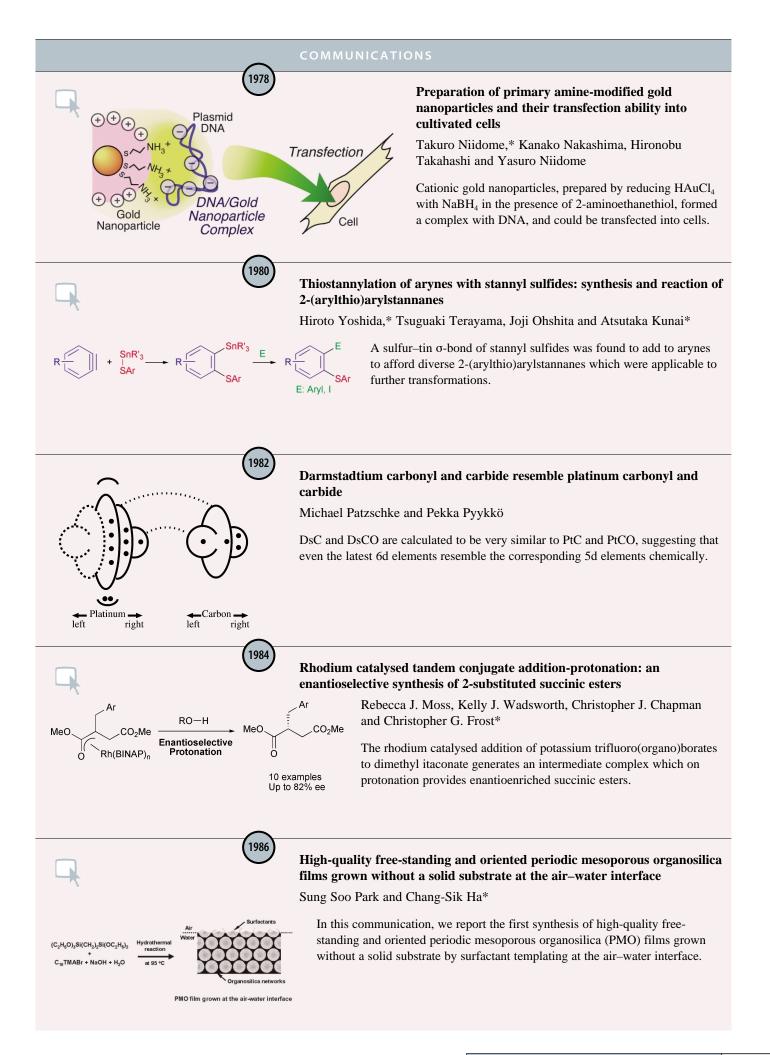
 $(Bu_3Sn)_2$ -TBAF is highly effective for the reduction of aromatic bromides and iodides. When the residual water in TBAF is exchanged for D<sub>2</sub>O, the halogen is replaced by a deuterium atom.

0

iх



х



хi



1988

1990

1994

## Super-long continuous Ni nanowires encapsulated in carbon nanotubes

Lunhui Guan, Zujin Shi, Huanjun Li, Liping You and Zhennan Gu\*

Super-long continuous Ni-filled carbon nanotubes were prepared by the chemical vapor deposition method with cloth-like single-walled carbon nanotube raw soot produced by the arc-discharge method as catalyst.

## A highly efficient heterogeneous catalytic system for Heck reactions with a palladium colloid layer reduced *in situ* in the channel of mesoporous silica materials

Liang Li, Jian-lin Shi\* and Ji-na Yan

The synthesis and characterization of a palladium colloid layer grafted mesoporous SBA-15 material, designated Pd–SBA, are described; the ultrahigh specific area, the large pore opening, and the highly dispersed catalyst species in the Pd–SBA material create one of the most active heterogeneous catalysts for Heck coupling reactions.

# Adjustable cavity for host-guest recognition in cofacial bis-porphyrinic tweezer

Régis Rein, Maurice Gross and Nathalie Solladié\*

The synthesis of a cofacial bis-porphyrinic tweezer bearing a tris-anthracenic spacer is reported. Its behavior as host has been evidenced as well as the ability of its cavity to adjust to guests of various sizes.

# Application of a diphosphinidenecyclobutene ligand in the solvent-free copper-catalysed amination reactions of aryl halides



poly(A)/lipid

Complementar

hydrogen-bonding

in organogel system

X = Br, Cl, I; R<sup>1</sup>= H, alkyl, aryl; R<sup>2</sup>= alkyl, aryl; Mes<sup>\*</sup>= 2,4,6- $tBu_3C_6H_2$ .

Anil S. Gajare, Kozo Toyota, Masaaki Yoshifuji\* and Fumiyuki Ozawa

Copper-catalysed amination reactions of halobenzenes with amines proceed at 100 °C in the presence of a diphosphinidenecyclobutene (2 mol%), CuI (2 mol%), and *t*-BuOK without solvent, providing an efficient method for the introduction of one or two aryl groups into amines.

## Complementary hydrogen-bonding between thymidinebased low molecular-weight gelator and polynucleotide in organic media

Kazunori Sugiyasu, Munenori Numata, Norifumi Fujita, Sun Min Park, Young Ji Yun, Byeang Hyean Kim and Seiji Shinkai\*

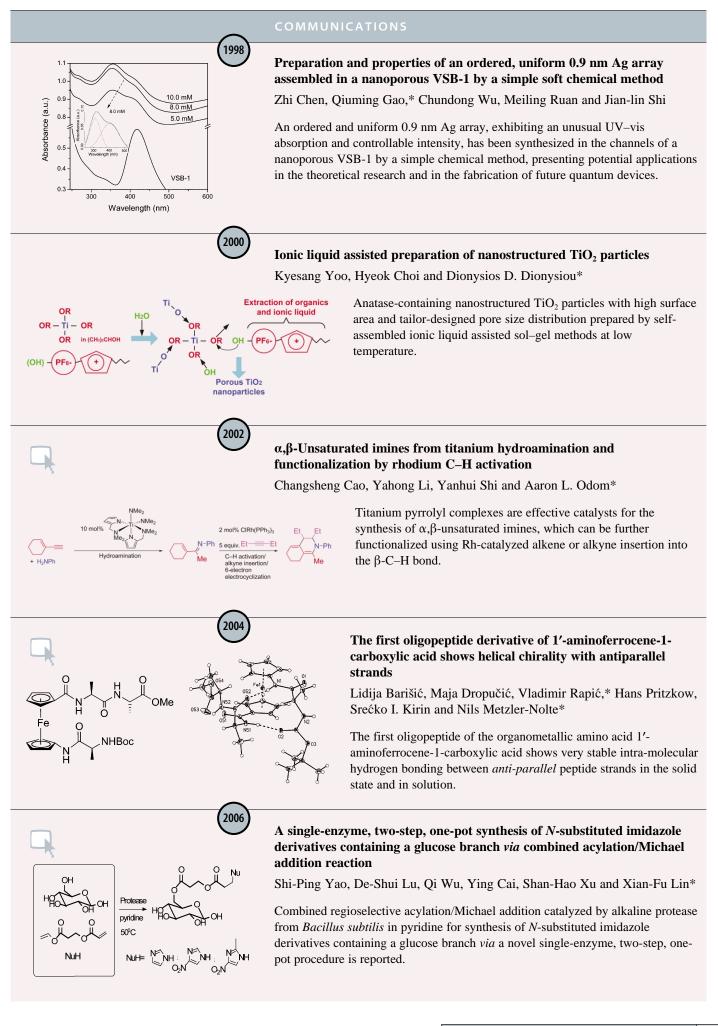
Aggregation mode and gelation property of thymidine-based organogelator **1** are affected by the addition of its complementary polynucleotide in organic media.

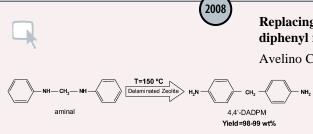
xii Chem. Commun., 2004

C10Ho

C12H25

poly(A)/lipid





Replacing HCl by solid acids in industrial processes: synthesis of diamino diphenyl methane (DADPM) for producing polyurethanes

Avelino Corma,\* Pablo Botella and Chris Mitchell

Delaminated zeolites show high activity and long catalyst life in the synthesis of diamino diphenyl methane (DADPM), the intermediate in the production of polyurethanes.

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## AUTHOR INDEX

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Gu, Zhennan, 1988 Guan, Lunhui, 1988 Guillon, Emmanuelle, 1974 Guy, Ian L., 1966, 1968 Ha, Chang-Sik, 1986 Hahn, Harald, 1898 Häner, Robert, 1908 Har Lam, Wai, 1922 Harger, Martin J. P., 1952 Harrowven, David C., 1966, 1968 Hazell, Rita G., 1962 Hitchcock, Peter B., 1932 Holmes, Andrew B., 1976 Höpfl, Herbert, 1940 Huang, Lijun, 1960 Huang, Xuefei, 1960 Hung Yeung, Chi, 1922 Hyean Kim, Byeang, 1996 Ikeda, Kyoko, 1936 Ishikawa, Yuichi, 1902 Ji Yun, Young, 1996 Jiang, Hui, 1938 Johannesen, Sine A., 1962 Kanthasamy, R., 1920 Kasaoka, Satoshi, 1910 Keith, Christina, 1898 Kirin, Srećko I., 2004 Kitaoka, Satoshi, 1902 Klumperman, B., 1950 Koczkur, Kallum, 1964 Kovalchuk, Anton, 1946 Kunai, Atsutaka, 1980 Lang, Heinrich, 1898 Larsen, S. C., 1920 Lee, Soo-Keun, 1912 Lepre, Anne, 1912 Li, Chao-Jun, 1930 Li, Haoran, 1938 Li, Huanjun, 1988 Li, Liang, 1990 Li, Yahong, 2002 Lin, Xian-Fu, 2006 Liu, Feng, 1934 López-Cardoso, Marcela, 1940 Lu, De-Shui, 2006 Luo, Yumei, 1930 Ma, Dawei, 1934 McKenzie, J. M., 1950 McLeary, J. B., 1950 Mahurin, Shannon M., 1918 Maruyama, Kazuo, 1910 Matsui, Masaki, 1928 Maurer, Jörg, 1900

Miller, Brad, 1964 Mills, Andrew, 1912 Min Park, Sun, 1996 Mitchell, Chris, 2008 Miyajima, Yusuke, 1910 Moss, Rebecca J., 1984 Mukhopadhyay, Sudip, 1948 Nagamori, Masashi, 1928 Nakamura, Hiroyuki, 1910 Nakashima, Kanako, 1978 Nash, Robert J., 1936 Ngaini, Zainab, 1914 Niidome, Takuro, 1978 Niidome, Yasuro, 1978 Nobuoka, Kaoru, 1902 Nocera, Daniel G., 1958 Nonami, Toru, 1924 Nonogawa, Mitsuru, 1926 Numata, Munenori, 1996 Nunn, Michael I. T., 1966 Odom, Aaron L., 2002 O'Hair, Richard A. J., 1944 Ohashi, Fumihiko, 1924 Ohnishi, Yasushi, 1924 Ohshita, Joji, 1980 Overbury, Steven H., 1918 Ozawa, Fumiyuki, 1994 Park, Sung Soo, 1986 Patzschke, Michael, 1982 Peng, Xinsheng, 1964 Penkett, Clive S., 1932 Preite, Marcelo D., 1970 Pritzkow, Hans, 2004 Puntes, Victor F., 1974 Pyykkö, Pekka, 1982 Ramnial, Taramatee, 1972 Rapić, Vladimir, 2004 Reck, Günter, 1946 Reddy, R. Amaranatha, 1898 Redshaw, Carl, 1954 Rein, Régis, 1992 Revell, Jefferson D., 1916 Román-Bravo, Perla, 1940 Roome, Stephen J., 1932 Ruan, Meiling, 1998 Rurack, Knut, 1946 Sanderson, R. D., 1950 Sarkar, Biprajit, 1900 Schulz, Burkhard, 1946 Shi, Jian-lin, 1990, 1998 Shi, Yanhui, 2002 Shi, Zujin, 1988 Shing Chan, Kin, 1922 Shinkai, Seiji, 1996 Shute, Richard E., 1976

Sims, Rupert O., 1932 Skrydstrup, Troels, 1962 Smith, Catherine J., 1976 Solladié, Nathalie, 1992 Song, W., 1920 Su, Gang, 1906 Sugiyasu, Kazunori, 1996 Sun, Hao-Ling, 1906 Szumna, Agnieszka, 1946 Takahashi, Hironobu, 1978 Takei, Toshiaki, 1910 Terayama, Tsuguaki, 1980 Tomov, Atanas K., 1956 Tona, Rolf, 1908 Tonge, M. P., 1950 Torrens, Hugo, 1942 Toyota, Kozo, 1994 Tozawa, Tomokazu, 1904 Tschierske, Carsten, 1898 Veige, Adam S., 1958 Villanueva, Luis, 1942 Wadsworth, Kelly J., 1984 Wang, Congmin, 1938 Wang, Mei-Xiang, 1936 Wang, Yong, 1938 Wang, Zhe-Ming, 1906 Wang, Zhen, 1960 Warford, Lee, 1954 Weißhoff, Hardy, 1946 Winter, Rainer F., 1900 Winters, Ana L., 1936 Wormald, Mark R., 1936 Wu, Chundong, 1998 Wu, Qi, 2006 Xu, Shan-Hao, 2006 Yamaguchi, Ryohei, 1926 Yan, Ji-na, 1990 Yan, Wenfu, 1918 Yao, Shi-Ping, 2006 Yee Kwong, Fuk, 1922 Yoo, Kyesang, 2000 Yoshida, Hiroto, 1980 Yoshifuji, Masaaki, 1994 Yoshimoto, Shoji, 1924 You, Liping, 1988 Yu, Chu-Yi, 1936 Zaher, Damien, 1956 Záliš, Stanislav, 1900 Zaworotko, Michael J., 1889 Zerella, Mark, 1948 Zhang, Shi-Wei, 1906 Zhang, Yuan-Zhu, 1906

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