









BELGIUM











VENSA



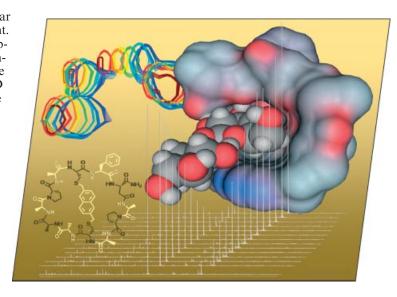




The EUChemSoc Societies have taken the significant step into the future by merging their traditional journals, to form two leading chemistry journals, the European Journal of Inorganic Chemistry and the European Journal of Organic Chemistry. Three further **EUChemSoc Societies (Austria,** Czech Republic and Sweden) are Associates of the two journals.

COVER PICTURE

The cover picture shows the artificial molecular recognition of cellobiose in an aqueous environment. The complex depicted forms from an organo-peptidic receptor composed of a bicyclic peptide containing a naphthalene bridge and the disaccharide cellobiose. The complex was analyzed by 2D NMR spectroscopy, and the changes in the chemical shift observed upon receptor titration (shown for 1-H of the naphthalene ring) with cellobiose is in agreement with the structure of the complex. The largest shifts were observed for those receptor protons in immediate contact with the saccharide. The formation of the complex measured by ESI-MS at increasing collision energies is shown in the stacked plot inset and can be performed as a high throughput screening (HTS) of carbohydrate-receptor interaction. Details are discussed in the article by M. Meldal et al. on p. 5003ff.



MICROREVIEW

CO₂ Incorporation

M. Mori* 4981-4993

Regio- and Stereoselective Synthesis of Triand Tetrasubstituted Alkenes by Introduction of CO_2 and Alkylzinc Reagents into Alkynes

Keywords: Carbon dioxide / Oxanickelacyclopentene / Transmetalation / Alkynes / Alkenes / Heterocycles / Erythrocarine / Tamoxifen

$$R^{1} \longrightarrow R^{2} \longrightarrow R^{1} \longrightarrow R^{2}$$

$$CO_{2} \longrightarrow Ni(0) \longrightarrow R^{1}$$

$$R^{1} \longrightarrow R^{2}$$

$$R^{3}ZnX \longrightarrow R^{3}$$

$$XZn'$$

Highly regio- and stereoselective syntheses of tri- and tetrasubstituted alkenes by introduction of CO₂ and an alkyl group into an alkyne have been developed.

SHORT COMMUNICATIONS

Spirocyclic Pyridoazepines

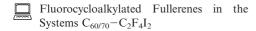
S. Vanlaer, W. M. De Borggraeve, F. Compernolle* 4995–4998

Synthesis of Spirocyclic Pyridoazepines as Analogues of Galanthamine by Nucleophilic Aromatic Substitution of 3-Substituted 2-Chloropyridines

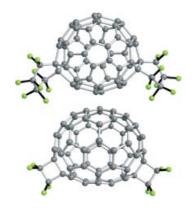
Keywords: Spiro compounds / Nucleophilic aromatic substitution / Nitrogen heterocycles / Acetylcholinesterase / Amines

The synthesis of spirocyclic pyridoazepines starting from easily available precursors is reported. The key step of our synthesis is an intramolecular nucleophilic aromatic substitution of the appropriate 3-substituted 2-chloropyridines. The final compounds, designed as simplified analogues of the alkaloid galanthamine, showed significant acetylcholinesterase inhibition activity.

Fluorinated Fullerenes



Keywords: Fullerenes / Biradical addition / Fluorine / Cycloaddition / Structure elucidation

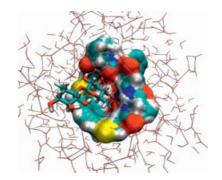


An addition reaction of biradicals thermally generated from $C_2F_4I_2$ was applied to functionalize fullerenes, which resulted in the formation of a number of $C_{60}(C_2F_4)_n$ and $C_{70}(C_2F_4)_m$ compounds. The molecular structures of $C_{60}(C_4F_8)_2$ (two isomers), $C_{60}(C_4F_8)_6$, and $C_{70}(C_2F_4)_2$ demonstrate different modes of [4+2], [4+3], and [2+2] addition, respectively.



FULL PAPERS

An artificial, water-soluble and bicyclic peptide receptor for carbohydrates was constructed and the binding of disaccharides was studied in water by using NMR spectroscopy and mass spectrometry. The receptor contains an aromatic bridge fused to a cyclic dodecapeptide to form a cagelike receptor.



Organic-Peptide Receptors

T. Reenberg, N. Nyberg, J. Ø. Duus, J. L. J. van Dongen, M. Meldal* 5003-5009

Specific Recognition of Disaccharides in Water by an Artificial Bicyclic Carbobydrate Receptor

hydrate Receptor

Keywords: Combinatorial chemistry / Molecular recognition / Carbohydrate sensors / Mass spectrometry / Supramolecular chemistry

Solid-State Fluorescent Dyes

Dramatic 6-alkoxy substituent effects on the solid-state photophysical properties of heterocyclic fluorophores — 6-alkoxy-3-dibutylamino-6-phenylnaphtho[2,3-b]benzofuran-11(6H)-ones (2) — are discussed on the basis of their X-ray crystal structures.

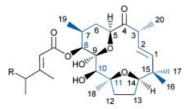
Y. Ooyama,* T. Mamura, K. Yoshida* 5010-5019

Synthesis, X-ray Crystal Structures and Solid-State Fluorescence Properties of 3-Dibutylamino-6-alkoxy-6-phenylnaphtho-[2,3-b]benzofuran-11(6H)-one Derivatives

Keywords: Fluorescent dyes / Solid-state fluorescence / Crystal structures / $\pi - \pi$ Interaction / Substituent effects

Diterpenes

Cembrene A, two diterpenes with the rare flexibilane skeleton (tonantzitlolone and its OAc derivative), two pimaranes, seven kauranes, of which two derivatives are new, three known atisanes, a new trachylobane, and a pentacyclic diterpene with a novel skeleton were isolated from *Stillingia sanguinolenta* and characterized.



R = H Tonantzitlolone R = OAc

G. Dräger, F. Jeske, E. Kunst,
E. G. Lopez, H. V. Sanchez,
F. Tsichritzis, A. Kirschning,*
J. Jakupovic* 5020-5026

Tonantzitlolone and other Diterpenes from *Stillingia sanguinolenta*

Keywords: Diterpenes / Natural products

New supramolecular ensembles involving exTTF and C₆₀ as electron donor and acceptor moieties, respectively, have been synthesized by threading exTTF-containing ammonium salts through a dibenzo-24-

crown-8 ring covalently linked to the fullerene sphere. Different experiments reveal the lack of communication in the ground state and electronic interaction in the excited state.

Supramolecular Threaded Complexes

B. M. Illescas, J. Santos, M. C. Díaz, N. Martín,* C. M. Atienza, D. M. Guldi* 5027-5037

Supramolecular Threaded Complexes from Fullerene—crown Ether and π -Extended TTF Derivatives

Keywords: Supramolecular chemistry / Tetrathiafulvalenes / Fullerenes / Noncovalent interactions

CONTENTS

Stereoselective 1,3-Dipolar Reactions

C. Nájera,* M. de Gracia Retamosa,

J. M. Sansano,* A. de Cózar,

F. P. Cossío 5038-5049

Diastereoselective 1,3-Dipolar Cycloaddition Reactions between Azomethine Ylides and Chiral Acrylates Derived from Methyl (S)- and (R)-Lactate – Synthesis of Hepatitis C Virus RNA-Dependent RNA Polymerase Inhibitors

Keywords: Azomethine ylides / Asymmetric synthesis / Silver / Antiviral / Hepatitis C

Diastereoselective 1,3-dipolar cycloadditions between azomethine ylides and acrylates derived from methyl (R)- or (S)-

lactate were used as key steps for the first asymmetric synthesis of hepatitis C virus inhibitors.

Protecting Groups

J. N. Hernández, F. R. P. Crisóstomo, T. Martín, V. S. Martín* 5050-5058

A Practical Method for Selective Cleavage of a *tert*-Butoxycarbamoyl *N*-Protective Group from *N*,*N*-Diprotected α-Amino Acid Derivatives Using Montmorillonite K-10

Keywords: α-Amino acids / Clays / Heterogeneous catalyst / Protecting groups / Lactones / Asymmetric synthesis

A new, practical, mild, and simple procedure for the selective cleavage of the *tert*-butoxycarbonyl group (Boc) in *N*-Boc-*N*-acyl-diprotected amines was found.

Asymmetric Synthesis

D. K. Mohapatra,* C. Pramanik,

M. S. Chorghade,

M. K. Gurjar 5059-5063

A Short and Efficient Synthetic Strategy for the Total Syntheses of (S)-(+)- and (R)-(-)-Plakolide A

Keywords: Inhibitors / Epoxidation / Olefination / Cross-coupling

Concise and efficient total syntheses of anticancer agents (S)-(+)- and (R)-(-)-Plakolide A were accomplished in eight steps in an overall yield of 39% starting from ger-

aniol. The key steps in our strategy are Sharpless asymmetric epoxidation, double elimination, and Stille coupling reactions.

Pheromone Synthesis

R. A. Fernandes* 5064-5070

An Efficient Synthesis of (-)-Posticlure: The Sex Pheromone of *Orgyia postica*

Keywords: Pheromones / *Orgyia postica* / Asymmetric synthesis / Epoxides / Diethyl L-tartrate

HO
$$CO_2Et$$
HO CO_2Et
O OBn

H (-)-posticlure

An efficient multigram synthesis of (-)-posticlure, the first *trans*-epoxide sex pheromone found in *Orgyia postica*, from diethyl L-tartrate is described. The synthesis

features double-Wittig olefination and a stereoselective one-pot conversion of diol to epoxide. The synthesis was completed in seven steps and 27 % overall yield.



Planar Oligonitriles

2,2,3,3-Tetramethylsuccinonitrile (1) and lithium amides provide access to dihydropyrroles **2** which give *N*-acyloligonitriles **5** upon treatment with 1-oxa-3,5-diazinium

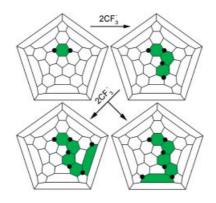
salts 4. Compounds 5 consist of a planar heterocyclic part and a three-dimensional oligonitrile chain in the crystalline state (X-ray diffraction).

(5-Imino-4,5-dihydro-3*H*-pyrrol-2-yl)amines as Sterically Restrained 1,3,5-Triazapenta-1,3-dienes: Useful Building Blocks for the Synthesis of Oligonitriles

Keywords: Oligonitriles / Dihydropyrroles / Hydrogen bonds / Imines / Amidines / Cyanides

Trifluoromethylated Fullerenes

A number of $C_{60}(CF_3)_n$ compounds (n=2-10) have been synthesized and isolated by means of HPLC. This resulted in the first crystal structure determination of six lower $C_{60}(CF_3)_n$ derivatives with n=2 (1 isomer), 4 (two isomers), and 6 (three isomers). A kinetic model of sequential trifluoromethylation based on the Bell–Evans–Polanyi principle has been used to explain the experimentally observed isomeric distribution.



Synthesis, Structure, and Theoretical Study of Lower Trifluoromethyl Derivatives of [60]Fullerene

Keywords: Fullerenes / Trifluoromethylation / Crystal structure / Reaction mechanisms / Density functional calculations

Ionic Liquids

Basic ionic liquids with tertiary aliphatic amine substituents can serve as both solvents and bases for Heck and Sonogashira reactions, homocoupling reactions of terminal alkynes, and reagents for the preparation of palladium nanoparticles.

$$R \leftarrow X + \underbrace{R' \xrightarrow{-NTf_2}}_{1 \text{ mol}-\$ \text{ Pd}(PPh_3)_2Cl_2} R \leftarrow X'$$
No base

C. Ye, J.-C. Xiao, B. Twamley, A. D. LaLonde, M. G. Norton, J. M. Shreeve* 5095-5100

Basic Ionic Liquids: Facile Solvents for Carbon-Carbon Bond Formation Reactions and Ready Access to Palladium Nanoparticles

Keywords: Ionic liquids / Sonogashira reaction / Knoevenagel reaction / Heck reaction / Palladium

Fluorinated Dihydrofuranones

Dihydrofuranones possessing fluorinated double bond systems afforded Diels—Alder reaction products with preferential *exo* configurations in reactions with diphenylisobenzofuran or cyclopentadiene.

Fluorinated Furan-2(5*H*)-ones: Reactivity and Stereoselectivity in Diels-Alder Reactions

Keywords: Cycloaddition / DFT calculations / Diels—Alder reactions / Fluorinated heterocycles / Lactones

CONTENTS

Bis(pyrazole) Ligands

A. S. Potapov, G. A. Domina,

A. I. Khlebnikov,*

V. D. Ogorodnikov 5112-5116

Facile Synthesis of Flexible Bis(pyrazol-1-yl)alkane and Related Ligands in a Superbasic Medium

Keywords: Pyrazole derivatives / Superbasicsystems / Chelating ligands / Iodination / Flexible ligands

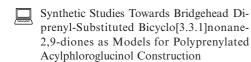
$$R \xrightarrow{R} \begin{array}{c} R \\ \text{Hal} \searrow X \searrow \text{Hal} \\ \text{DMSO/KOH} \end{array} \xrightarrow{R} \begin{array}{c} R \\ N \\ N \\ N \\ X \searrow N \end{array} \xrightarrow{R} \begin{array}{c} R \\ \text{He} \\ X = -\text{CH}_2 -; -\text{CH}_2 \text{OCH}_2 -; -\text{CH}_2 \text{NHCH}_2 -; -\text{CH}_2 -; -\text{CH}_2$$

A facile synthetic procedure for the preparation of 1,3-bis(pyrazol-1-yl)propanes, bis[2-(pyrazol-1-yl)ethyl] ethers, and bis[2-(3,5-dimethylpyrazol-1-yl)ethyl]amine by the reaction of pyrazoles with 1,3-dibromo-

propane, bis(2-chloroethyl) ether, or bis(2-chloroethyl)amine hydrochloride in a superbasic medium (dimethyl sulfoxide/potassium hydroxide) was proposed.

Cyclohexanone α,α' -Annulation

T. Pouplin, B. Tolon, P. Nuhant, B. Delpech,* C. Marazano* 5117-5125



Keywords: Enol lactones / Reduction / Rearrangement / Michael addition / Aldol reactions / Acylphloroglucinols

Bridgehead diprenyl-substituted bicyclo-[3.3.1]nonane-2,9-diones have been prepared as models for the synthesis of polyprenylated acylphloroglucinols (PPAPs). This has been achieved, starting from 2,6-diprenylcyclohexanones, either by a re-

ductive rearrangement of an enol lactone or more directly by a one-step sequence involving Michael addition onto acrolein and aldol reaction followed in both cases by oxidation.

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