

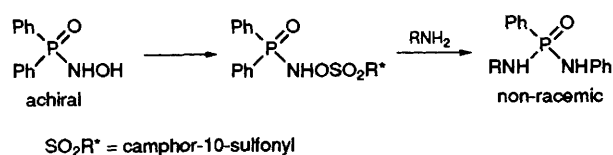
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Chemical Communications

Number 14
1994

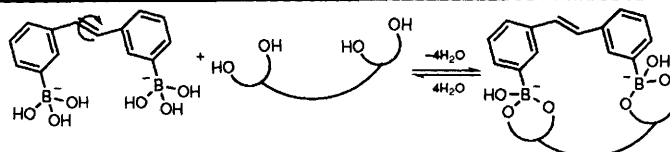
CONTENTS

- 1619 **Asymmetric Induction in the Base Induced Rearrangement of *N*-(Diphenylphosphinoyl)-*O*-(camphor-10-sulfonyl)hydroxylamine**



Martin J. P. Harger, Ramesh Sreedharan-Menon

- 1621 **Specific Recognition of Disaccharides by *trans*-3,3'-Stilbenediboronic Acid: Rigidification and Fluorescence Enhancement of the Stilbene Skeleton upon Formation of a Sugar–Stilbene Macrocycle**

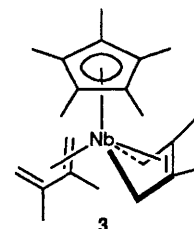


The first known fluorescent sensor for disaccharides is rationally designed using the rigidification of the stilbenediboronic acid skeleton upon disaccharide binding. Fluorescence enhancement of 3,3'-stilbenediboronic in basic aqueous media shows very high specificity for disaccharides against monosaccharides.

K. R. A. Samankumara Sandanayake, Kazuaki Nakashima, Seiji Shinkai

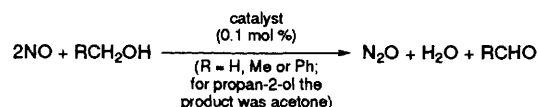
- 1623 **Polyethylene with Extremely Narrow Polydispersity obtained from the New Catalyst Systems Nb(η^5 -C₅Me₅)(η^4 -diene)Cl₂-MAO and Nb(η^5 -C₅Me₅)(η^4 -diene)₂-MAO**

Polyethylene with highly narrow dispersities (M_w/M_n as low as 1.05) has been obtained by use of the niobium complexes [Nb(η^5 -C₅Me₅)(η^4 -diene)Cl₂] **1** and **3** in the presence of a large excess of methylaluminoxane.



Kazushi Mashima, Shinjiro Fujikawa, Hisao Urata, Eiji Tanaka, Akira Nakamura

- 1625 **Catalytic Reduction of Nitric Oxide to Nitrous Oxide by Alcohols mediated by Copper(I) Complexes**



In a reaction relevant to environmentally important processes carried out by copper-containing enzymes and heterogeneous catalysts, Cu^I complexes of 1,4,7-triisopropyl-1,4,7-triazacyclononane dissolved in alcohols efficiently promote the reduction of NO to N₂O by the solvent at room temperature, yielding the respective carbonyl compounds.

Samiran Mahapatra, Jason A. Halfen, William B. Tolman

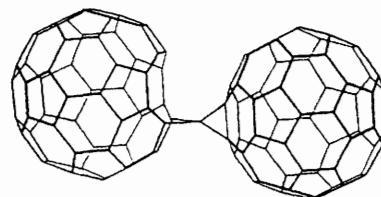
- 1627 **A New Halide-free Route to Metallo-organic Cadmium Complexes; Syntheses and Structural Characterisations of $[\text{Cd}(\text{PhC}\equiv\text{C})_2(\text{tmeda})]$ and $[\text{Cd}(\text{C}_{12}\text{H}_8\text{N}_4)]^{2-}\cdot 2\text{Li}(\text{thf})_4^+$ ($\text{tmeda} = \text{Me}_2\text{NCH}_2\text{CH}_2\text{NMe}_2$)**

Donald Barr, Andrew J. Edwards, Paul R. Raithby, Moira-Ann Rennie, Kerry Verhorevoort, Dominic S. Wright

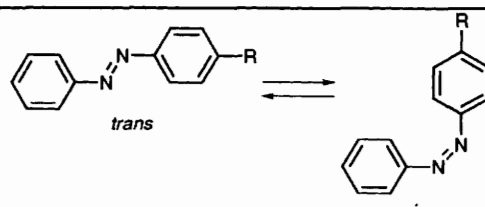
Nucleophilic substitution of $\text{Cd}(\text{NSiMe}_3)_2$ with various anions is an effective and simple halide-free route to a variety of metallo-organic cadmium complexes. This new synthetic strategy is exemplified by the syntheses of $[\text{Cd}(\text{PhC}\equiv\text{C})_2(\text{tmeda})]$ **1**, the first structurally characterised cadmium acetylide complex, and $[\text{Cd}(\text{carbazoly})_4]^{2-}\cdot 2[\text{Li}(\text{thf})_4]^+$ **2**, containing the first structurally characterised amido cadmium anion, by this route.

- 1629 **Is C_{119} a Spirane and the First Fullerene to Contain a Four-membered Ring?**

Roger Taylor



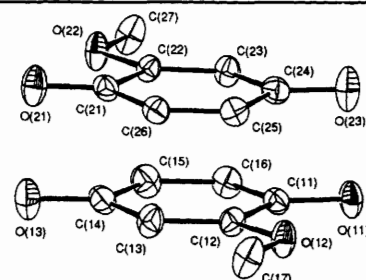
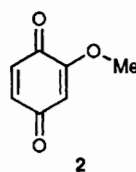
- 1631 **A Stable *cis*-Azobenzene in Aqueous Solution**



Eric J. Chambers, Ian S. Haworth

In the *cis-trans* equilibrium of azobenzene, for $\text{R} = \text{CH}_2\text{OCO}$. Pro.Leu and CH_2OCO .Pro.Leu.Gly.Pro.D-Arg at least one third of molecules are present in the *cis* form in water.

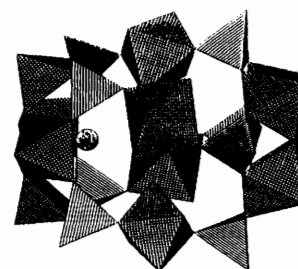
- 1633 **The Crystal Structure of 2-Methoxy-1,4-benzoquinone: Molecular Recognition involving Intermolecular Dipole-Dipole- and C-H \cdots O Hydrogen Bond Interactions**



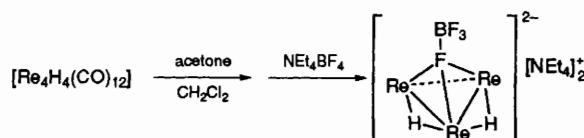
Erik M. D. Keegstra, Anthony L. Spek, Jan W. Zwikker, Leonardus W. Jenneskens

- 1635 **Investigations of the Vanadium-oxo-organophosphonato System: Preparation and Structural Characterization of a Mixed-valence V^V-V^{IV} Cluster encapsulating Chloride Anions, $(\text{Bu}_4\text{N})_2\text{[(V}_8\text{O}_{16})\{\text{V}_4\text{O}_4(\text{H}_2\text{O})_{12}\}(\text{PhPO}_3)_8\text{Cl}_2]\cdot 2\text{Et}_2\text{O}\cdot 2\text{MeOH}\cdot 4\text{H}_2\text{O}$**

Qin Chen, Jon Zubieta

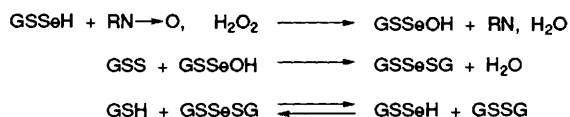


- 1637 **Novel Coordination Mode of Boron Tetrafluoride Anion: Structure of a BF_4^- -capped Trirhenium Cluster: $[\text{NEt}_4]^+{}_2[\text{Re}_3\text{H}_2(\text{CO})_9\text{BF}_4]^{2-}$**



C. S. Yang, H. C. Horng, F. L. Liao, C. P. Cheng

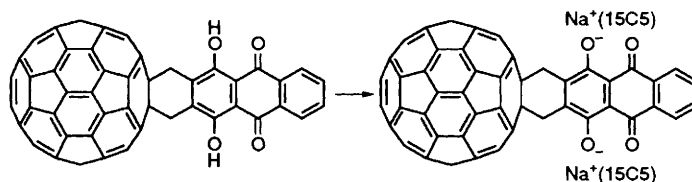
1639 **Reduction of Resazurin by Glutathione activated by Sulfanes and Selenite**



Walter A. Prütz

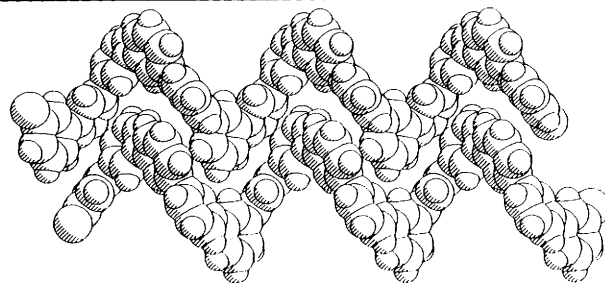
Catalytic reduction of resazurin or H_2O_2 by the seleno-persulfide of GSH.

1641 **A Diels–Alder Adduct of C_{60} containing Hydroxyquinone Functionalities**



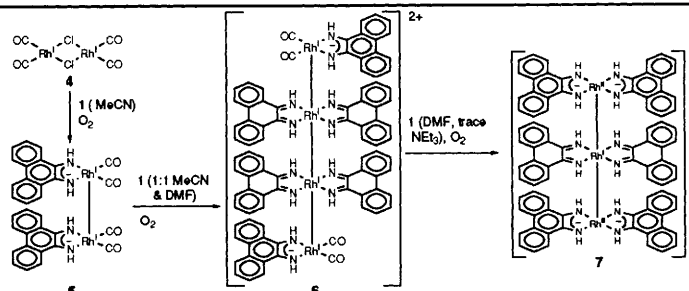
Wolfgang Bidell, Richard E. Douthwaite, Malcolm L. H. Green, Adam H. H. Stephens, John F. C. Turner

1643 **The Structure of Poly(*m*-Phenylene): a Prediction from Single-crystal X-Ray Studies of *m*-Deciphenyl and *m*-Undeciphenyl**



David J. Williams, Howard M. Colquhoun, Caroline A. O'Mahoney

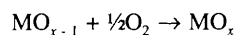
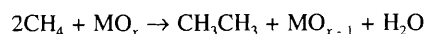
1645 **Unique Eclipsed Cofacial Oligomeric Complexes of Rhodium containing Three-layered Bonding of 9,10-Phenanthroquinonediimine**



Shuenn-Shing Chern, Gene-Hsiang Lee, Shie-Ming Peng

1647 **Oxidative Coupling of Methane on an ABO_3 Type Oxide with Mixed Conductivity**

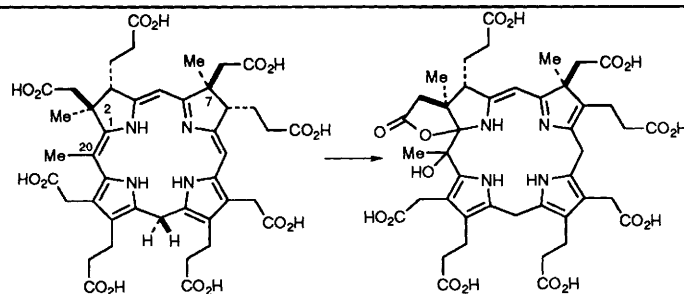
Substituted SrCoO_3 δ -perovskite oxide showed high selectivity (> 98%) and stable activity for oxidative coupling of methane at 1023 K in the redox system shown below (MO_x = oxide).



Kohji Omata, Osamu Yamazaki, Kazuyuki Tomita, Kaoru Fujimoto

The catalyst showed a high tolerance to a reductive atmosphere.

1649 **Biosynthesis of Vitamin B_{12} : Studies of the Oxidative and Lactone-forming Steps by ^{18}O -Labelling**

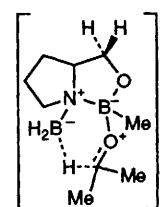


Alex I. D. Alanine, Yongfu Li, N. Patrick J. Stamford, Finian J. Leeper, Francis Blanche, Laurent Debussche, Alan R. Battersby

1651 **Computational Elucidation of the Catalytic Mechanism for Ketone Reduction by an Oxazaborolidine–Borane Adduct**

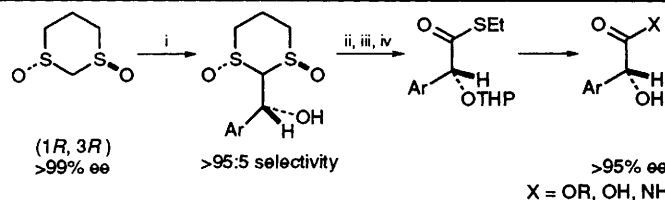
Lynda P. Linney, Christopher R. Self, Ian H. Williams

Semiempirical characterisation of transition state structures and reaction pathways for catalytic cycle.



1653 ***trans*-1,3-Dithiane-1,3-Dioxide, a New Chiral Acyl Anion Equivalent for the Preparation of Masked Activated Acids: Application to the Synthesis of α -Hydroxy Acid Derivatives**

Varinder K. Aggarwal, Abraham Thomas, Richard J. Franklin



i, NaHMDS, RCHO, THF, py; ii, DHP, CH₂Cl₂, TsOH (cat.); iii, TFAA, py, CH₂Cl₂, 0 °C, 15 min; iv, LiOH, EtSH, THF, H₂O, 0 °C to room temp.

1655 **New Actinide Hydrogen Transition Metal Compounds. Synthesis of [K(C₁₂H₂₄O₆)][(η -C₅Me₅)₂(Cl)UH₆Re-(PPh₃)₂] and the Crystal Structure of its Benzene Solvate**

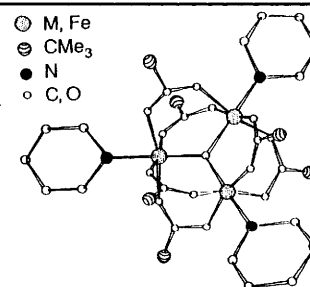
Sophie M. Cendrowski-Guillaume, Monique Lance, Martine Nierlich, Julien Vigner, Michel Ephritikhine

The title compound, which has been synthesized by the reaction of [K(C₁₂H₂₄O₆)][(PPh₃)₂ReH₆] with [{(η -C₅Me₅)₂U(μ -Cl)]₃] is a unique example of an anionic heterobimetallic polyhydride, the first complex exhibiting a transition metal and an actinide(III) in close proximity, and the sole 5f element–hydrogen–transition metal compound to have been crystallographically characterized. This compound has been oxidised by TIBPh₄ into [(η -C₅Me₅)₂(Cl)UH₆Re(PPh₃)₂].

1657 **Electron Transfer Rates in a Trinuclear Mixed-valence Iron(III,III,II) Molecule: A Variable-temperature Infrared Spectroscopic Study**

Ruowen Wu, Samuel K. Arap Koske, Ross P. White, Christopher E. Anson, Upali A. Jayasooriya, Roderick D. Cannon

The rate of iron(II)–iron(III) electron transfer in the complex [Fe₃O(OCCMe₃)₆(py)₃] is estimated as $5 \times 10^{11} \text{ s}^{-1}$ at 300 K, by infrared line broadening.



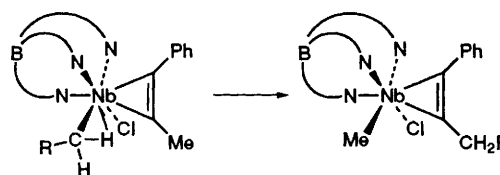
1659 **Dibenzo[*a,e*]cyclooctene: a Nonplanar Radical Cation**

Fabian Gerson, Patrick Felder, Reto Schmidlin, Henry N. C. Wong

According to its ESR hyperfine data, the radical cation of dibenzo[*a,e*]cyclooctene has a tub-shaped geometry. In this respect it resembles the neutral molecule, but differs from the planar radical anion. These structural features are shared with the radical ions of cyclooctatetraene.

1661 **Unprecedented Metathesis of Metal–Carbon and Carbon–Carbon Bonds in α -Agostic *n*-Alkyl Niobium Alkyne Complexes**

Michel Etienne, Fabienne Biasotto, René Mathieu

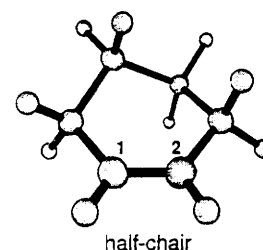


$$\Delta H^\ddagger = 113 \text{ kJmol}^{-1}; \Delta S^\ddagger = 4 \text{ J K}^{-1} \text{ mol}^{-1}$$

1663 **Boat versus Half-chair Cyclohexyl Rings: Determinants of Conformational Preference**

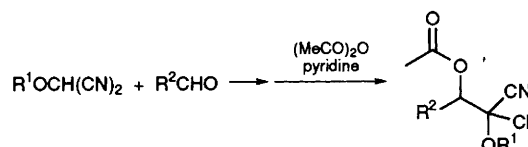
Scott McN. Sieburth

For partially flattened six-membered rings, bond angles around C(1) and C(2) of $< 115^\circ$ lead to a greater stability for the boat than the half-chair conformation.



1665 **Palladium Catalysed Addition of Masked Formyl Cyanides $\text{ROCH}(\text{CN})_2$ to Aldehydes**

Hisao Nemoto, Yasufumi Kubota, Yoshinori Yamamoto



1667 **Synthesis and Catalytic Properties of Titanium-substituted Silicoaluminophosphate TAPSO-5**

A. Tuel, Y. Ben Taârit

The titanium substituted silicoaluminophosphate TAPSO-5 with the AFI structure has been synthesised hydrothermally with Ti/Si ratios up to 0.08 in the as-synthesised product. These materials show interesting properties in the epoxidation of large molecules with alkyl hydroperoxides under mild conditions. The activity as well as the selectivities depend strongly on the nature of the peroxide.

1669 **New Ligands for Complexation of Lanthanoids: The Synthesis and Structures of a Nonadentate Schiff-base Ligand (L^1) and of the Complexes $[\text{ML}^1(\text{OH}_2)](\text{ClO}_4)_3 \cdot 3\text{MeNO}_2$ ($M = \text{La}, \text{Pr}$) and $[\text{YL}^1](\text{ClO}_4)_3 \cdot 3\text{MeCN}$**

Stephen J. Archibald, Alexander J. Blake, Martin Schröder, Richard E. P. Winpenny

The synthesis and structure of a new nonadentate Schiff base ligand and of its complexes with M^{III} ions are reported. For $M = \text{La}$ or Pr the metals are ten-coordinate while for $M = \text{Y}$ the metal is nine-coordinate.

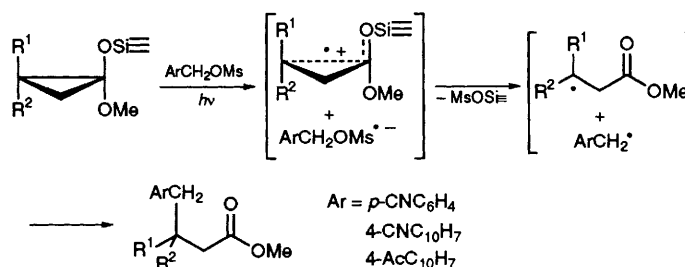
1671 **Micropore Structure of Zeolite MCM-22 as determined by the Decane Catalytic Test Reaction**

Wim Souverijns, Wim Verrelst, Gina Vanbutsele, Johan A. Martens, Pierre A. Jacobs

According to the decane catalytic test reaction, the pore architecture of the zeolite MCM-22, whose topology is unknown, contains two micropore systems circumscribed by 10- and 12-rings. The 12-ring channel is monodimensional and has no connections with the 10-ring pores. The strongest acid sites are located in the 10-ring channels.

1673 **Photoinduced Electron Transfer Reaction of Cyclopropanone Acetals with Arylmethyl Methanesulfonate: Generation of β -Keto Radical Species and Application to C-C Bond Formation**

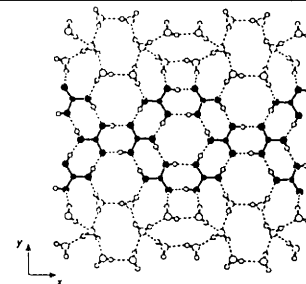
Manabu Abe, Akira Oku



1675 **Brønsted Conjugate Acid–Base Species $B(OH)_3/[BO(OH)_2]^-$ Coexist in the Crystalline Solid $(NEt_4)_2[BO(OH)_2]_2 \cdot B(OH)_3 \cdot 5H_2O$**

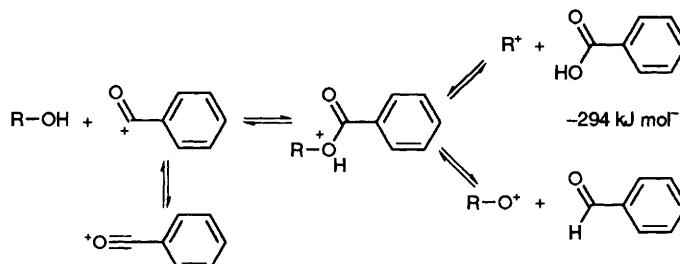
Clemens C. Freyhardt, Michael Wiebcke

In the title compound, which crystallizes in the ternary system $(NEt_4)_2O-B_2O_3-H_2O$ from strongly basic solution, extensive hydrogen bonding links the species $B(OH)_3$, $[BO(OH)_2]^-$ and H_2O into two-dimensional layers that are interleaved by NEt_4^+ cations.



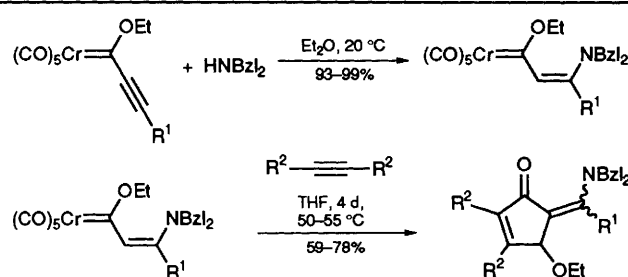
1677 **Selective Gas-phase Ion–Molecule Reactions of the Benzoyl Ion**

Colin S. Creaser, Brian L. Williamson



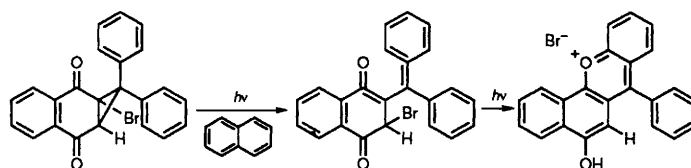
1679 **5-Methylene-2-cyclopentenones as New Formal [2 + 2 + 1] Cycloadducts from [2-(Dibenzylamino)ethenyl]-carbenechromium Complexes and Alkynes**

Michael Duetsch, Sara Vidoni, Frank Stein, Frank Funke, Matthias Noltemeyer, Armin de Meijere



1681 **Photoisomerization of Bromonaphthoquinone-fused Diphenylcyclopropane into Xanthylium Salt in the Presence of Arene Donors**

Hiroshi Moriwaki, Takumi Oshima, Toshikazu Nagai



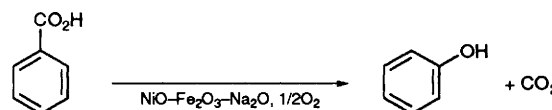
1683 **Solvent Effects on Hetero Diels–Alder Reactions of Sulfur Dioxide with 1,3-Dienes**

D. Suárez, X. Assfeld, Javier González, M. F. Ruiz-López, T. L. Sordo, J. A. Sordo

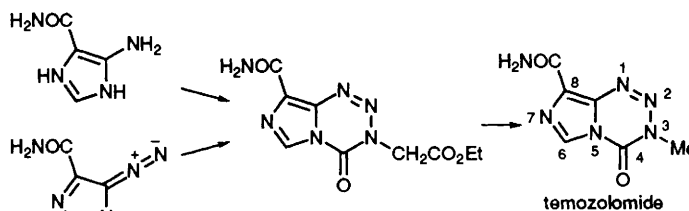
Solvent effects on thermal and Lewis acid catalysed hetero-Diels–Alder reactions of sulfur dioxide with buta-1,3-diene and isoprene have been studied theoretically by *ab initio* calculations. The electrostatic solvent effect plays an important role in the acid-catalysed reaction between isoprene and sulfur dioxide by reinforcing the methyl substitution effect and the catalytic action by BH_3 , rendering the 'meta' adduct as the major product in the reaction proceeding through the *endo* transition state, in agreement with experiment.

1685 **Alkali-promoted $NiO-Fe_2O_3$ Catalysts for Vapour-phase Oxidation of Benzoic Acid to Phenol**

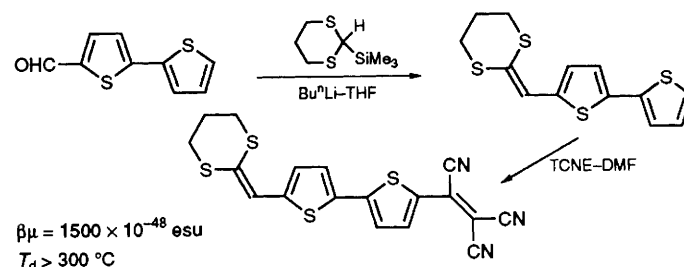
Jun Miki, Minoru Asanuma, Yakudo Tachibana, Tsutomu Shikada



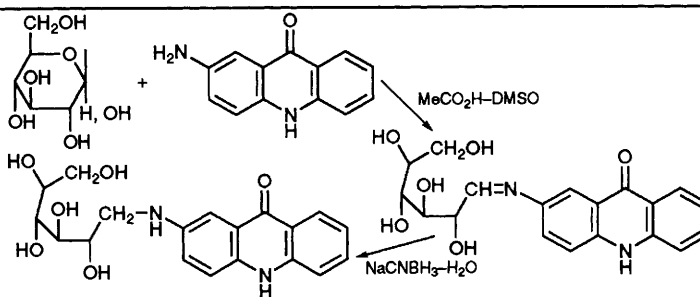
The catalyst $Ni-Fe_2O_3-Na_2O$ showed excellent performance in the vapour-phase oxidation of benzoic acid. The addition of Na_2O to the $NiO-Fe_2O_3$ catalyst enhanced the space time yield of phenol to more than $1000 \text{ g (l cat)}^{-1} \text{ h}^{-1}$, maintaining high phenol selectivity.

1687 **Alternative Syntheses of the Antitumour Drug Temozolomide avoiding the Use of Methyl Isocyanate**

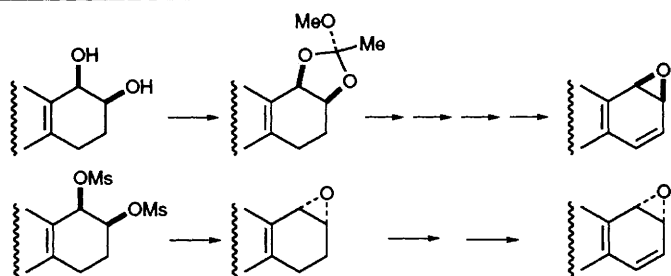
Yongfeng Wang, Malcolm F. G. Stevens, W. Thomson

1689 **Ketene Dithioacetal as a π -Electron Donor in Second-order Nonlinear Optical Chromophores**

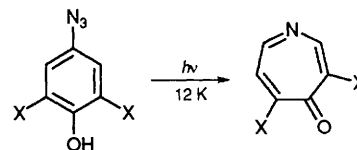
Varanosi Pushkara Rao, Y. M. Cai, Alex K-Y. Jen

1691 **A Sensitive and Selective Method for the Analysis of Complex Mixtures of Sugars and Linear Oligosaccharides**

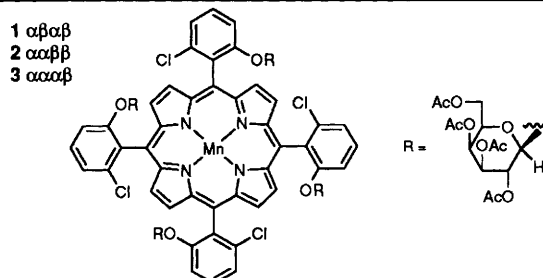
Mark Greenaway, George N. Okafo, Patrick Camilleri, Dashyant Dhanak

1693 **A New Synthetic Route to Non-K and Bay Region Arene Oxide Metabolites From *cis*-Diols**

Derek R. Boyd, Narain D. Sharma, Rajiv Agarwal, Nuala A. Kerley, R. Austin S. McMordie, Allison Smith, Howard Dalton, A. John Blacker, Gary N. Sheldrake

1695 **A Synthetic Approach to Azepin-4-ones exploiting Azide Photolysis in Low-temperature Matrices**

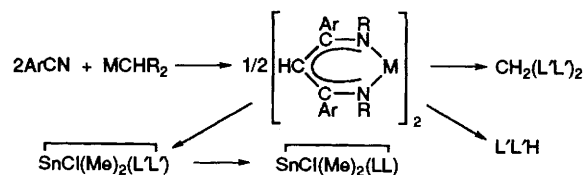
Ian R. Dunkin, Abdunaser El Ayeb, Michael A. Lynch

1697 **Enantiomeric Epoxidation of 4-Chlorostyrene with H_2O_2 catalysed by Robust Chloro Manganese(III) *meso*-5,10,15,20-Tetrakis[2-chloro-6-(2,3,4,6-tetraacetyl- β -glucosyl)-phenyl] Porphyrins**

Sandrine Vilain, Philippe Maillard, Michel Momenteau

- 1699 Transformation of the Bis(trimethylsilyl)methyl into a β -Diketiminato Ligand; the X-Ray Structure of $[\text{Li}(\text{L}'\text{L}')_2]_2$, $\text{SnCl}(\text{Me})_2(\text{L}'\text{L}')$ and $\text{SnCl}(\text{Me})_2(\text{LL})$, $[\text{L}'\text{L}' = \text{N}(\text{R})\text{C}(\text{Ph})\text{C}(\text{H})\text{C}(\text{Ph})\text{NR}$, $\text{LL} = \text{N}(\text{H})\text{C}(\text{Ph})\text{C}(\text{H})\text{C}(\text{Ph})\text{NH}$, $\text{R} = \text{SiMe}_3]$

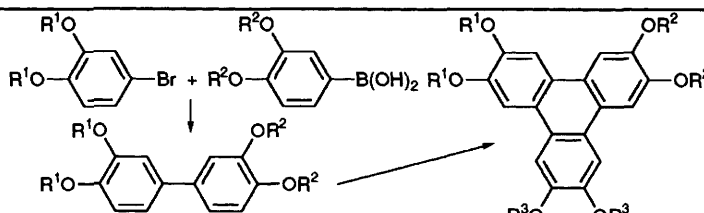
Peter B. Hitchcock, Michael F. Lappert, Dian-Sheng Liu



$[\text{Ar} = \text{Ph}$ or $\text{C}_6\text{H}_4\text{Me-4}$; $\text{R} = \text{SiMe}_3$; $\text{M} = \text{Li}$ or K ; $\text{L}'\text{L}' = (\text{N}(\text{R})\text{C}(\text{Ar}))_2\text{CH}$; $\text{LL} = (\text{N}(\text{H})\text{C}(\text{Ar}))_2\text{CH}]$

- 1701 A Novel, Efficient and General Synthetic Route to Unsymmetrical Triphenylene Mesogens using Palladium-catalysed Cross-coupling Reactions

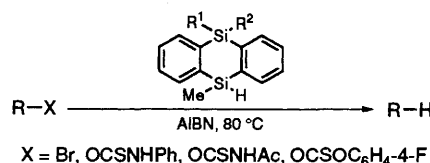
John W. Goodby, Michael Hird, Kenneth J. Toyne, Timothy Watson



Unsymmetrical hexa-substituted mesogenic triphenylenes have been prepared by using palladium-catalysed cross-couplings involving arylboronic acids and aryl bromides.

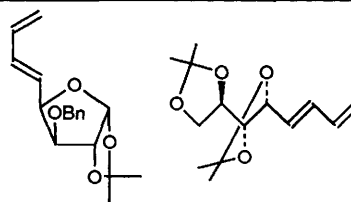
- 1703 9,10-Dihydro-9,10-disilaanthracenes as a New Radical-based Reducing Agent: Importance of Transannular Interaction Between Silyl Radical and Silicon Atom

Makoto Oba, Kozaburo Nishiyama



- 1705 Stereochemistry of Osmylation of Chiral Dienes: Diastereoselective Synthesis of Octitols

Kesavaram Narkunan, Madhavarao Nagarajan

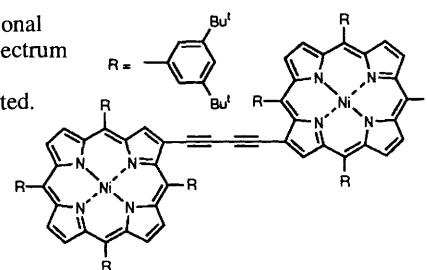


Bis-hydroxylation of the above dienes was carried out and its diastereoselectivity was determined.

- 1707 A Conformationally Constrained Conjugated Porphyrin Dimer

Jeffrey J. Gosper, Mukhtar Ali

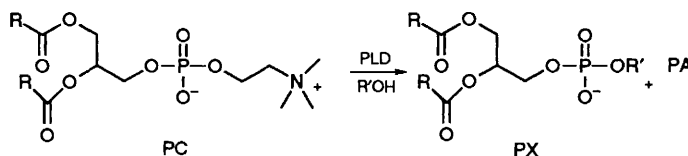
The synthesis, conformational analysis and electronic spectrum for this conjugated porphyrin dimer are reported.



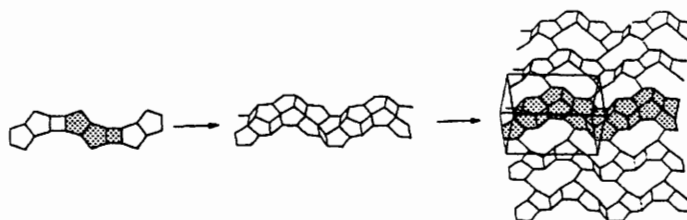
- 1709 Phospholipase D from *Streptomyces* Catalyses the Transfer of Secondary Alcohols

Paola D'Arrigo, Lorenzo de Ferra, Valentino Piergianni, Andrea Ricci, Domenico Scarcelli, Stefano Servi

Phospholipase D efficiently catalyses the transfer of various secondary alcohols R'OH.

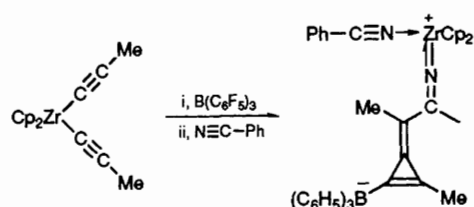


1711 Enumeration of Chiral Zeolite Frameworks



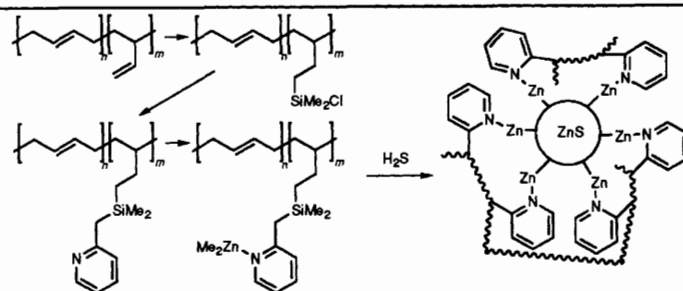
D. E. Akporiaye

1713 Boron-induced Formation of a Methylene-cyclopropene Unit in the Bis(cyclopentadienyl)zirconium Coordination Sphere



Bodo Temme, Gerhard Erker, Roland Fröhlich, Matthias Grehl

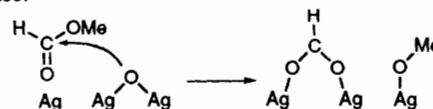
1715 A New, Simple and Versatile Method for the Production of Nano-scale Particles of Semiconductors



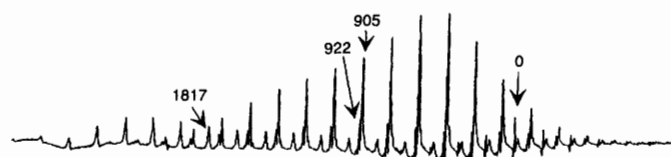
Xiaochang Li, John R. Fryer, David J. Cole-Hamilton

1717 Characterization of the Active Site for the Selective Oxidation of Methanol to Formaldehyde on Polycrystalline Silver Catalyst

The adsorption of methyl formate on an unmodified silver surface can be represented as below. In contrast, on a reconstructed silver surface which is active in the selective oxidation of methanol to formaldehyde, no reaction occurs owing to the electrophilicity of adsorbed oxygen and the steric inaccessibility of subsurface oxygen which is present on such a surface.



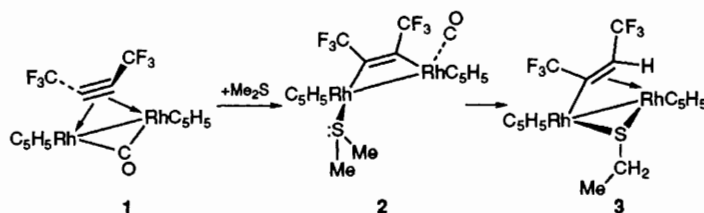
Graeme J. Millar, James B. Metson, Graham A. Bowmaker, Ralph P. Cooney

1719 A ^6Li and ^7Li MAS NMR Study of the Spinel-type Manganese Oxide LiMn_2O_4 and the Rock Salt-type Manganese Oxide Li_2MnO_3 

Keith R. Morgan, Susan Collier, Gary Burns, Kenta Ooi

^6Li and ^7Li MAS NMR has been used to characterise lithium sites in two paramagnetic lithium manganese oxides.

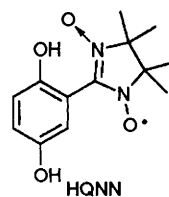
1721 The Unusual Stevens Type Rearrangements of Some Dialkyl Sulfides on a Rh–Rh Bond



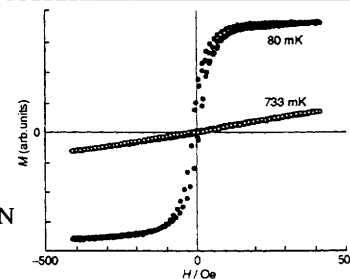
Michael P. Devery, Ron S. Dickson

1723 **An Organic Ferromagnet: α -Phase Crystal of 2-(2',5'-Dihydroxyphenyl)-4,4,5,5-tetramethyl-4,5-dihydro-1H-imidazolyl-1-oxy-3-oxide (α -HQNN)**

Tadashi Sugawara, Michio M. Matsushita, Akira Izuoka, Nobuo Wada, Naoya Takeda, Masayasu Ishikawa

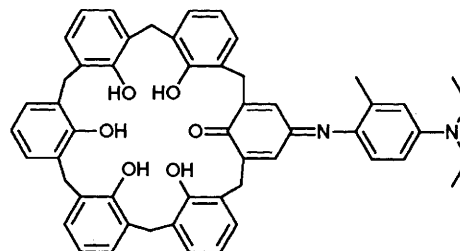


α -Phase crystals of HQNN showed a ferromagnetic phase transition at 0.5 K.



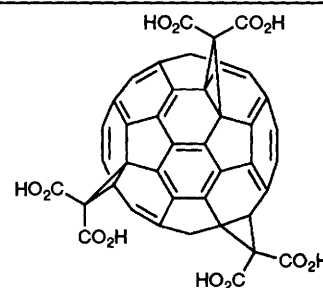
1725 **A Uranyl Ion-sensitive Chromoionophore based on Calix[6]arene**

Yuji Kubo, Shin'ya Maeda, Minoru Nakamura, Sumio Tokita



1727 **Water-soluble Malonic Acid Derivatives of C₆₀ with a Defined Three-dimensional Structure**

Iris Lamparth, Andreas Hirsch



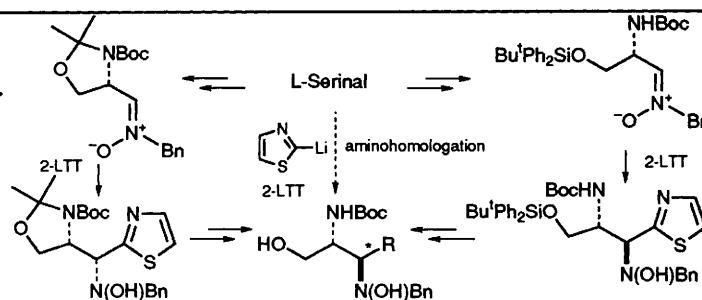
1729 **Synthesis and Characterization of Tantalum Pillared Montmorillonite**

Garbine Guiu, Paul Grange

The first reported method for the synthesis of tantalum-pillared montmorillonite (Ta-PILC) is described. Thermal stability measurements by XRD and BET surface area determination show a large pore structure. Results from catalytic tests on butan-1-ol dehydration are promising because of the activity of Ta-PILC and the 100% selectivity for formation of butenes.

1731 **Tunable Stereoselectivity in the Addition of 2-Lithiothiazole to L-Serinal Derived *N*-Benzyl Nitrones. Synthesis of C-2 Epimer 2,3-Diamino-4-Hydroxybutanals**

Alessandro Dondoni, Francisco L. Merchan, Pedro Merino, Tomás Tejero, Valerio Bertolasi



AUTHOR INDEX

- Abe, Manabu, 1673
 Agarwal, Rajiv, 1693
 Aggarwal, Varinder K., 1653
 Akporiaye, D. E., 1711
 Alanine, Alex I. D., 1649
 Ali, Mukhtar, 1707
 Anson, Christopher E., 1657
 Arap Koske, Samuel K., 1657
 Archibald, Stephen J., 1669
 Asanuma, Minoru, 1685
 Assfeld, X., 1683
 Barr, Donald, 1627
 Battersby, Alan R., 1649
 Ben Taârit, Y., 1667
 Bertolasi, Valerio, 1731
 Biasotto, Fabienne, 1661
 Bidell, Wolfgang, 1641
 Blacker, A. John, 1693
 Blake, Alexander J., 1669
 Blanche, Francis, 1649
 Bowmaker, Graham A., 1717
 Boyd, Derek R., 1693
 Burns, Gary, 1719
 Cai, Y. M., 1689
 Camilleri, Patrick, 1691
 Cannon, Roderick D., 1657
 Cendrowski-Guillaume, Sophie M., 1655
 Chambers, Eric J., 1631
 Chen, Qin, 1635
 Cheng, C. P., 1637
 Chern, Shuenn-Shing, 1645
 Cole-Hamilton, David J., 1715
 Collier, Susan, 1719
 Colquhoun, Howard M., 1643
 Cooney, Ralph P., 1717
 Creaser, Colin S., 1677
 Dalton, Howard, 1693
 D'Arrigo, Paola, 1709
 de Ferra, Lorenzo, 1709
 de Meijere, Armin, 1679
 Debussche, Laurent, 1649
 Devery, Michael P., 1721
 Dhanak, Dashyant, 1691
 Dickson, Ron S., 1721
 Dondoni, Alessandro, 1731
 Douthwaite, Richard E., 1641
 Duetsch, Michael, 1679
 Dunkin, Ian R., 1695
 Edwards, Andrew J., 1627
 El Ayeb, Abdunaser, 1695
 Ephritikhine, Michel, 1655
 Erker, Gerhard, 1713
 Etienne, Michel, 1661
 Felder, Patrick, 1659
 Franklin, Richard J., 1653
 Freyhardt, Clemens C., 1675
 Fröhlich, Roland, 1713
 Fryer, John R., 1715
 Fujikawa, Shinjiro, 1623
 Fujimoto, Kaoru, 1647
 Funke, Frank, 1679
 Gerson, Fabian, 1659
 González, Javier, 1683
 Goodby, John W., 1701
 Gosper, Jeffrey J., 1707
 Grange, Paul, 1729
 Green, Malcolm L. H., 1641
 Greenaway, Mark, 1691
 Grehl, Matthias, 1713
 Guiu, Garbine, 1729
 Halfen, Jason A., 1625
 Harger, Martin J. P., 1619
 Haworth, Ian S., 1631
 Hird, Michael, 1701
 Hirsch, Andreas, 1727
 Hitchcock, Peter B., 1699
 Horng, H. C., 1637
 Ishikawa, Masayasu, 1723
 Izuoka, Akira, 1723
 Jacobs, Pierre A., 1671
 Jayasooriya, Upali A., 1657
 Jen, Alex K-Y., 1689
 Jenneskens, Leonardus W., 1633
 Keegstra, Erik M. D., 1633
 Kerley, Nuala A., 1693
 Kubo, Yuji, 1725
 Kubota, Yasufumi, 1665
 Lamparth, Iris, 1727
 Lance, Monique, 1655
 Lappert, Michael F., 1699
 Lee, Gene-Hsiang, 1645
 Leeper, Finian J., 1649
 Li, Xiaochang, 1715
 Li, Yongfu, 1649
 Liao, F. L., 1637
 Linney, Lynda P., 1651
 Liu, Dian-Sheng, 1699
 Lynch, Michael A., 1695
 McMordie, R. Austin S., 1693
 Maeda, Shin'ya, 1725
 Mahapatra, Samiran, 1625
 Maillard, Philippe, 1697
 Martens, Johan A., 1671
 Mashima, Kazushi, 1623
 Mathieu, René, 1661
 Matsushita, Michio M., 1723
 Merchan, Francisco L., 1731
 Merino, Pedro, 1731
 Metson, James B., 1717
 Miki, Jun, 1685
 Millar, Graeme J., 1717
 Momenteau, Michel, 1697
 Morgan, Keith R., 1719
 Moriwaki, Hiroshi, 1681
 Nagai, Toshikazu, 1681
 Nagarajan, Madhavarao, 1705
 Nakamura, Akira, 1623
 Nakamura, Minoru, 1725
 Nakashima, Kazuaki, 1621
 Narkunan, Kesavaram, 1705
 Nemoto, Hisao, 1665
 Nierlich, Martine, 1655
 Nishiyama, Kozaburo, 1703
 Noltemeyer, Matthias, 1679
 Oba, Makoto, 1703
 Okafo, George N., 1691
 Oku, Akira, 1673
 O'Mahoney, Caroline A., 1643
 Omata, Kohji, 1647
 Ooi, Kenta, 1719
 Oshima, Takumi, 1681
 Peng, Shie-Ming, 1645
 Piergianni, Valentino, 1709
 Prütz, Walter A., 1639
 Raithby, Paul R., 1627
 Rao, Varanosi Pushkara, 1689
 Rennie, Moira-Ann, 1627
 Ricci, Andrea, 1709
 Ruiz-López, M. F., 1683
 Sandanayake, K. R. A. Samankumara, 1621
 Scarcelli, Domenico, 1709
 Schmidlin, Reto, 1659
 Schröder, Martin, 1669
 Self, Christopher R., 1651
 Servi, Stefano, 1709
 Sharma, Narain D., 1693
 Sheldrake, Gary N., 1693
 Shikada, Tsutomu, 1685
 Shinkai, Seiji, 1621
 Sieburth, Scott McN., 1663
 Smith, Allison, 1693
 Sordo, J. A., 1683
 Sordo, T. L., 1683
 Souverijns, Wim, 1671
 Spek, Anthony L., 1633
 Sreedharan-Menon, Ramesh, 1619
 Stamford, N. Patrick J., 1649
 Stein, Frank, 1679
 Stephens, Adam H. H., 1641
 Stevens, Malcolm F. G., 1687
 Suárez, D., 1683
 Sugawara, Tadashi, 1723
 Tachibana, Yakudo, 1685
 Takeda, Naoya, 1723
 Tanaka, Eiji, 1623
 Taylor, Roger, 1629
 Tejero, Tomás, 1731
 Temme, Bodo, 1713
 Thomas, Abraham, 1653
 Thomson, W., 1687
 Tokita, Sumio, 1725
 Tolman, William B., 1625
 Tomita, Kazuyuki, 1647
 Toyne, Kenneth J., 1701
 Tuel, A., 1667
 Turner, John F. C., 1641
 Urata, Hisao, 1623
 Vanbutsele, Gina, 1671
 Verhorevoort, Kerry, 1627
 Verrelst, Wim, 1671
 Vidoni, Sara, 1679
 Vigner, Julien, 1655
 Vilain, Sandrine, 1697
 Wada, Nobuo, 1723
 Wang, Yongfeng, 1687
 Watson, Timothy, 1701
 White, Ross P., 1657
 Wiebecke, Michael, 1675
 Williams, David J., 1643
 Williams, Ian H., 1651
 Williamson, Brian L., 1677
 Winpenny, Richard E. P., 1669
 Wong, Henry N. C., 1659
 Wright, Dominic S., 1627
 Wu, Ruowen, 1657
 Yamamoto, Yoshinori, 1665
 Yamazaki, Osamu, 1647
 Yang, C. S., 1637
 Zubieta, Jon, 1635
 Zwikker, Jan W., 1633

