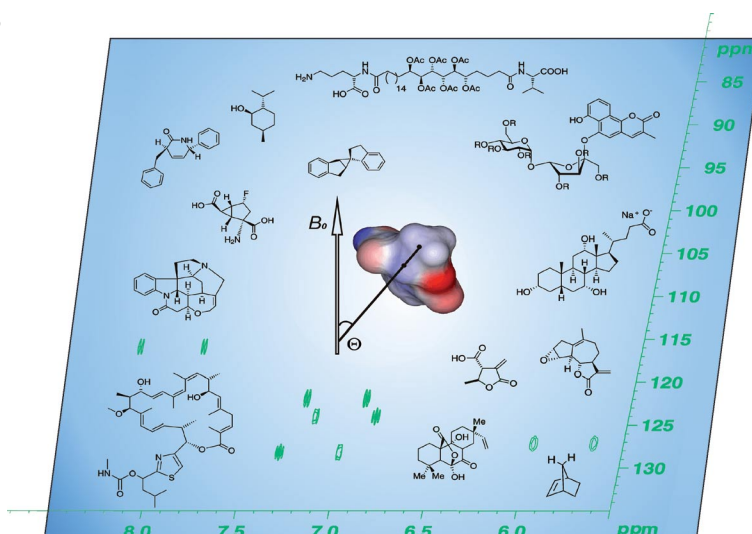


A union formed by chemical societies in Europe (ChemPubSoc Europe) has taken the significant step into the future by merging their traditional journals, to form two leading chemistry journals, the *European Journal of Inorganic Chemistry* and the *European Journal of Organic Chemistry*. Three further members of ChemPubSoc Europe (Austria, Czech Republic and Sweden) are Associates of the two journals.

## COVER PICTURE

The cover picture shows all organic compounds to which residual dipolar couplings (RDCs) were applied in the NMR spectroscopic determination of their relative configuration or in the assignment of diastereotopic moieties. The details on how RDCs can be applied for structure determination, as well as the necessary prerequisites and the current developments in the area of flexible compounds, are presented in the Microreview by C. Thiele on p. 5673ff.



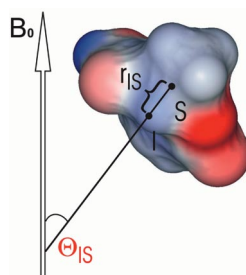
## MICROREVIEW

### NMR Spectroscopy

C. M. Thiele\* ..... 5673–5685

Residual Dipolar Couplings (RDCs) in Organic Structure Determination

**Keywords:** NMR spectroscopy / Configuration determination / Conformation analysis / Residual dipolar couplings



Residual dipolar couplings (RDCs), which become observable after the compound in question is introduced into an anisotropic environment, are increasingly important as additional NMR restraints in the determination of the structure of organic compounds. An overview of the alignment media for organic compounds and the corresponding measurement methods and applications is given.

## SHORT COMMUNICATIONS

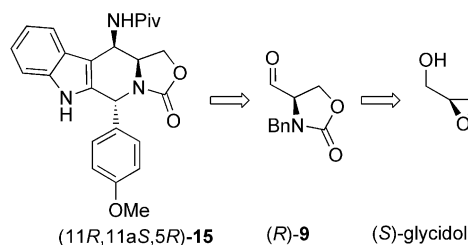
### Stereoselective Synthesis

J. Routier, M. Calancea, M. David,  
Y. Vallée, J.-N. Denis\* ..... 5687–5691



Heterolignans: Stereoselective Synthesis of an 11-Amino Analog of Azaelliptitoxin

**Keywords:** Stereoselective synthesis /  $\alpha$ -Amino aldehydes / Bioactive heterolignans / Azaelliptitoxin / Pictet–Spengler reaction



The stereoselective synthesis of the *N*-protected 11-amino-azaelliptitoxin analog (11*R*,11*aS*,5*R*)-**15** from (*S*)-glycidol (**7**) is described. The key intermediate of this original synthetic approach is the first

prepared enantiopure  $\alpha$ -amino aldehyde **9** ( $\geq 99\%$  *ee*), inducing the expected configurations of the two other stereogenic centers C-5 and C-11 of the final target **15**.

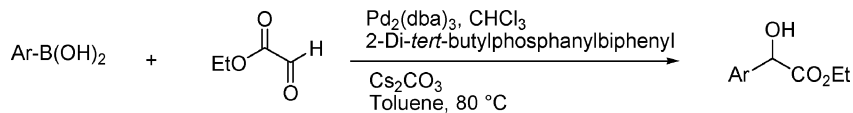
### Mandellate Derivatives

I. N. Francesco, A. Wagner,\*  
F. Colobert\* ..... 5692–5695



Suzuki–Miyaura Coupling Reaction of Boronic Acids and Ethyl Glyoxylate: Synthetic Access to Mandelate Derivatives

**Keywords:** C–C coupling / Boronic acids / Boron / Glyoxylates / Palladium

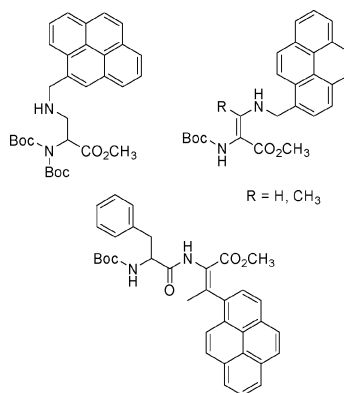


The palladium-catalyzed coupling of arylboronic acids with ethyl glyoxylate provides a straightforward method for the synthesis of mandelic esters.  $\text{Pd}_2(\text{dba})_3 \cdot \text{CHCl}_3$  in combination with 2-di-*tert*-butylphosphan-

ylbiaryl as the catalytic system and  $\text{Cs}_2\text{CO}_3$  as the base were used. The reaction tolerates a wide range of functionalized boronic acids.

## FULL PAPERS

$\beta$ -Pyrenylamino acid derivatives have been synthesized from dehydroamino acids by using several types of reactions, namely, Michael addition, substitution and Suzuki cross-coupling reactions. The latter type of reaction was also applied to the synthesis of  $\beta$ -(pyren-1-yl)dehydriptides. The electrochemical behaviour and photophysical properties of some of the compounds were studied.

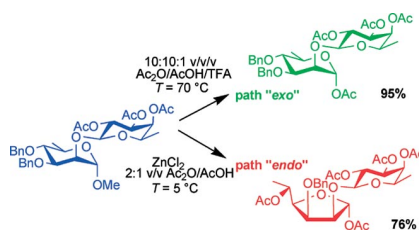


**A. S. Abreu, E. M. S. Castanheira,  
P. M. T. Ferreira,\* L. S. Monteiro,  
G. Pereira,  
M.-J. R. P. Queiroz ..... 5697–5703**

Pyrenylamino Acids: Synthesis, Photophysical and Electrochemical Studies

**Keywords:** Dehydroamino acids / Pyrene / Cross-coupling / Cyclic voltammetry / Fluorescence

The mild and selective acetolysis of 6-deoxy-sugar (rhamnose, fucose, and quinovose) derivatives under thermodynamic and kinetic conditions has been investigated. The products obtained are highly dependent on the competition between *exo* and *endo* anomeric oxygen by the  $\text{Ac}^+$  ion.

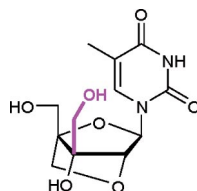


**L. Cirillo, E. Bedini,\*  
M. Parrilli ..... 5704–5714**

Acetolysis of 6-Deoxysugar Disaccharide Building Blocks: *exo* versus *endo* Activation

**Keywords:** Acetolysis / Deoxysugars / Oligosaccharides / Hexofuranoses / Zinc

The 3'-C-hydroxymethyl-branched Locked Nucleic Acid (LNA) monomer was synthesized in 19 synthetic steps from diacetone- $\alpha$ -D-glucose. The nucleoside is locked in an *N*-type conformation with the hydroxymethyl substituent in a pseudoaxial position.

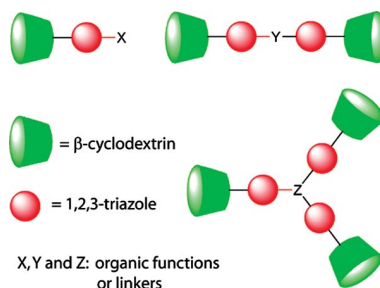


**S. Kumar, P. K. Sharma, P. C. Stein,  
P. Nielsen\* ..... 5715–5722**

Synthesis of the 3'-C-Hydroxymethyl-Branched Locked Nucleic Acid Thymidine Monomer

**Keywords:** Locked nucleic acid / Nucleosides / Oligonucleotides / Ruthenium oxidation

A variety of mono- and polytopic 1,2,3-triazole  $\beta$ -CD derivatives have been synthesized by click chemistry. The synthetic procedure is based on the  $\text{Cu}^I$ -catalyzed azide-alkyne cycloaddition reaction between hydroxylated or randomly methylated  $\beta$ -CD monoazides and alkynyl precursors. Easy to use, the reaction is also high-yielding for many molecules.



**M. Mourer, F. Hapiot, S. Tilloy,  
E. Monflier, S. Menuel\* ..... 5723–5730**

Easily Accessible Mono- and Polytopic  $\beta$ -Cyclodextrin Hosts by Click Chemistry

**Keywords:** Click chemistry / Cycloaddition / Cyclodextrins

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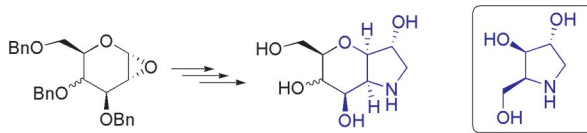
## Iminosugar Hybrid Molecules

V. R. Doddi, H. P. Kokatla,  
A. P. J. Pal, R. K. Basak,  
Y. D. Vankar\* ..... 5731–5739



Synthesis of Hybrids of D-Glucose and D-Galactose with Pyrrolidine-Based Iminosugars as Glycosidase Inhibitors

**Keywords:** Glycosidase inhibitors / Iminosugars / Azasugars / Oxidative cyclization



3,4,6-Tri-*O*-benzyl glycal epoxides have been efficiently converted into four sugar–iminosugar hybrid molecules made up of D-glucose and D-galactose with pyrrolidine-

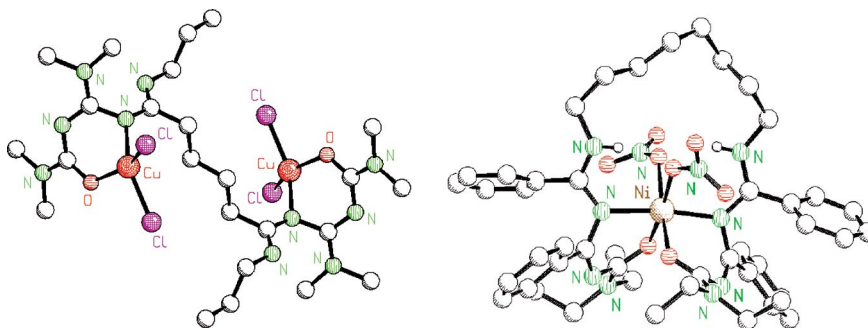
based iminosugars. These hybrid molecules were found to be moderate glycosidase inhibitors.

## 1-Oxa-3,5,7-triazaheptatrienes

J.-B. Greving, H. Behrens, R. Fröhlich,  
E.-U. Würthwein\* ..... 5740–5754

Multivalent 1-Oxa-3,5,7-triazahepta-1,3,5-trienes: Synthesis, Structural Properties and Metal Coordination

**Keywords:** Ligand design / Oligonitriles / N ligands / Pincer complexes / Ring-opening reaction



Multivalent amino-substituted bis- and tris-(armed) oligonitrile derivatives are synthesized by two alternative reaction pathways. In the solid state some derivatives show in-

ter- and intramolecular hydrogen bonding. They act as chelating ligands with various metal ions to form novel types of coordination compounds.

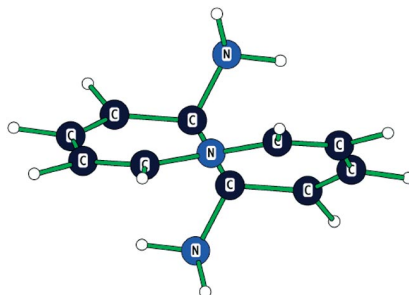
## Möbius Aromaticity

M. Mauksch,\*  
S. B. Tsogoeva\* ..... 5755–5763



Neutral Möbius Aromatics: Derivatives of the Pyrrole Congener Aza[11]annulene as Promising Synthetic Targets

**Keywords:** Aromaticity / Annulenes / Density functional calculations / Triplet state / Nitrogen heterocycles / Boron



Computational evidence at the B3LYP/6-31G\* level of theory suggests that the 6,7-diamino-substituted helical “Möbius” 1H-aza[11]annulene is aromatic and a neutral ground state on its potential surface.

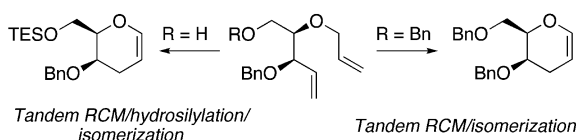
## 3-Deoxy Glycals

B. Schmidt,\* A. Biernat ..... 5764–5769



Synthesis of 3-Deoxy Glycals via Tandem Metathesis Sequences and Their Use in an Intermolecular Heck Arylation

**Keywords:** Olefin metathesis / Isomerization / Carbohydrates / Glycals

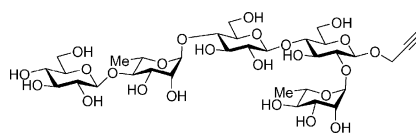


3-Deoxygalactal was synthesized by using the tandem RCM/isomerization method. The tandem sequence can also be extended by a dehydrogenative silylation step, re-


sulting in the formation of a 3-deoxy glycal bearing two orthogonally protected alcohol groups.

## Synthetic Carbohydrate

The total synthesis of the carbohydrate portion of steroidal saponins, isolated from *Calamus insignis*, is reported. A simple route was followed, starting from commercially available D-glucose and L-rhamnose, through high-yielding protecting group manipulation strategies.



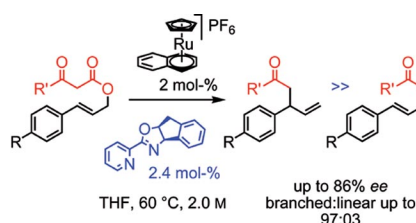
**S. Dasgupta,**  
**B. Mukhopadhyay\*** ..... 5770–5777

Synthesis of Oligosaccharides Related to a Biodynamic Saponin from *Calamus Insignis* as Their Propargyl Glycosides 


**Keywords:** Carbohydrates / Oligosaccharides / Glycosides / Saponins

## Homogeneous Catalysis

A combination of [CpRu( $\eta^6$ -naphthalene)](PF<sub>6</sub>) and enantiopure pyridine-mono-oxazoline ligands catalyze the regio- and enantioselective decarboxylative Carroll rearrangement of allyl  $\beta$ -keto esters into  $\gamma,\delta$ -unsaturated ketones.



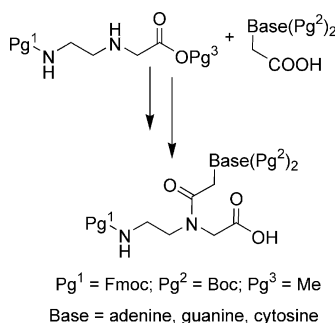
**D. Linder, F. Buron, S. Constant,**  
**J. Lacour\*** ..... 5778–5785

Enantioselective CpRu-Catalyzed Carroll Rearrangement – Ligand and Metal Source Importance 


**Keywords:** Allylic compounds / Air-stable precatalyst / C–C coupling / N ligands / Ruthenium / Enantioselective catalysis

## PNA Synthesis

A straightforward synthesis of orthogonally protected PNA monomers is described. The new monomers are demonstrated to be valid building blocks for PNA oligomerization.



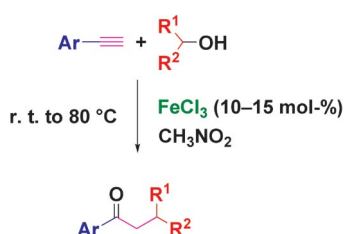
**A. Porcheddu,\* G. Giacomelli, I. Piredda,**  
**M. Carta, G. Nieddu** ..... 5786–5797

A Practical and Efficient Approach to PNA Monomers Compatible with Fmoc-Mediated Solid-Phase Synthesis Protocols 

**Keywords:** Peptide nucleic acids / Guanine / Protecting groups / Solid-phase synthesis

## Addition of Alcohols to Alkynes

A novel, efficient and atom-economical reaction for the preparation of diversely substituted aryl ketones by the simple treatment of terminal aryl alkynes and benzylic alcohols with catalytic FeCl<sub>3</sub> has been developed. The reaction is highly solvent dependent; nitromethane was the best solvent for this transformation. The mechanism of this reaction is proposed and discussed.



**U. Jana,\* S. Biswas,**  
**S. Maiti** ..... 5798–5804

Iron(III)-Catalyzed Addition of Benzylic Alcohols to Aryl Alkynes – A New Synthesis of Substituted Aryl Ketones

**Keywords:** Alkynes / Alcohols / Ferric chloride / Addition reaction / Atom economy

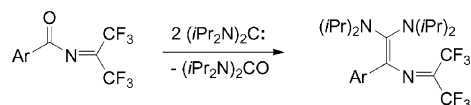
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## Carbenes

D. Poliakov, A. Rogalyov,  
I. Shevchenko\* ..... 5805–5809

Interaction of Bis(diisopropylamino)carbene with Aroylimines Activated by Trifluoromethyl Groups

**Keywords:** Aroylimine / Carbenes / Carbonyl group / Deoxygenation / Cyclization



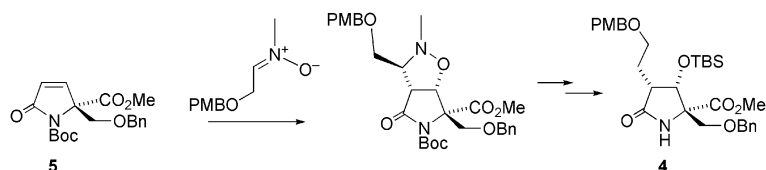
Bis(diisopropylamino)carbene deoxygenates the carbonyl group of aroylimines  $\text{Ar}-\text{C}(\text{O})-\text{N}=\text{C}(\text{CF}_3)_2$  to form alkenes.

## 1,3-Dipolar Cycloaddition of Nitrone

N. Langlois,\* J.-C. Legeay,  
P. Retailleau ..... 5810–5814

C-(4-Methoxybenzyloxymethyl)-N-methylnitrone Cycloaddition to Highly Functionalized Pyrrolinone: A Regio- and Stereoselective Approach to New Omuralide–Salinosporamide A Hybrids

**Keywords:** Cycloaddition / Nitrone /  $\gamma$ -Lactams / Proteasome inhibitors / Salinosporamide



The advanced intermediate **4** in the synthesis of new omuralide–salinosporamide A hybrids was prepared in a few steps from pyrrolinone **5**, which was

derived from methyl pyroglutamate, with a regio- and stereoselective C-(4-methoxybenzyloxymethyl)-N-methylnitrone cycloaddition.

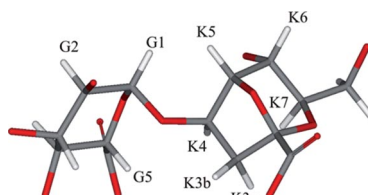
## Structures of Disaccharides

F. J. Fernández de Córdoba,  
M. A. Rodríguez-Carvajal,\*  
P. Tejero-Mateo,  
A. M. Gil-Serrano\* ..... 5815–5822



Structure and Conformational Studies of the Disaccharides Derived from the Inner Core of the Lipopolysaccharide Isolated from *Sinorhizobium fredii* SMH12

**Keywords:** Oligosaccharides / Structure elucidation / Conformation analysis / Rhizobium / Lipopolysaccharide



The structure and conformational behaviour of the core region of the lipopolysaccharide isolated from *S. fredii* SMH12 have been studied. The results indicate that the core oligosaccharide contains a disaccharide made up of  $\alpha$ -D-glucopyranuronic acid (1 $\rightarrow$ 4)-linked to D-Kdo. In solution, this disaccharide exists as an equilibrium of three different structures.

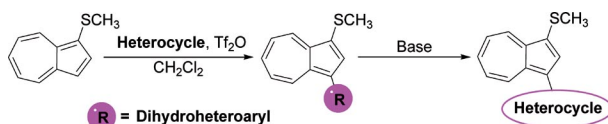
## Azulene Chemistry

J. Higashi, T. Shoji,\* S. Ito, K. Toyota,  
M. Yasunami, N. Morita\* ..... 5823–5831



Heteroarylation of 1-Azulenyl Methyl Sulfide: Two-Step Synthetic Strategy for 1-Methylthio-3-(heteroaryl)azulenes Using the Triflate of N-Containing Heterocycles

**Keywords:** Azulene / Heteroarylation / Electrophilic Substitution / Electrochemistry

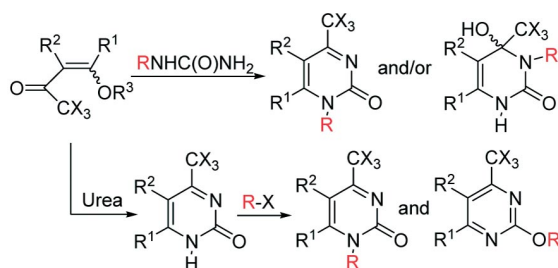


1-Azulenyl methyl sulfide reacts with highly electrophilic trifluoromethanesulfonates of N-heterocycles to give 1-methylthio-3-(dihydroheteroaryl)azulenes in good yields. Treatment of these azulenes

with KOH or *t*BuOK afforded 1-methylthio-3-(heteroaryl)azulenes in good yields. The redox behavior of the 1-methylthio-3-(heteroaryl)azulenes was examined by cyclic voltammetry.



## *N*-Alkylation of Pyrimidin-2-ones



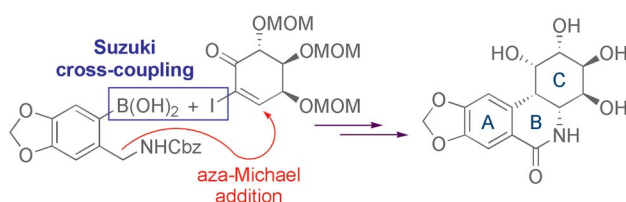
The chemoselective control of the synthesis of *N*-alkyl-4-(trihalomethyl)-1*H*-pyrimidin-2-ones is reported.

**N. Zanatta,\* D. Faoro, L. da S. Fernandes,  
P. B. Brondani, D. C. Flores,  
A. F. C. Flores, H. G. Bonacorso,  
M. A. P. Martins ..... 5832–5838**

Comparative Study of the Chemoselectivity and Yields of the Synthesis of *N*-Alkyl-4-(trihalomethyl)-1*H*-pyrimidin-2-ones

**Keywords:** Pyrimidines / Heterocycles / Alkylation / Enones / Chemoselectivity

## Phenanthridone Synthesis



A convergent synthesis of a highly oxygenated phenanthridone, featuring a Suzuki cross-coupling and intramolecular aza-

Michael addition reaction sequence to generate the fused B–C ring juncture, is accomplished.

**G. Pandey,\* M. Balakrishnan,  
P. S. Swaroop ..... 5839–5847**

A Suzuki Cross-Coupling and Intramolecular Aza-Michael Addition Reaction Sequence Towards the Synthesis of 1,10*b*-*epi*-7-Deoxypancratistatins and Their Cytotoxicity Studies

**Keywords:** Antitumor agents / Chiral pool / Cross-coupling / Intramolecular aza-Michael addition / Pancratistatin

\* Author to whom correspondence should be addressed.

Supporting information on the WWW (see article for access details).