

A 256-membered oligothiophene

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# ents





256 Oligothiophene Library

1015



### combinatorial library construction and screening Christoph A. Briehn and Peter Bäuerle\*

From solid-phase synthesis of  $\pi$ -conjugated oligomers to

A feature article describing concepts for the solid-phase synthesis of  $\pi$ -conjugated oligomers as material-related structures and the translation of the synthetic routes into combinatorial protocols.



Synthesis and crystal structure of a novel pentaconta-nuclear silver anionic cluster complex [HNEt<sub>3</sub>]<sub>4</sub>[Ag<sub>50</sub>S<sub>7</sub>(SC<sub>6</sub>H<sub>4</sub>Bu<sup>t</sup>-4)<sub>40</sub>]·2CS<sub>2</sub>·6C<sub>3</sub>H<sub>6</sub>O

Kaluo Tang, Xiangjin Xie, Yaohua Zhang, Xia Zhao and Xianglin Jin\*

A silver 4-tert-butylthiophenolate complex generated in situ reacted with CS<sub>2</sub> in acetone to give an anionic cluster  $[Ag_{50}S_7(SC_6H_4Bu^t-4)_{40}]^{4-}$ , the core of which consists of fifty silver atoms and forty-seven sulfur atoms, shaped like a discus with a diameter of *ca*. 2.0 nm.

### Preparation of B-free Ti-MWW through reversible structural conversion

Peng Wu and Takashi Tatsumi\*

Three-dimensional MWW silicate is converted reversibly into its lamellar precursor in the presence of piperidine solution, allowing a uniform Ti incorporation to result in a highly active B-free Ti-MWW catalyst.



Piperidine

Ti(OBu)



1026



집 0.2

0.1

1030

1034

1036

Carbon–epoxy electrodes: unambiguous identification of authentic triplephase (insulator/solution/electrode) processes

Nathan S. Lawrence, Mary Thompson, James Davis, Li Jiang, Timothy G. J. Jones and Richard G. Compton

The communication reports a new methodology which unequivocally *proves* the three-phase junction mechanism for the electro-oxidation of an organic crystal imbedded in a carbon electrode by the introduction of insonation to the electrode surface.

# Photosensitization of nanocrystalline TiO<sub>2</sub> by self-assembled layers of CdS quantum dots

Laurence M. Peter,\* D. Jason Riley, Elizabeth J. Tull and K. G. Upul Wijayantha

We report the preparation and characterization of CdS photosensitized  $TiO_2$  electrodes. Photocurrent spectroscopy indicate that the size of CdS Q-dots formed can be controlled *via* the concentration of stabilizer present during deposition.

# A novel [60]fullerene receptor with a Pd(II)-switched bisporphyrin cleft

Masatsugu Ayabe, Atsushi Ikeda, Seiji Shinkai,\* Shigeru Sakamoto and Kentaro Yamaguchi

Porphyrin dimer 1, which does not have an inside cavity and cannot interact with [60]fullerene ( $C_{60}$ ), becomes an excellent  $C_{60}$ -acceptor with a large cavity in the presence of a Pd(II) complex.



### The regioselective preparation of 1,3-diketones within a micro reactor

Charlotte Wiles, Paul Watts, Stephen J. Haswell\* and Esteban Pombo-Villar

We have developed a simple, room temperature route to the regioselective formation of uncontaminated 1,3-diketones or *O*-acylated products within a micro reactor.

### Approaches to a photocleavable protecting group for alcohols

Gary A. Epling and Anthony A. Provatas\*

A new protecting group for the alcohol functionality was devised and shown to be removed photochemically under ultraviolet light in the presence of a radical scavenger in high yields.



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Discovery of a new family of chromium ethylene polymerisation catalysts using high throughput screening methodology

David J. Jones, Vernon C. Gibson,\* Simon M. Green and Peter J. Maddox

High Throughput Screening (HTS) methodology has been employed to facilitate the discovery of an exceptionally active ethylene polymerisation catalyst family based on tridentate salicylaldimine ligands bearing bulky tryptycenyl substituents.



1040

1042

1046

Ruthenium catalyzed oxidation of tertiary nitrogen compounds with molecular oxygen: an easy access to *N*-oxides under mild conditions

Suman L. Jain and Bir Sain\*

Ruthenium catalyzed oxidation of tertiary nitrogen compounds with molecular oxygen: an easy access to *N*-oxides under mild conditions.

### Koilands from thiophiles: mercury(II) clusters from thiacalixarenes

Huriey Akdas, Ernest Graf, Mir Wais Hosseini,\* André De Cian, Alex Bilyk, Brian W. Skelton, George A. Koutsantonis, Ian Murray, Jack M. Harrowfield\* and Allan H. White

Tetra- and hexa-nuclear mercury(II) clusters encapsulated by  $S_4$  and  $S_8$  thiacalix[4]arenes provide new digonal and trigonal koilands.



# Investigation of the morphology–catalytic reactivity relationship for Pt nanoparticles supported on alumina by using the reduction of NO with $CH_4$ as a model reaction

Ioan Balint,\* Akane Miyazaki and Ken-ichi Aika

The relationship between the morphology of Pt nanoparticles supported on alumina and the catalytic reactivity for the NO/CH<sub>4</sub> reaction is examined. The conversion of the low index facets of the well-structured Pt nanoparticles to higher index planes is associated with the decrease in reaction selectivity to N<sub>2</sub>O and CO, and with the prevention of NH<sub>3</sub> formation.

New approaches to high-activity transition-metal catalysts for carbon–carbon bond forming reactions. Rhenium-containing phosphorus donor ligands for palladium-catalyzed Suzuki cross-couplings



Sandra Eichenseher, Klemenz Kromm, Olivier Delacroix and J. A. Gladysz\*

The rhenium complexes  $(\eta^5-C_5H_5)Re(NO)(PPh_3)((CH_2)_nPR_2:)$  (*n*/R = 0/Ph, 0/t-Bu, 0/Me, 1/Ph, 1/t-Bu), which contain electron-rich and sterically congested phosphido moieties, give active catalysts for the title reaction.

S S S S S S Bri and S S S S S S S Tet thi

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using H<sub>2</sub>O<sub>2</sub> as oxidising as well as reducing agent

Tridib Kumar Sarma, Devasish Chowdhury, Anumita Paul and Arun

This communication reports the generation of an Au-nanoparticle polyaniline composite with H2O2 used as both oxidising and reducing agent; the composite thus generated has enhanced electrical conductivity.

### Layered metal organosulfides: hydrothermal synthesis, structure and magnetic behaviour of the spin-canted magnet Co(1,2-(O<sub>2</sub>C)(S)C<sub>6</sub>H<sub>4</sub>)

Dale Cave, José-Miguel Gascon, Andrew D. Bond, Simon J. Teat and Paul T.

The new layered thiolate/carboxylate material  $Co(1,2-(O_2C)(S)C_6H_4)$  has been prepared by hydrothermal synthesis. The sulfur bridges couple the spins of the trigonal bipyramidal Co(II) ions very strongly and spin canting produces a

## 1,3-Dibromo-2,4,6-trinitrobenzene (DBTNB). Crystal engineering and perfect polar alignment of two-dimensional hyperpolarizable

Praveen K. Thallapally, Gautam R. Desiraju,\* Muriel Bagieu-Beucher, René Masse,\* Cyril Bourgogne and Jean-François Nicoud\*

1,3-Dibromo-2,4,6-trinitrobenzene, a new NLO organic crystal built with 2D hyperpolarisable chromophores, crystallises in a perfect polar order leading to an intense powder SHG signal.

# Engineering of ferrimagnetic Cu<sub>12</sub>-cluster arrays through supramolecular

Muralee Murugesu, Christopher E. Anson and Annie K. Powell\*

Zero-dimensional Cu<sub>12</sub> aggregates with non-zero ground state spins can be engineered via counter ions and solvent molecules into 1, 2 and 3-D arrays.

### An evanescent field driven mono-molecular layer photoswitch: coordination and release of metallated macrocycles

Michael J. Cook,\* Anne-Mette Nygård, Zhenxin Wang and David A. Russell\*

Photoswitching of 'on' and 'off' coordination sites in a self assembled monolayer containing the 4-(arylazo)pyridine chromophore has been achieved using waveguided light and illustrated through a coordination-release cycle involving zinc tetraphenylporphyrin.



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Chiral copper(II) bisoxazoline covalently anchored to silica and mesoporous MCM-41 as a heterogeneous catalyst for the enantioselective Friedel–Crafts hydroxyalkylation

Avelino Corma,\* Hermenegildo García,\* Ahmed Moussaif, María J. Sabater, Rachid Zniber and Achour Redouane

Chiral copper(II) diphenyl bisoxazoline covalently anchored on silica and MCM-41 exhibits a higher enantioselectivity (up to 92% ee) for the Friedel-Crafts hydroxyalkylation of 1,3-dimethoxybenzene with 3,3,3-trifluoropyruvate than does the same complex in solution.

Reversible near-infrared fluorescence switch by novel

Bingzhi Chen, Mingzhong Wang, Yingqi Wu and He Tian\*

A novel family of photochromic hybrids, 2,3-bis(2,5-dimethyl-3-

thienyl) unsymmetrical-phthalocyanines (BTE-uPcs), which show a photoregulating fluorescence switch in the near-infrared spectral

photochromic unsymmetrical-phthalocyanine hybrids based on



Fluorescent

Non-fluorescent



### Impact of Pd-mordenite pretreatment on the heterogeneity of Heck catalysis

M. Dams, L. Drijkoningen, D. De Vos and P. Jacobs\*

bisthienylethene

region.

 $Pd^{0}$  and  $Pd(NH_{3})_{4}^{2+}$  on mordenite are truly heterogeneous catalysts in Heck chemistry, while Pd<sup>2+</sup> in an all oxygen environment leaches and should be prevented.



### The versatile conversion of acyclic amides to α-alkylated amines

Young-Ger Suh,\* Dong-Yun Shin, Jae-Kyung Jung and Seok-Ho Kim

A general and efficient method for the versatile functionalization of acyclic amide via N,O-acetal TMS ether, an excellent precursor for the N-acyliminium ion, has been developed.

### Stereoselective cycloadditions of chiral acyl-nitroso compounds; selective reactions of ring-cleaved cycloadducts leading to a new approach to polyoxamic acid

Adrian G. Pepper, Garry Procter and Martyn Voyle



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The chemistry of stereoselective acyl-nitroso cycloadditions, and the regioselective differentiation of resulting diesters is explored, leading to a new approach to a polyoxamic acid from the polyoxin series of antibiotics.

v

# Catalytic direct 1,4-conjugate addition of aldehydes to vinylketones on secondary-amines immobilised in FSM-16 silica

Ken-ichi Shimizu,\* Hiromasa Suzuki, Eidai Hayashi, Tatsuya Kodama, Yoshimi Tsuchiya, Hisahiro Hagiwara and Yoshie Kitayama



1068

1070

1072

1074

H2WO4 (NH4)2WO4 1023k

Zr(OH)<sub>4</sub>

1076

ZrO<sub>2</sub> (2)

Zr(CH)<sub>4</sub>

NH<sub>3</sub>

CI//, | CI - Ru= Direct 1,4-conjugate addition of naked aldehydes to vinylketones is catalysed effectively by *N*-methyl-3-aminopropylated FSM-16 mesoporous silica, which can be regarded as a novel heterogeneous catalysis for a practical C–C bond formation reaction.

# Ring closing metathesis in protic media by means of a neutral and polar ruthenium benzylidene complex

Thomas Rölle\* and Robert H. Grubbs

The ring closing olefine metathesis in protic solvents using a new ruthenium benzylidene complex is described.

# Using a liquid emulsion membrane system for the encapsulation of organic and inorganic substrates within inorganic microcapsules

Julie A. Thomas,\* Linda Seton, Roger J. Davey and Christine E. DeWolf

Scanning electron microscope image of a polystyrene latex bead encapsulated inside a vaterite shell that has been produced *via* a biomimetic double emulsion templating system.

# 12-Tungstophosphoric acid/zirconia—a highly active stable solid acid—comparison with a tungstated zirconia catalyst

Biju M. Devassy, S. B. Halligudi,\* S. G. Hegde, A. B. Halgeri and F. Lefebvre

A highly active and stable zirconia supported 12-tungstophosphoric acid catalyst (1) is found to be 2–3 times more active than a tungstated zirconia catalyst (2) in benzylation and acylation reactions.

# Highly reactive heterogeneous Heck and hydrogenation catalysts constructed through 'bottom-up' nanoparticle self-assembly

Trent H. Galow, Ulf Drechsler, Jarrod A. Hanson and Vincent M. Rotello\*

The polymer-mediated electrostatic self-assembly of functionalized silicaand palladium nanoparticles provides porous aggregates. After calcination, these aggregates exhibit high catalytic activities in hydrogenation and Heck coupling reactions.

1023K H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub> 12-TPA

Zr(OH)<sub>4</sub> DMF/MeOH

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1080

1082

### COMMUNICATIONS

### $[Cu(tn)]_3[Cr(CN)_6]_2$ ·3H<sub>2</sub>O: a unique two-dimensional Cu–Cr cyanobridged ferromagnet (tn = 1,3-diaminopropane)

Franck Thétiot, Smaïl Triki,\* Jean Sala Pala, Carlos J. Gómez-García and Stéphane Golhen

Reaction of the cationic connecting block  $[Cu(tn)]^{2+}$  (tn = 1,3-diaminopropane) with the anionic building block  $[Cr(CN)_6]^{3-}$  leads to the first two-dimensional cyano-bridged ferromagnet ( $T_c = 9.5 \text{ K}$ ) involving Cu<sup>II</sup> ions; the structure displays unusual  $\mu_{3-}$  and  $\mu_4$ -bridging  $[Cu(tn)]^{2+}$  units.

Novel hetero-bimetallic metalla-macrocycles based on the bis-1-pyridyl ferrocene [Fe( $\eta^5$ -C<sub>5</sub>H<sub>4</sub>-1-C<sub>5</sub>H<sub>4</sub>N)<sub>2</sub>] ligand. Design, synthesis and structural characterization of the complexes [Fe( $\eta^5$ -C<sub>5</sub>H<sub>4</sub>-1-C<sub>5</sub>H<sub>4</sub>N)<sub>2</sub>](Ag<sup>1</sup>)<sub>2</sub><sup>2+</sup>/(Cu<sup>II</sup>)<sub>2</sub><sup>4+</sup>/(Zn<sup>II</sup>)<sub>2</sub><sup>4+</sup>

Dario Braga,\* Marco Polito, Marco Bracaccini, Daniela D'Addario, Emilio Tagliavini,\* Davide M. Proserpio and Fabrizia Grepioni\*

The bidentate sandwich ligand  $[Fe(\eta^5-C_5H_4-1-C_5H_4N)_2]$  has been prepared, structurally characterized and employed in the preparation of novel supramolecular heterobimetallic metalla-macrocycles.

# The direct synthesis of thioesters using an intermolecular radical reaction of aldehydes with dipentafluorophenyl disulfide in water

Hisanori Nambu, Kayoko Hata, Masato Matsugi and Yasuyuki Kita\*

$$R = alky| ary| = 0$$

$$R = alky| ary| = 0$$

$$VA-044 (1 equiv) = 0$$

$$CTAB (0.2 equiv) = 0$$

$$R = C_6F_5S - SC_6F_5 = 0$$

$$CTAB (0.2 equiv) = 0$$

$$R = C_6F_5S - SC_6F_5 = 0$$

The combination of the water-soluble radical initiator, 2,2'-azobis[2-(2imidazolin-2-yl)propane] dihydrochloride (VA–044), and surfactant, cetyltrimethylammonium bromide (CTAB), was found to be the most suitable condition for the effective direct synthesis of useful active thioesters (pentafluorophenyl thioesters) in water.

### On the electron delocalization in the radical cations formed by oxidation of MM quadruple bonds linked by oxalate and perfluoroterephthalate bridges

Malcolm H. Chisholm,\* Brian D. Pate, Paul J. Wilson and Jeffrey M. Zaleski\*

The oxalate-bridged compounds are electron delocalized over 4 metal centres, but the perfluoroterephthalate-bridged species are electon delocalized for W, but valence trapped for Mo.

### *In vitro* inhibition of gene transcription by novel photoactivated polyazaaromatic ruthenium(II) complexes

Marc Pauly, Isabelle Kayser, Martine Schmitz, Mario Dicato, André Del Guerzo, Isabelle Kolber, Cécile Moucheron and Andrée Kirsch-De Mesmaeker\*

Ru(II) complexes that exhibit a type I photo-oxidation of DNA, are able to inhibit the *in vitro* transcription of a plasmid DNA template.







1088

1090

140

# First *in vitro* norlignan formation with *Asparagus officinalis* enzyme preparation

Shiro Suzuki, Tomoyuki Nakatsubo, Toshiaki Umezawa\* and Mikio Shimada

The first report of the enzymatic formation of (*Z*)-hinokiresinol from two non-identical phenylpropanoid monomers, 4-coumaryl alcohol and 4-coumaroyl CoA, and from a dimer, 4-coumaryl 4-coumarate.

# Competing Diels–Alder reactions of activated nitroethylene derivatives and [3,3]-sigmatropic rearrangements of the cycloadducts

 $( ) ^{0} N^{*}^{0} \rightarrow ( ) ^{COPh} NO_{2}$ 

Peter A. Wade,\* James K. Murray Jr., Sharmila Shah-Patel and Hung T. Le

Diels–Alder reaction of  $CH_2=CXNO_2$  with cyclohexa-1,3-diene gave bicyclo[2.2.2]octenes, nitronic esters, and an enol ether (X = COPh). The nitronic esters and enol ether rearranged to single diastereomers of the bicyclo[2.2.2]octenes.

### Muonium addition to DMPO and PBN sorbed in silica-gel

Christopher J. Rhodes,\* Ivan D. Reid and Ulrich Zimmermann

Radiolabelled nitroxides were formed by muonium addition to the spin-traps PBN and DMPO, sorbed in silica-gel: these were investigated using a combination of transverse-field, avoided-level-crossing, and longitudinal-field muon spin relaxation methods.



100 120 FREQUENCY (MHz)

40



# Fabrication of two-dimensional layered perovskite $[NH_3(CH_2)_{12}NH_3]PbX_4$ thin films using a self-assembly method

Takashi Matsui, Akane Yamaguchi, Yuko Takeoka, Masahiro Rikukawa\* and Kohei Sanui

High-quality thin films of two-dimensional layered perovskites have been fabricated by a self-assembly method. The organic and inorganic perovskite films are uniform and smooth, and the thickness can be controlled at the nano-meter level.

# Novel ferrocene-containing helical triangular macrocycle achieved *via* an exchange reaction

Guo Dong, Han Gang, Duan Chun-ying,\* Pang Ke-liang and Meng Qing-jin\*

A novel ferrocene-containing helical triangular macrocyclic compound has been constructed through an exchange reaction of hydrazone groups in which Co(II) ions acts as both template and catalyst.



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electron  $Fe^{I}$  complex that reduces  $C_{60}$  to its mono-anion Jaime Ruiz, Charlotte Pradet, François Varret and Didier Astruc\*

The metallodendrimer [dendr-64-NHCOCpFe<sup>II</sup>( $\eta^{6}$ -C<sub>6</sub>Me<sub>6</sub>)]<sup>64+/0</sup>, that contains 64 redox centers, has been synthesized and characterized, and the 19-electron form reduces C<sub>60</sub> to [dendr-64-NHCOCpFe<sup>II</sup>( $\eta^{6}$ -C<sub>6</sub>Me<sub>6</sub>)]<sup>64+</sup>(C<sub>60</sub><sup>--</sup>)<sub>64</sub>.

Molecular batteries: synthesis and characterization of a dendritic 19-

# ESI-FTICR investigation of triethylammonium ion-driven resorcin[4]arene dimer formation and structure

Elina Ventola,\* Kari Rissanen and Pirjo Vainiotalo

Novel gas-phase dimer structure of tosylium substituted resorcin[4]arene with a triethylammonium ion as a charge carrying guest has been obtained from an ESI-FTICR measurement and *ab initio* geometry optimization calculations.

### Carbon nanohorns grown from ruthenium nanoparticles

Junfeng Geng, Caterina Ducati, Douglas S. Shephard, Manish Chhowalla, Brian F. G. Johnson\* and John Robertson

A nanoscale ruthenium/gold bimetallic cluster of clusters has been used as a molecular precursor to produce pure ruthenium nanoparticles (seeds) as catalysts for the growth of carbon nanohorns.

# New stereoselective reaction of methylglyoxal with 2-aminopyridine and adenine derivatives: Formation of imino acid-nucleic base derivatives in water under mild conditions

Christel Routaboul, Lionel Dumas, Isabelle Gautier-Luneau, Jacques Vergne, Marie-Christine Maurel and Jean-Luc Décout\*

A remarkable stereoselective reaction of methylglyoxal with 2-aminopyridine, the nucleic base adenine and adenine nucleosides leads in good yield to heterocycles of a new family in water under mild conditions and should be of interest in the understanding of the biological effects of methylglyoxal.

# Synthesis of (+)-polyoxamic acid and D-sorbitol from simple achiral allylic halides employing (*S*,*S*)-hydrobenzoin as a chiral source

Kwan Soo Kim,\* Yong Joo Lee, Jin Hwan Kim and Da Kyung Sung

Coupling of *cis*-1-bromo-2-pentene and (S,S)-hydrobenzoin stannylene acetal followed by regio- and stereoselective transformations of the resulting allylic ether gave (+)-polyoxamic acid and a similar procedure was applied to the synthesis of D-sorbitol from *trans*-1-iodo-2-hexene.



1114





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### 2-(1,3-Dithiolan-2-ylidene)-5-(1,3-dithian-2-ylidene)-1,3,4,6tetrathiapentalene(DHDA-TTP), a hybrid of BDH-TTP and BDA-TTP, and its metallic cation-radical salts

Jun-ichi Yamada,\* Maki Watanabe, Takashi Toita, Hiroki Akutsu, Shin'ichi Nakatsuji, Hiroyuki Nishikawa, Isao Ikemoto and Koichi Kikuchi\*

The synthesis of the unsymmetrical donor molecule DHDA-TTP and its ability to produce metallic cation-radical salts is described.

# Controlling the variation of axial water exchange rates in macrocyclic lanthanide(III) complexes

Silvio Aime,\* Alessandro Barge, Andrei S. Batsanov, Mauro Botta, Daniela Delli Castelli, Franco Fedeli, Armando Mortillaro, David Parker\* and Horst Puschmann

Axial water exchange rates in well-defined series of cationic lanthanide complexes have revealed the importance of the second sphere of hydration whose structure is controlled by the choice of anion and the nature of the substituents on the ligand.

### Laterally-extended porphyrin systems incorporating a switchable unit

Maxwell J. Crossley\* and Lesley A. Johnston

*p*-Quinone units incorporated into the central portion of rigid  $\pi$ -systems can function as a chemically and electrochemically controllable switch, thereby acting as a means of modulating electronic communication between the two end groups.

# Synthesis of novel BINOL-derived chiral bisphosphorus ligands and their application in catalytic asymmetric hydrogenation

Yong-Gui Zhou and Xumu Zhang\*

Some novel *ortho*-substituted BINOL-derived bisphosphorus ligands (*o*-BINAPO and *o*-NAPHOS) were synthesis from (*S*)-BINOL; these ligands showed excellent ee in Rh(I)-catalyzed asymmetric hydrogenation.

# 25.3 deg

# Fabrication of vertically and unidirectionally oriented polypeptide assemblies on self-assembled monolayers by stepwise polymerization

Masahiro Higuchi,\* Tomoyuki Koga, Kazuhiro Taguchi and Takatoshi Kinoshita

A polypeptide assembly prepared by stepwise polymerization on a self-assembled monolayer consisting of amino-alkanethiol and dialkyl disulfide oriented vertically and unidirectionally to the surface.

$$1122$$

$$Laterally-extended
$$1122$$

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$$Maxwell J. Cr
$$p-Quinone unint
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as a means of t$$$$$$$$

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1120

COOMe R NHAc R NHAC

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### ADDITIONS AND CORRECTIONS



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S. Aldridge, C. Bresner, I. A. Fallis, S. J. Coles and M. B. Hursthouse

Multidentate Lewis acids: synthesis, structure and mode of action of a redox-based fluoride ion sensor

J. Matthew Wood, Paul S. Hinchliffe, Andy M. Davis, Rupert P. Austin and Michael I. Page

Hydrolysis of a sulfonamide by a novel elimination mechanism generated by carbanion formation in the leaving group

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\* Indicates the author for correspondence: see article for contact details. Supplementary crystallographic data are available: see article for further information.

Electronic supplementary information is available on http://www.rsc.org/esi: see article for further information.

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