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Cover

Asymmetric two-center catalysis applied to the direct enantioselective aldol reaction and catalytic cyanosilylation of aldehydes and ketones.



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contents

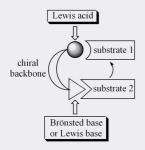
FEATURE ARTICLE



Recent progress in asymmetric two-center catalysis

Masakatsu Shibasaki,* Motomu Kanai and Ken Funabashi

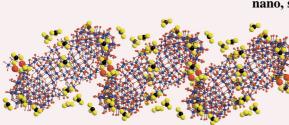
Recent progress using two types of enantioselective two-center catalysts, Lewis acid-Brönsted base and Lewis acid-Lewis base bifunctional complexes, is described.



COMMUNICATIONS

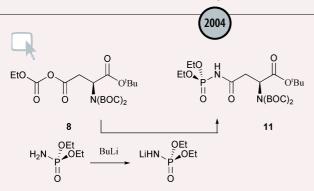


Urea as 'deus ex machina' in giant molybdenum blue type cluster synthesis: an unusual hybrid compound with perspectives for related nano, supramolecular and extended structures



Achim Müller,* Soumyajit Roy, Marc Schmidtmann and Hartmut Bögge

Protonated urea can be used as 'glue' for anionic ring shaped metal—oxide based nanoobjects allowing—due to the comparable complementary hydrogen bonding sites of both—an effective route for the covalent linking of giant anionic clusters.



A new synthesis of phosphoramidates: inhibitors of the key bacterial enzyme aspartate semi-aldehyde dehydrogenase

Luke A. Adams, Russell J. Cox,* Jennifer S. Gibson, M. Belén Mayo-Martín, Magnus Walter and William Whittingham

A new and mild synthesis of phosphoramidates is described: potassium salts of carboxylic acids are treated with ethylchloroformate and the resulting activated anhydride-carbonates are then treated with $LiNHP(O)(OEt)_2$ in situ.

2006



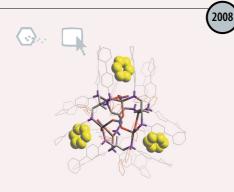
Enhanced activity and stability of Zr-promoted Pd/HZSM-5 catalyst for low-temperature methane combustion

Chun-Kai Shi, Le-Fu Yang,* Xiang-E. He and Jun-Xiu Cai

 $CH_4 + 2O_2 = \frac{1 \text{wt}\% \text{Pd-Zr/HZSM-5}}{1 \text{ atm. } 370 \text{ °C}} = 2H_2O + CO_2$

CH₄ Conversion = 100%; Gas Hourly Space Velocity = 48000 h⁻¹

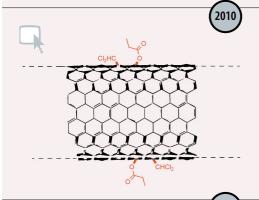
At 370 °C, 1 wt% Pd-Zr/HZSM-5 completely converts CH_4 to CO_2 and H_2O by catalytic combustion and possesses high thermal/hydrothermal stability. Feed composition: CH_4 (vol. 2%), O_2 (vol. 8%) and N_2 balance; $GHSV = 48000 \ h^{-1}$



Assembly of a coordination cage with four aromatic channel receptors on the outside

Philip W. Miller, Mark Nieuwenhuyzen, Xingling Xu and Stuart L. James*

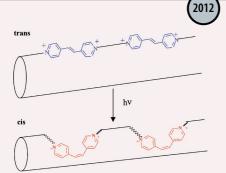
Coordination cages can include guests inside them, but the binding of guests on the exterior is less well explored. Pseudo-tetrahedral silver coordination cages with four aromatic channel receptors on the outside are described.



Sidewall functionalization of single-walled carbon nanotubes through electrophilic addition

Nikos Tagmatarchis, Vasilios Georgakilas, Maurizio Prato* and Hisanori Shinohara*

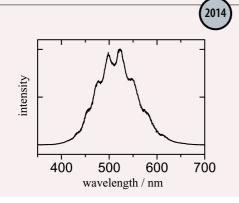
Electrophilic addition of chloroform to SWNTs followed by hydrolysis resulted in the addition of hydroxy groups to the surface of the nanotubes; further esterification with propionyl chloride led to the corresponding ester derivatives, which allowed us to identify their structure, also providing better solubility in organic solvents.



Photochemical modification of the surface area and tortuosity of a *trans*-1,2-bis(4-pyridyl)ethylene periodic mesoporous MCM organosilica

Mercedes Alvaro, Belén Ferrer, Hermenegildo García* and Fernando Rey

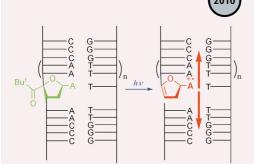
A photoactive MCM-41 that changes its area, pore volume and pore size upon irradiation has been prepared by grafting a stilbene into the walls.



Fine structural photoluminescence spectra of silica-supported zirconium oxide and its photoactivity in direct methane conversion

Hisao Yoshida,* Manohar G. Chaskar, Yuko Kato and Tadashi Hattori

Highly dispersed zirconium oxide on silica exhibits fine structure in phosphorescence emission spectra. The vibration energy of the photoactive Zr–O–Si linkage is estimated to be 955 cm⁻¹. The luminescence species can promote the photoinduced non-oxidative methane coupling at room temperature.



Charge transfer through DNA triggered by site selective charge injection into adenine

Thomas Kendrick and Bernd Giese*

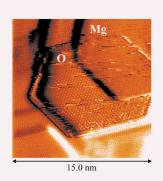
Distance independent charge transfer through DNA double strands via (A:T)_n sequences was observed following site selective charge injection into an adenine.



The direct use of phenyldimethylsilanes in silicon assisted palladium catalysed cross coupling

James C. Anderson,* Stéphane Anguille and Rosalyn Bailey

A vinyl-phenylsilane can be used as a masked vinyl organometallic for cross coupling reactions with phenyl iodide to provide the cine substitution product in high yield.



2020 Atom

Atom resolved evidence for a defective chemisorbed oxygen state at a Mg(0001) surface

Albert F. Carley, Philip R. Davies, Rhys V. Jones, K. R. Harikumar and M. Wyn Roberts

A chemisorbed oxygen state, compatible with $O^{\delta-}$, has been revealed for the first time by scanning tunnelling microscopy at the interface between a (1 \times 1)-O structure and overlying magnesium atoms.



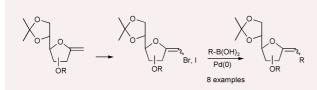
One-pot synthesis of 1-exo-alkylidene-2,3-anhydro furanoses: convenient precursors for exo-glycals and functionalized C-glycals

Ana M. Gómez,* Ana Pedregosa, Serafín Valverde and J. Cristóbal López*

1-*exo*-Methylene-2,3-anhydro furanoses, obtained from *C*-glycals in a one-pot, three step operation can be readily transformed into functionalized *C*-glycals by palladium-catalyzed nucleophilic addition.



A general method for convergent synthesis of functionalized *exo*-glycals based on halogenation and Suzuki cross-coupling of 1-*exo*-methylene sugars



Ana M. Gómez,* Gerardo O. Danelón, Ana Pedregosa, Serafín Valverde and J. Cristóbal López*

Functionalized *exo*-glycals can be readily obtained by palladium catalyzed Suzuki cross-coupling of halo-*exo*-glycals with boronic acids.



oc co

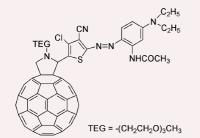
A facile synthetic route to bimetallic Re^I complexes containing two dppz DNA intercalating ligands

Clive Metcalfe, Michelle Webb and Jim A. Thomas*

Using commercially available starting materials, the two-step synthesis of metallo-intercalators with two coplanar dppz ligands is described. Preliminary DNA binding studies indicate that these systems are capable of inter-duplex cross-linking.

2028

Solar cells based on a fullerene-azothiophene dyad



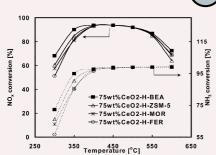
Michele Maggini,* Giorgia Possamai, Enzo Menna, Gianfranco Scorrano, Nadia Camaioni,* Giovanni Ridolfi, Giuseppe Casalbore-Miceli, Lorenzo Franco, Marco Ruzzi and Carlo Corvaja*

Solar cells made from a fulleropyrrolidine functionalized with a thienylazobenzeneamine derivative show power conversion efficiencies up to 0.37% under white light illumination of 80 mW cm⁻² intensity.

Very active CeO₂-zeolite catalysts for NO_x reduction with NH₃

K. Krishna,* G. B. F. Seijger, C. M. van den Bleek and H. P. A. Calis

Selective catalytic reduction of NO with NH₃ over high weight percentage CeO₂zeolites showed excellent NO_x [%] conversions at very high space velocities under simulated exhaust gas conditions in the presence of H₂O.



Starlike nanostructures of polyoxometalates K₃[PMo₁₂O₄₀]·nH₂O synthesized and assembled by an inverse microemulsion method

Xian-Hua Zhang, Su-Yuan Xie,* Zhi-Yuan Jiang, Li-Chuan Zhou, Zhao-Xiong Xie, Rong-Bin Huang and Lan-Sun Zheng

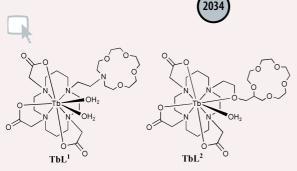
The starlike nanostructure of the polyoxometalates, $K_3[PMo_{12}O_{40}] \cdot nH_2O$, was synthesized in a nonionic inverse microemulsion system, surfactant $(C_{12-18}EO_9)$ /cyclohexane/water.

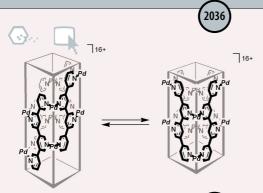


Luminescent terbium(III) complexes with pendant crown ethers responding to alkali metal ions and aromatic antennae in aqueous solution

Cong Li and Wing-Tak Wong*

The maximum amplifications of the luminescence intensity of TbL¹ containing aza-15-crown-5 reached a factor of 47 upon addition of the aromatic antenna sodium p-chlorobenzoate; the luminescence lifetime of TbL² with a pendant 15-crown-5 increased by 65% to 2.95 ms at an [Na⁺] concentration of 0.13 M in aqueous solution.





Spectroscopic and crystallographic studies on the stability of selfassembled coordination nanotubes

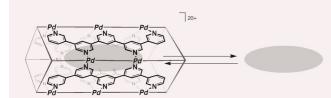
Masaru Aoyagi, Shohei Tashiro, Masahide Tominaga, Kumar Biradha and Makoto Fujita*

Tetrakis(3,5-pyridine) ligand gives two isomers of coordination nanotubes on complexation with (en)Pd(NO₃)₂, one of which is isolated and crystallographically analyzed, while two isomers are shown to be in slow equilibrium despite the presence of sixteen Pd–N bonds in the tube framework.

2038

Dynamic aspects in host-guest complexation by coordination nanotubes

Masahide Tominaga, Shohei Tashiro, Masaru Aoyagi and Makoto Fujita*

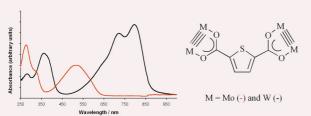


Rod-like guests are in one-dimensional motion within self-assembled coordination nanotubes: they stay in the tubes without flipping along its length at room temperature, but exchange intermolecularly at high temperature.



Thienyl carboxylate ligands bound to M_2 quadruple bonds involving molybdenum and tungsten. Models for dimetallated polythiophenes

Matthew J. Byrnes and Malcolm H. Chisholm*

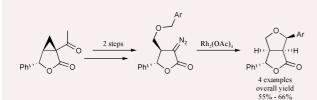


Thienyl-carboxylate and -dicarboxylate groups attached to dinuclear centers ($M=Mo\ or\ W$) having MM quadruple bonds show interesting electronic properties and provide insight into the probable nature of related dimetallated polythiophenes.

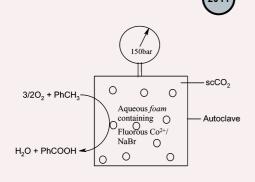


An efficient synthesis of endo, exo-furofuranone derivatives

Nigel A. Swain, Richard C. D. Brown* and Gordon Bruton



The ring openings of 1-acetyl-4-phenyl-3-oxabicyclo[3.1.0]hexane afforded α -acetyl- γ -butyrolactones that underwent a novel diazotransfer reaction, followed by C–H insertion, to provide a series of *endo,exo*-furofuranone analogues.



Aqueous emulsion containing fluorous cobalt species in supercritical ${\rm CO}_2$ for catalytic air oxidation of toluene

Jie Zhu, Alan Robertson and Shik Chi Tsang*

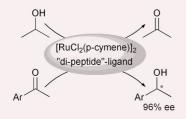
An aqueous emulsion containing ionic Co^{2+} and Br^- species stabilised by fluorous surfactant-like species in supercritical CO_2 –air acts as a *nano-reactor* with excellent interfacial contacts of all necessary hydrophilic/hydrophobic species, which renders safe operation of catalytic aerial oxidation of toluene at high yields without use of acetic acid.



Novel simple and highly modular ligands for efficient asymmetric transfer-hydrogenation of ketones

Isidro M. Pastor, Patrik Västilä and Hans Adolfsson*

Novel simple and highly modular dipeptide-analogue ligands combined with $[RuCl_2(p\text{-cymene})]_2$ were demonstrated to efficiently catalyze the reduction of ketones under hydrogen transfer conditions with enantioselectivities up to 96%.

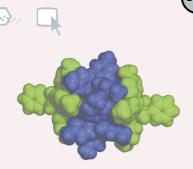




Monocarbaborane anion chemistry. An interesting encapsulation of the $Pd_2I_2\{P(C_6H_4-4-Me)_3\}_4]^{2+}$ cation by a pair of $[PhCB_9H_4I(C_6H_4Me)_4]^-$ anions

Andreas Franken, Colin A. Kilner, Mark Thornton-Pett and John D. Kennedy

The $[Pd_2I_2\{P(C_6H_4-4-Me)_3\}_4]^{2+}$ salt of the $[1-Ph-closo-1-CB_9H_4-10-I-6,7,8,9-(C_6H_4-4-Me)_4]^-$ anion exhibits an unusual neutral supramolecular assembly in the solid state in which the dication is encapsulated by two four-armed 'tetrapus' monoanionic units.

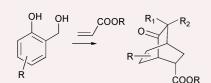


2050

Cycloaddition of cyclohexa-2,4-dienones with electron deficient 2π partners: a novel and stereoselective route to functionalised bicyclo[2,2,2]octenones

Vishwakarma Singh,* Shantanu Pal and Shaikh M. Mobin

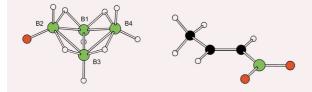
A regio- and stereoselective synthesis of functionalised bicyclo[2.2.2]octanes via cycloaddition of cyclohexadienones and electron deficient 2π partners is described.



2052

Unexpected formation of new fluoroboranes from the reaction of NMe₄B₃H₈ with BF₃ and MeC≡CH: *exo*-2-FB₄H₉ and *trans*-MeCH=CHBF₂

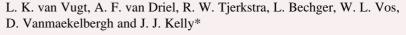
Mark A. Fox, Robert Greatrex* and Daniel L. Ormsby



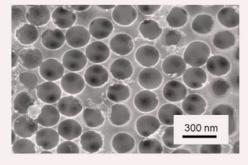
The new fluoroboranes exo-2-FB₄H₉ and trans-MeCH=CHBF₂ have been obtained unexpectedly and in good yield from the reaction of tetramethylammonium octahydrotriborate (NMe₄B₃H₈) with boron trifluoride and propyne (MeC=CH).

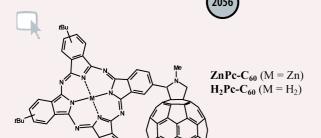


Macroporous germanium by electrochemical deposition



A macroporous germanium-air sphere matrix is obtained by electrodeposition in a template formed from a dried suspension of silica spheres.



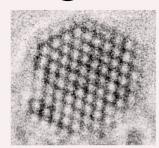


Charge-transfer states in strongly coupled phthalocyanine fullerene ensembles

Dirk M. Guldi,* Andreas Gouloumis, Purificación Vázquez and Tomás Torres*

Fluorescence and transient absorption measurements show that in strongly coupled **ZnPc-C**₆₀ and **H**₂**Pc-C**₆₀ dyads charge-separated states are formed. Large $-\Delta G_{\rm CR}{}^{\circ}$ and small λ assist in stabilising **ZnPc**⁻⁺-**C**₆₀⁻-/**H**₂**Pc**⁻⁺-**C**₆₀⁻-.





Direct formation of hydrogen peroxide from H_2/O_2 using a gold catalyst

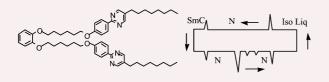
Philip Landon, Paul J. Collier, Adam J. Papworth, Christopher J. Kiely and Graham J. Hutchings*

Supported Au catalysts are very selective for the direct formation of hydrogen peroxide from H_2/O_2 mixtures at 2 °C. The rate of H_2O_2 synthesis is markedly increased if Au–Pd alloy nanoparticles are generated by the addition of Pd.

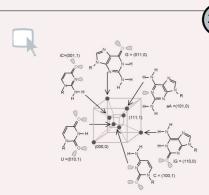


Kinetically induced intermolecular association: unusual enthalpy changes in the nematic phase of a novel dimeric liquid-crystalline molecule

Atsushi Yoshizawa* and Akihisa Yamaguchi



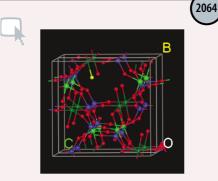
A novel liquid crystal in which two mesogenic groups are connected *via* catechol was found to show unusual enthalpy changes in the nematic phase on heating from the monotropic SmC phase.



A parity code interpretation of nucleotide alphabet composition

Dónall A. Mac Dónaill*

A numerical representation of nucleotide recognition features is proposed. So interpreted, the nucleotide alphabet appears to be structured as a digital parity code, offering a possible explanation of nucleotide alphabet composition.



Synthesis, characterization of an AlPO-CJ₂ analogue containing heteroatomic Eu

Lei Zhang, Caiyi Lu and Yingcai Long*

A microporous aluminophosphate AlPO-CJ2 containing heteroatomic Eu was synthesized and Eu was proved to substitute the Al(v) site.

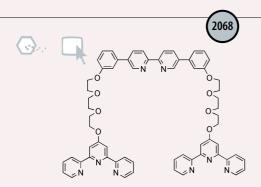


и́НСО₂Ме

A formal total synthesis of leucascandrolide A

Peter Wipf* and Jonathan T. Reeves

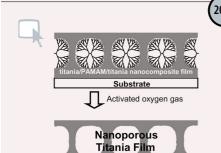
Highlights of this convergent synthesis of the macrocyclic core of the marine macrolide leucascandrolide A include a bi-directional synthesis strategy, an arene–pyran conversion, an alkyne hydrozirconation–transmetalation–aldehyde addition, and a Mitsunobu macrocyclization.



Formation of a [1+1] metallomacrocycle from a heterotritopic ligand containing two terpy and one bipy metal-binding domains

Christopher B. Smith,* Edwin C. Constable,* Catherine E. Housecroft and Benson M. Kariuki

The heterotritopic ligand ${\bf L}$ forms a single 1+1 complex with iron(II) in which only the terpy domains are coordinated.



Replication of dendrimer monolayer as nanopores in titania ultrathin film

Jianguo Huang, Izumi Ichinose and Toyoki Kunitake*

An ultrathin titania film with molecular-sized cavities was synthesized by treating a ca. 8 nm thick $(TiO_2)_3$ (dendrimer) $(TiO_2)_2$ sandwich film with activated oxygen gas.



2072

A novel scandium *ortho*-methoxynitrosobenzene-dimer complex: mechanistic implications for the nitroso-Diels-Alder reaction

Andrew P. Lightfoot, Robin G. Pritchard, Hayley Wan, John E. Warren and Andrew Whiting*

Arylnitroso dienophiles exist in equilibrium with their dimeric counterparts, which in turn form stable bidentate complexes with scandium(III) triflate and react with cyclohexadiene to give the corresponding Diels-Alder adduct at the same rate as the normal thermal process.

ArNO + $Sc(OTf)_3$ TfO CON_A CON_A OTf CON_A OTf CON_A OTf CON_A CON_A OTf CON_A C

Substrate

2074

Polystyrene grafted multi-walled carbon nanotubes

M. S. P. Shaffer* and K. Koziol

Free radical polymerisation of styrene in the presence of an aqueous dispersion of multi-walled carbon nanotubes leads to the formation of a polymer-grafted nanotube product that is compatible with a range of solvents.



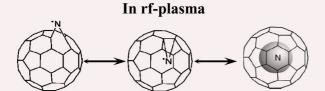
Toluene + grafted nanotubes

Water

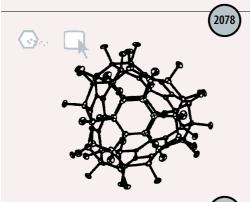


¹⁴N@C₆₀ formation in a nitrogen rf-plasma

Houjin Huang,* Masafumi Ata and Matthias Ramm



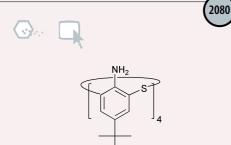
Atomic nitrogen was encapsulated inside a vaporized C₆₀ molecule in rf-plasma, which was confirmed by ESR.



Single crystal X-ray structure of tetrahedral $C_{60}F_{36}$: the most aromatic and distorted fullerene

Peter B. Hitchcock and Roger Taylor*

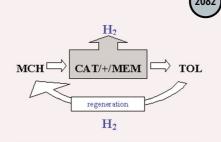
Tetrahedral $C_{60}F_{36}$ is shown by its single-crystal X-ray structure to be the most aromatic (and distorted) fullerene derivative, having four planar hexagons with almost equal bond lengths, the average of which (1.373 Å) is the same as in $C_{60}F_{18}$; one exceptionally long FC–CF bond (1.665 Å) corresponds to the similarly long bond in $C_{60}F_{18}$ (a motif of T $C_{60}F_{36}$) and is likely to be the site of oxygen insertion in $C_{60}F_{36}O$



'Thiacalix[4]aniline' as a highly specific extractant for $Au({\rm III})$ and $Pd({\rm II})$ ions

Hiroshi Katagiri, Nobuhiko Iki,* Yoshiaki Matsunaga, Chizuko Kabuto and Sotaro Miyano*

Thiacalix[4]aniline extracted Au(III) and Pd(II) ions specifically from acidic solutions among 41 metal ions including soft metal ions such as Hg(II), Cd(II), Zn(II), Pb(II) and Cu(II).



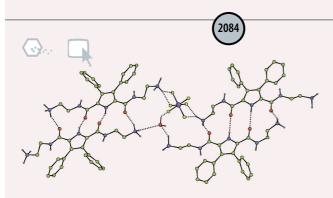
Thiacalix[4]aniline

Hydrogen Storage and Generation Method

Pure hydrogen production from methylcyclohexane using a new high performance membrane reactor

Paloma Ferreira-Aparicio,* Inmaculada Rodríguez-Ramos and Antonio Guerrero-Ruiz

A novel membrane reactor integrated by a Pd-porous stainless steel composite membrane and a Pt/Al₂O₃ catalyst has been developed for H₂ generation from methylcyclohexane (MCH). This reactor–separator assembly constitutes a high efficiency system to obtain the H₂ stored in the form of liquid MCH with yields close to 100%–an interesting alternative for H₂ storage and production.



Pendant arm pyrrolic amide cleft anion receptors

Korakot Navakhun, Philip A. Gale,* Salvatore Camiolo, Mark E. Light and Michael B. Hursthouse

Amine, ammonium and amide pendant arm 2,5-diamidopyrrole cleft species have been synthesized. The pendant arm has been shown to alter the anion affinity of the receptor with the bis-amine receptor showing high selectivity for ${\rm HSO_4}^-$.



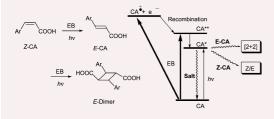
Preparation of oxime oxalate amides and their use in free-radical mediated syntheses of lactams

Eoin M. Scanlan and John C. Walton*

Oxime oxalate amides are a new class of radical precursors that cleanly yield aminoacyl radicals and hence β - and γ -lactams.

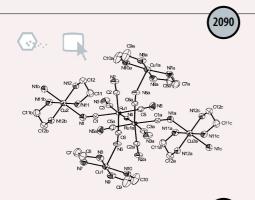


Radiation-induced reactions *via* the lowest excited states in cinnamic acid crystals



Yasunari Maekawa,* Tomonori Inaba, Hiroki Hobo, Tadashi Narita, Hiroshi Koshikawa, Seongyun Moon, Jun Kato and Masaru Yoshida

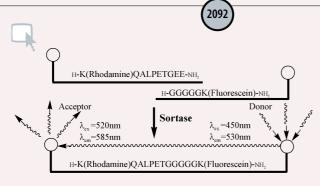
Radiation-induced reactions of cinnamic acid derivatives have been examined and compared with photoreactions in the crystalline state; all the reaction products were exactly the same as those of the photoreactions, indicating that the reactions proceed only via the lowest excited state to give [2+2] cycloadducts, E/Z isomerization products, or starting molecules.



1-D polymer containing the [Ru–N–Ru] μ -nitrido moiety: crystal structure and magnetic properties of $\{[Cu(en)_2]_3[Ru_2N(CN)_{10}]\cdot ClO_4\}_n$ (en = 1,2-diaminoethane)

Xian-Ru Sun, Jiang-Lin Liang, Chi-Ming Che,* Nianyong Zhu, X. X. Zhang and Song Gao

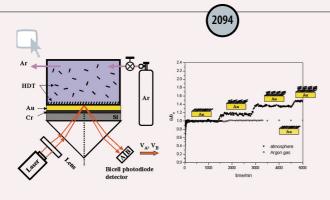
The μ -nitrido–ruthenium unit bridged 1-D coordination polymer was formed from reaction of $K_5[Ru_2N(CN)_{10}]$ with $[Cu(en)_2][(ClO_4)_2]$, characterised by X-ray crystallography and variable temperature susceptibility measurements showed a weak ferromagnetic interaction between the Cu(II) ions in 1-D polymer.



An economical and preparative orthogonal solid phase synthesis of fluorescein and rhodamine derivatized peptides: FRET substrates for the *Staphylococcus aureus* sortase SrtA transpeptidase reaction

Ryan G. Kruger, Patrick Dostal and Dewey G. McCafferty*

A general method of synthesizing peptide substrates containing rhodamine and fluorescein FRET donor/acceptor pairs placed at site-specific locations within a peptide sequence.



In-situ analysis of stepwise self-assembled 1,6-Hexanedithiol multilayers by surface plasmon resonance measurements

Soonwoo Chah, Janos H. Fendler and Jongheop Yi*

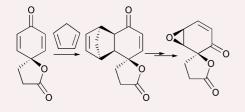
1,6-Hexandithiol (HDT) forms 6.9 ± 1.0 Å thick defect-free monolayers on gold substrates if the solution is purged by argon during the adsorption while long term (>1000 min) exposure of the substrate to alcoholic HDT results in the stepwise formation of multilayers in the absence of argon purging.



Efficient synthesis of a 4,5-epoxy-2-cyclohexen-1-one derivative bearing a spirolactone via a Diels–Alder reaction with high π -facial selectivity: a synthetic study towards scyphostatin

Ryukichi Takagi, Wataru Miyanaga, Yukiko Tamura and Katsuo Ohkata*

The Diels–Alder reaction of spirolactones with cyclopentadiene afforded the adduct with high π -facial selectivity; a hydrophilic analogue of scyphostatin was synthesized from the Diels–Alder adduct.



O₂N OTBDMS OH OH OTBDMS OH OH OTBDMS OF OTBDMS OTBDMS OF OTBDMS OTBDMS OF OTBDMS OF OTBDMS OF OTBDMS OF OTBDMS OTTDMS OTBDMS OTBDMS OTBDMS OTBDMS OTBDMS OTBDMS OTTDMS OTBDMS OTBDMS OTTDMS OTBDMS OTTDMS OTTDMS OTTDMS OTTDMS OTTDMS OTTDMS OTTDM

Zn(ODf)₂: preparation and application in asymmetric alkynylation of aldehydes

Zili Chen, Wennan Xiong and Biao Jiang*

A new Lewis acid, $Zn(ODf)_2$, first prepared from commercially available 3,3,4,4-tetrafluoro[1,2]oxathietane 2,2-dioxide, was used to catalyze highly enantioselective alkynylation of aldehydes in the presence of some ligands to afford the corresponding propargylic alcohols in high yields with up to 99% ee.

4

A new approach to oligonucleotide labelling using Diels–Alder cycloadditions and detection by SERRS

Ljiljana Fruk, Antonio Grondin, W. Ewen Smith and Duncan Graham*

Diels-Alder cycloaddition has been used to attach a benzotriazole maleimide dye to a diene tagged oligonucleotide in an aqueous coupling reaction. This makes the oligonucleotide SERRS active and gives a distinctly different spectrum to that of the maleimide dye.

2102

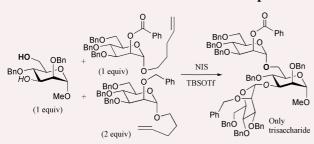
Facile C_{21} functionalization through a novel functional group transfer reaction in $16\alpha,17\alpha$ -epoxy-3 β -hydroxypregn-5-en-20-one and its applications

Navdeep K. Girdhar and Mohan Paul S. Ishar*

An efficient functionalization of C_{21} in preganane series through a novel functional group transfer reaction in a 16,17-epoxy steroid is described.



One pot/two donors/one diol give one differentiated trisaccharide: powerful evidence for reciprocal donor-acceptor selectivity (RDAS)



Bert Fraser-Reid,* J. Cristóbal López,* K. V. Radhakrishnan, M. V. Nandakumar, Ana M. Gómez and Clara Uriel

Three component, one-pot reactions involving equimolar amounts of the acceptor diol and both armed and disarmed donors presented simultaneously, produce a *single* double-differential glycosidation product; this phenomenon provides evidence for Reciprocal Donor Acceptor Selectivity (RDAS).



Structure and chemistry of a zwitterionic amine-aldehyde adduct

Anthony J. Kirby,* Igor V. Komarov,* Vitaliy A. Bilenko, John E. Davies and Jeremy M. Rawson

The first crystal structure of the zwitterionic tetrahedral addition product of a tertiary amino-group to an aldehyde reveals the vital contribution of a molecule of water of solvation.



Urea porphyrins as simple receptors for sugars

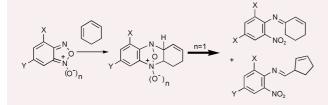
Kalliopi Ladomenou and Richard P. Bonar-Law

Urea-functionalised porphyrins with amino acid side chains are shown to bind carbohydrate derivatives strongly ($10^4 < K < 10^6$) and with unusual selectivity in dichloromethane.



A novel reactivity pattern of nitro-benzofuroxans and -benzofurazans: the heterodienic behaviour of the five-membered ring

Régis Goumont,* Muriel Sebban and François Terrier*

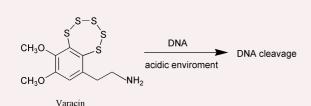


Evidence is presented that the annelated ring of nitrobenzofuroxans and nitrobenzofurazans may act as a heterodiene contributor, thus highlighting the multifaceted reactivity of these compounds.

2112

Acid-accelerated DNA-cleaving activities of antitumor antibiotic varacin

Alex H. F. Lee, Albert S. C. Chan and Tianhu Li*



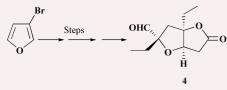
It is demonstrated for the first time in this report that the authentic molecular structure of varacin is capable of causing DNA-cleavage chemically with high efficiency, a process that can be accelerated by its acidic surroundings.



An enantioselective synthetic pathway towards plakortones

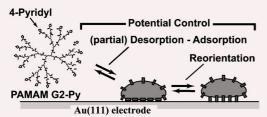
Hing Ken Lee and Henry N. C. Wong*

Enantioselective synthesis of bicyclic lactone **4**, a core structure of plakortones, is described and its absolute configuration was confirmed by an X-ray crystallographic analysis of its precursor **24**.



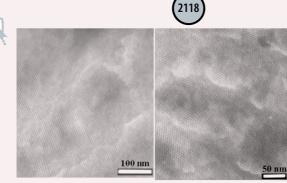


Dynamic dendrimer at electrified interface: potential dependent adsorption—desorption and reorientation of a 4-pyridyl-modified PAMAM dendrimer



Takamasa Sagara,* Kumi Nagata, Hiroaki Tsuruta and Naotoshi Nakashima

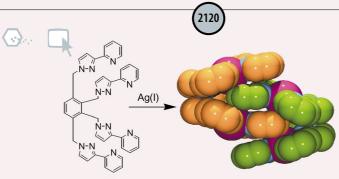
A dendrimer (PAMAM G2), the periphery of which was modified with 4-pyridyl groups, is found to exhibit potential dependent dynamic behaviour on a Au(111) electrode surface.



Periodic mesoporous organosilica from micellar oligomer template solution

Safia Hamoudi and Serge Kaliaguine*

A thick walled highly ordered periodic mesoporous organosilica (PMO) having two-dimensional hexagonal symmetry was first synthesised using a bridged silsesquioxane (CH₃O)₃Si-CH₂-CH₂-Si(CH₃O)₃ as precursor and polyoxyethylene non-ionic surfactant (Brij-56) as template.



A self-complementary molecular cleft

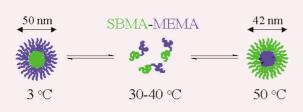
David A. McMorran* and Peter J. Steel

Reaction of the new ligand 1 with silver(I) salts generates a novel self-complementary molecular cleft, which forms dimers in the solid state, stabilised by intermolecular π -stacking interactions and C-H···M interactions.

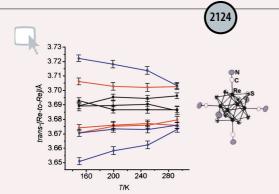
2122

Synthesis and aqueous solution properties of a well-defined thermoresponsive schizophrenic diblock copolymer

J. V. M. Weaver, S. P. Armes* and V. Bütün



Near-monodisperse 'schizophrenic' diblock copolymers based on 2-(N-morpholino)ethyl methacrylate (MEMA) and a sulfobetainized 2-(dimethylamino)ethyl methacrylate (SBMA) were synthesized in very high yield by GTP; these novel thermo-responsive copolymers dissolve molecularly at 30–40 °C, form SBMA-core micelles at 3 °C and form MEMA-core micelles above 50 °C.



Jahn–Teller distortion of the open-shell 23-electron chalcogenide rhenium cluster cores in crystals of the series, $\{[Re_6Q_8]^{3+}(X^-)_6\}^{3-}$ (Q = S, Se; X = Cl, CN)

Stéphane A. Baudron, André Deluzet, Kamal Boubekeur and Patrick Batail*

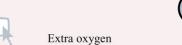
Comparison of 14 precise crystal structures of the Re_6 cluster cores at different low temperatures reveals that the open-shell (23 electron) cores undergo a Jahn–Teller distortion of their parent 24 electron octahedral cores and that $D_{4\rm h}$ and $D_{2\rm h}$ forms may co-exist in the solid state.



First hexadecavanadate compound: hydrothermal synthesis and characterization of a three-dimensional framework [{Cu(1,2-pn)_2}_7{V_{16}O_{38}(H_2O)}_2]\cdot 4H_2O

Bi-Zhou Lin* and Shi-Xiong Liu

Each new mixed-valence $[V_{16}O_{38}(H_2O)]^{7-}$ cluster is connected with seven others through seven bridging $[Cu(1,2-pn)_2]^{2+}$ groups into a three-dimensional open-framework structure.



in the

framework



Evidence of surface reconstructions and incorporation of oxygen into the oxide framework on the hydroxylated La₂O₃ $\{001\}$ surface

Maria Alfredsson,* C. Richard A. Catlow, Anastasia Paulidou and Roger M. Nix

By performing *first-principles* Molecular Dynamics simulations at 300 K, we show that water dissociation on the $A-La_2O_3\{001\}$ surface is associated with a surface reconstruction, yielding a surface structure that is oxygen rich.

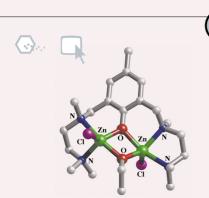


Reductive decomplexation of π -allyltricarbonyliron lactone complexes; a new route to stereo-defined 1,7-diols and 2,3-diene-1,7-diols

Christopher J. Hollowood and Steven V. Ley*

$$\begin{array}{c} \text{Fe(CO)}_3 \\ \text{H} \\ \text{C}_6\text{H}_{11} \\ \text{R} \end{array} \\ \begin{array}{c} \text{(i) Li naphthalenide, THF} - 78^{\circ}\text{C to rt;} \\ \text{(ii) H}_2, \text{Pd/C, EtOAc, }92\%. \\ \\ \text{C}_6\text{H}_{11} \\ \text{R} \end{array}$$

Treatment of π -allyltricarbonyliron lactone complexes bearing an adjacent leaving group, with lithium naphthalenide causes decomplexation to acyclic dienols in excellent yield and without any scrambling of the allylic centre.



 $R^1 = H \text{ or } Ac$

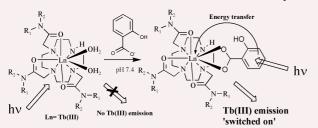
Metalloenzyme inspired dizinc catalyst for the polymerization of lactide

Charlotte K. Williams, Neil R. Brooks, Marc A. Hillmyer* and William B. Tolman*

A new dizinc-monoalkoxide complex supported by a dinucleating ligand was structurally characterized and shown to be a highly active catalyst for the controlled polymerization of lactide.



Delayed lanthanide luminescence sensing of aromatic carboxylates using heptadentate triamide $Tb({\sc iii})$ cyclen complexes: the recognition of salicylic acid in water



Thorfinnur Gunnlaugsson,* Andrew J. Harte, Joseph P. Leonard and Mark Nieuwenhuyzen

The coordinately unsaturated Tb complexes possess two labile metal-bound water molecules that can be displaced upon metal chelation to aromatic carboxylic anions such as salicylic acid in water, which gives rise to large enhancements in the Tb(III) luminescence.



Rapid hydrolytic cleavage of the mRNA model compound HPNP by glycine based macrocyclic lanthanide ribonuclease mimics

Thorfinnur Gunnlaugsson,* R. Jeremy H. Davies, Mark Nieuwenhuyzen, Clarke S. Stevenson, Romain Viguier and Sinead Mulready

The lanthanide ion based macrocyclic complexes **Ln·1** mimic the hydrophobic nature of ribonucleases, where the lanthanide ion induce the formation of a hydrophobic cavity in **1**, which gives rise to a large order of magnitude enhancement in the hydrolytic cleavage of HPNP under physiological conditions.

2138

Carbolithiation of aromatic rings: cyclohexadienes from N-aroyl-2,2,6,6-tetramethylpiperidines

Jonathan Clayden,* Yann J. Y. Foricher and Ho Kam Lam

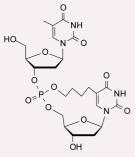
Instead of promoting ortholithiation, a tetramethylpiperidine-derived amide group directs organolithiums to attack aromatic rings, leading to dearomatisation.

2140

Tandem ring-closing metathesis and hydrogenation towards cyclic dinucleotides

Philip Børsting and Poul Nielsen*

A new synthetic concept in the construction of conformationally restricted nucleic acid model structures is presented. Stable saturared cyclic phosphotriester structures are obtained using a ruthenium based precatalyst for both RCM and hydrogenation.

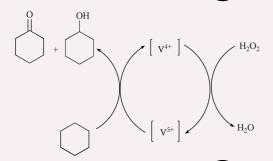


2142

A highly efficient oxidation of cyclohexane over VPO catalysts using hydrogen peroxide

Unnikrishnan R. Pillai and Endalkachew Sahle-Demessie*

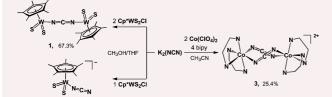
A highly efficient oxidation of cyclohexane to cyclohexanol and cyclohexanone is accomplished over calcined vanadium phosphorus oxide (VPO) catalysts in a relatively mild condition using hydrogen peroxide under a nitrogen atmosphere.



2144

Use of dipotassium cyanamide for the synthesis of cyanoimido (NCN²⁻) complexes of tungsten and cobalt

Rong Cao and Kazuyuki Tatsumi*



Three cyanoimido complexes synthesized from dipotassium cyanamide, $(Cp*WS_2)_2(\mu-NCN)$ (1), $K[(Cp*WS_2)(NCN)]$ (2) and $\{[(bipy)_2Co]_2(\mu-NCN)_2\}(ClO_4)_2$ (3), and their X-ray derived structures are reported.



Transfer of alk-1-enyl group from boron to aluminium: a novel way to prepare (E)-alk-1-enyldiisobutylalanes

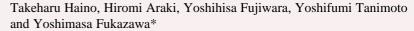
Masayuki Hoshi* and Kazuya Shirakawa

$$\begin{array}{c|c} & i\text{-Bu}_2\text{AlH} \\ & \downarrow \\ \text{H} & C = C \\ \text{H} & R \\ & \text{r.t., 2h} \\ \end{array} \begin{array}{c} i\text{-Bu}_2\text{Al} \\ & \downarrow \\ \text{R} \\ \end{array}$$

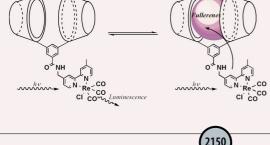
Treatment of (E)-alk-1-enyldicyclohexylboranes $\mathbf 1$ with diisobutylaluminium hydride (DIBAL-H) in the presence of hex-1-ene at room temperature results in transfer of the alk-1-enyl group from boron to aluminium to give (E)-alk-1-enyldiisobutylalanes $\mathbf 2$ with retention of configuration at the double bond.



Fullerene sensors based on calix[5]arene



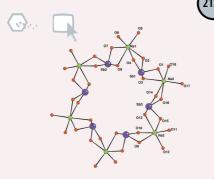
The combination of supramolecular concepts and luminescence techniques is the key for the successful generation of selective supramolecular sensors. A new class of fullerene sensors based on calix[5]arenes has produced the highly sensitive detection of C_{60} and C_{70} .



A twenty-four membered mixed-metal macrocycle; Synthesis and structure of *cyclo*-[(3-Me-1,2-C₆H₃O₂)₂SbNa(THF)₂]₆

Michael A. Paver,* Jonathan S. Joy and Michael B. Hursthouse

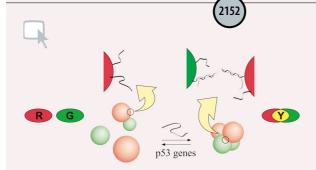
The structure and synthesis of the first example of a twenty-four membered mixed p-/s-block macrocycle, *cyclo*-[(3-Me-1,2-C₆H₃O₂)₂SbNa(THF)₂]₆ is reported, consisting of a cyclic array of alternating Sb and Na metal centres doubly bridged by catecholato groups.



${\bf Colorimetric\ SNP\ analysis\ using\ oligonucleotide-modified\ nanoparticles}$

Toshihiro Ihara,* Yasushi Chikaura, Shojiro Tanaka and Akinori Jyo

Novel colorimetry for gene analysis has been developed using selective aggregation of DNA-modified nanospheres. The signal was observed by fluorescence microscopy and FRET.



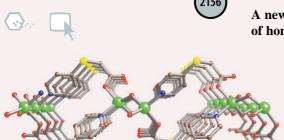
0.03 0.02 0.01 0 5 10 15 20

Time (µs)

Increasing the life expectancy of carbanions by zeolite inclusion

Michelle N. Chrétien, Gonzalo Cosa, Hermenegildo García* and J. C. Scaiano*

Carbanions are long lived in zeolites, where they show Grignard-like behaviour.



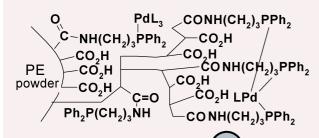
A new Zn(II) coordination polymer with 4-pyridylthioacetate: assemblies of homo-chiral helices with sulfide sites

Mitsuru Kondo,* Makoto Miyazawa, Yasuhiko Irie, Reiko Shinagawa, Tomonori Horiba, Akira Nakamura, Tetsuyoshi Naito, Kenji Maeda, Shunji Utsuno and Fumio Uchida

The reaction of $Zn(NO_3)_2 \cdot 6H_2O$ with 4-pyridylthioacetic acid (Hpyta) in the presence of triethylamine produces a new coordination polymer [Zn(pyta)(OH)], which is constructed by the alternating assembly of two types of homo-chiral helical columns.

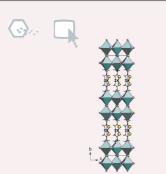
2158

Functionalized hyperbranched grafts on polyethylene powder for support of $Pd(\theta)$ -phosphine catalyst



David E. Bergbreiter,* Andrew M. Kippenberger and Guoliang Tao

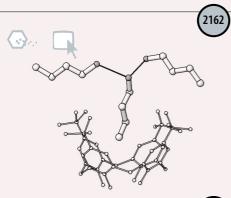
Poly(acrylic acid) grafts on polyethylene (PE) powder are converted to uniformly loaded insoluble polymeric phosphine ligands (loadings of >0.1 mmol g $^{-1}$) that complex Pd(0) catalysts useful in allylic substitution chemistry. Such supported catalysts can be reused without detectable Pd leaching.



$(C_4H_3SCH_2NH_3)_2(CH_3NH_3)Pb_2I_7$: non-centrosymmetrical crystal structure of a bilayer hybrid perovskite

Xu-Hui Zhu, Nicolas Mercier,* Amédée Riou, Philippe Blanchard and Pierre Frère

The analysis of the crystal stucture of $(C_4H_3SCH_2NH_3)_2(CH_3NH_3)Pb_2I_7$, displaying a well-ordered acentric inorganic $Pb_2I_7^{3-}$ bilayer, is reported, and compared to the related monolayer hybrid perovskite.



Pseudopolymorphism in the p-tert-butylcalix[4]arene—n-butylamine system: directing the structural motifs

Konstantin A. Udachin, Gary D. Enright, Philip O. Brown and John A. Ripmeester*

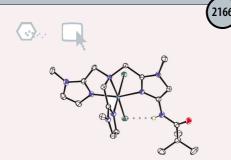
p-tert-Butylcalix[4]arene forms three compounds with n-butylamine, two weakly interacting guest–host compounds of 1:1 and 1:2 stoichiometry, as well as a hydrogen-bonded 3:1 compound containing some n-butylamine molecules not coordinated to the host.



Spirometallodendrimers: terpyridine-based *intra*macromolecular cyclization upon complexation

George R. Newkome,* Kyung Soo Yoo and Charles N. Moorefield

Intramacromolecular cyclization *via* terpyridine–metal–terpyridine complex formation has facilitated the creation of dentrimer-based spiranes.



Structure and properties of an Fe(III) complex containing a novel amide functionalized polyimidazole ligand

Lionel E. Cheruzel, Jianping Wang, Mark S. Mashuta and Robert M. Buchanan*

A novel amide functionalized polyimidazole tripod ligand has been synthesized and used to prepare a mononuclear Fe(III) complex that has been characterized by X-ray crystallography and other physical methods.

2168

A coordination network containing metal—organometallic secondary building units based on π -bonded benzoquinone complexes

Moonhyun Oh, Gene B. Carpenter and Dwight A. Sweigart*

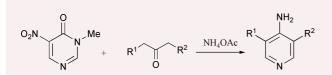
Mn(CO)₃

Thermal treatment of $(\eta^5$ -semiquinone)Mn(CO) $_3$ and Mn(OAc) $_2$ in MeOH–DMSO produces a neutral 3D metal–organometallic network (MOMN) consisting of pairs of Mn $^{2+}$ ions linked by $(\eta^4$ -benzoquinone)Mn(CO) $_3$ $^-$ spacers.

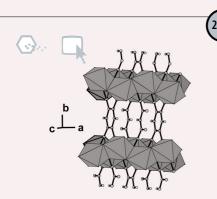
2170

Facile synthesis of functionalized 4-aminopyridines

Nagatoshi Nishiwaki,* Mayumi Azuma, Mina Tamura, Kazushige Hori, Yasuo Tohda and Masahiro Ariga*



Functionalized 4-aminopyridines are readily available by ring transformation by ring transformation of nitropyrimidinone with active methylene compounds in the presence of ammonium acetate.



[Fe $_2$ (C $_{10}$ O $_8$ H $_2$)]: An antiferromagnetic 3D iron(II) carboxylate built from ferromagnetic edge-sharing octahedral chains (MIL-62)

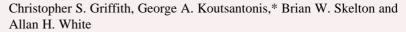
M. Sanselme, J. M. Grenèche, M. Riou-Cavellec* and G. Férey*

MIL-62 or [Fe₂(C₁₀O₈H₂)] is the first three-dimensional iron hybrid compound based on ferromagnetic chains coupled antiferromagnetically by π -delocalized 1,2,4,5-benzenetetracarboxylates.

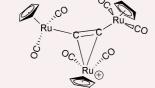


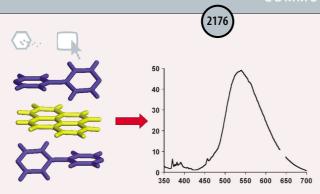
2174

Reactions of metalloal kynes. New \mathbf{C}_2 bonding mode in a trimetallic complex



A trimetallic cationic complex not containing metal–metal bonds has been synthesised which represents a new type of bonding mode for the ${\rm C_2}^{2-}$ ligand. Our investigation is suggestive of a labile ligand–metal interaction appearing to involve fluxional motion of the ${\rm C_2}^{2-}$ ligand about the bimetallic core.



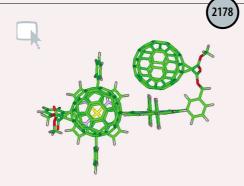


Exciplex fluorescence of

 ${[Zn(bipy)_{1.5}(NO_3)_2]}\cdot CH_3OH\cdot 0.5pyrene_n$: a coordination polymer containing intercalated pyrene molecules (bipy = 4,4'-bipyridine)

Brian D. Wagner,* Gregory J. McManus, Brian Moulton and Michael J. Zaworotko*

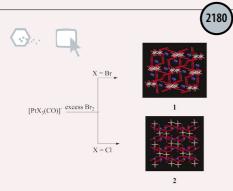
Pyrene-bipyridine exciplex fluorescence is used to probe the polarity of the cavities in a ladder coordination polymer.



Strong intramolecular chromophore interactions in novel bis([60]fullerene)-oligoporphyrin nanoarrays

Davide Bonifazi and François Diederich*

The synthesis of novel nanodimensional dyads was accomplished by Bingel reaction of a series of meso, meso-linked oligoporphyrin bis-malonates with C_{60} . Distinct conformational effects, together with changes in the porphyrin-centred UV-Vis absorption and a large quenching of the porphyrin fluorescence provide evidence for strong fullerene–porphyrin interactions in these systems.



Polymeric anionic networks using dibromine as a crosslinker; the preparation and crystal structure of $[(C_4H_9)_4N]_2[Pt_2Br_{10}]\cdot(Br_2)_7$ and $[(C_4H_9)_4N]_2[PtBr_4Cl_2]\cdot(Br_2)_6$

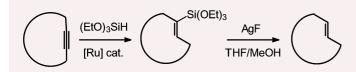
Michael Berkei, Jamie F. Bickley, Brian T. Heaton* and Alexander Steiner

The reaction of M[PtX₃(CO)] ($M^+ = [(C_4H_9)_4N]^+, X = Br$, Cl) with an excess of Br₂ gives the new platinum(IV) salts, $[(C_4H_9)_4N]_2[Pt_2Br_{10}] \cdot (Br_2)_7$, **1**, and $[(C_4H_9)_4N]_2[PtBr_4Cl_2] \cdot (Br_2)_6$, **2**, which, in the solid state, contain strong Br Br interactions resulting in the formation of polymeric networks.

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A chemo- and stereoselective reduction of cycloal kynes to (E)-cycloal kenes

Alois Fürstner* and Karin Radkowski



A sequence comprising a ruthenium catalyzed hydrosilylation followed by a protodesilylation reaction mediated by AgF allows for the mild and highly stereoselective conversion of cycloalkynes into (*E*)-cycloalkenes.

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