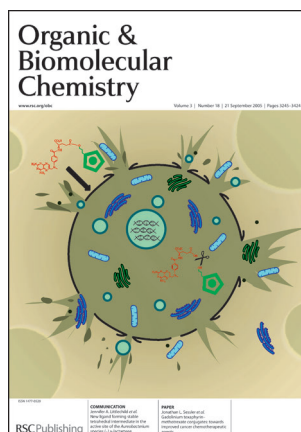


**Cover**

See Zhong-Lin Lu, Alexei A. Neverov and R. Stan Brown, pp. 3379–3387. The complex Pd(*N,N*-dimethylbenzylamine)(pyridine) (trifluoromethanesulfonate) shown in the ORTEP background on the cover is extremely effective for catalytic methanolysis of P=S pesticides. The active form shown as the space-filling model is one having an Pd<sup>II</sup>-coordinated methoxide generated with a *pK<sub>a</sub>* of 10.8. This system promotes the turnover methanolysis of excess fenitrothion, diazinon, quinalphos, coumaphos and dichlofenthion at 25 °C. An associative mechanism is proposed where the Pd<sup>II</sup>-coordinated methoxide intramolecularly attacks a transiently coordinated S=P substrate.

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**Inside cover**

See Wen-Hao Wei, Mark Fountain, Darren Magda, Zhong Wang, Phil Lecane, Mimi Mesfin, Dale Miles and Jonathan L. Sessler, pp. 3290–3296.

The illustration provides a schematic representation of the anti-proliferative effect produced by an ester-linked conjugate between methotrexate, an anti-folate cancer chemotherapeutic, and motexafin gadolinium, an experimental cancer agent demonstrating selective tumour localization. The ester linkage undergoes cleavage within the A549 human lung cancer cells used for analysis and, presumably as a consequence, this conjugate displays greater activity at short incubation times than does methotrexate alone. Neither the amide conjugate, which is stable under these *in vitro* conditions, nor motexafin gadolinium on its own, shows any appreciable activity under these conditions.

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## C65

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**Chemical Science**

September 2005/Volume 2/Issue 9

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## COMMUNICATIONS

## 3257

**Controlled intracellular localization and enhanced antisense effect of oligonucleotides by chemical conjugation**

Takanori Kubo, Zhivko Zhelev, Bakalova Rumiana, Hideki Ohba, Keiko Doi and Masayuki Fujii\*

Antisense oligonucleotides conjugated with nuclear localizing signals could inhibit human telomerase in human leukemia cells much more strongly than phosphorothioate oligonucleotides.



5'-CAGTTAGGGTTAG-3'

Antisense oligonucleotide-nuclear localization signal peptide conjugate could inhibit human telomerase in 99.6 %

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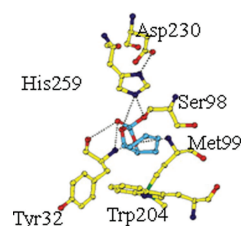
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3260

**Synthesis and characterisation of a ligand that forms a stable tetrahedral intermediate in the active site of the *Aureobacterium* species (–)  $\gamma$ -lactamase**

Stephen Connelly, Kirsty Line, Michail N. Isupov and Jennifer A. Littlechild\*

Synthesis of a trapped tetrahedral intermediate identified in the crystal structure of  $\gamma$ -lactamase and characterisation of its inhibitory activity.

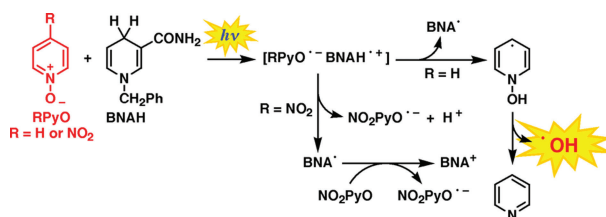


3263

**Hydroxyl radical generation *via* photoreduction of a simple pyridine *N*-oxide by an NADH analogue**

I. Nakanishi,\* C. Nishizawa, K. Ohkubo, K. Takeshita, K. T. Suzuki, T. Ozawa, S. M. Hecht, M. Tanno, S. Sueyoshi, N. Miyata, H. Okuda, S. Fukuzumi, N. Ikota\* and K. Fukuhara\*

Photoreduction of pyridine *N*-oxide, which has a key structure of antitumor agents for hypoxic solid tumors, by 1-benzyl-1,4-dihydronicotinamide (BNAH) in deaerated aprotic media resulted in generation of hydroxyl radical ( $\cdot\text{OH}$ ), leading to the oxidation of salicylic acid to 2,3- and 2,5-dihydroxybenzoic acids, and catechol.

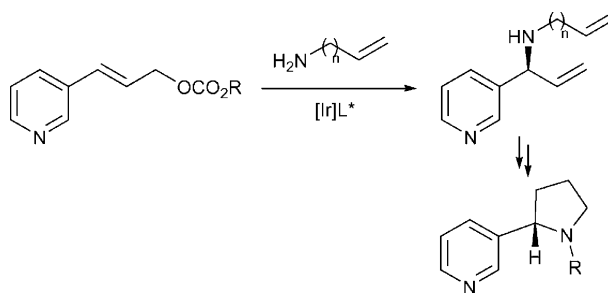


3266

**Enantioselective synthesis of (+)(*R*)- and (–)(*S*)-nicotine based on Ir-catalysed allylic amination**

Carolyn Welter, Rosa M. Moreno, Stephane Streiff and Günter Helmchen\*

The synthesis of nicotine with enantiomeric excess of >99% ee was accomplished by asymmetric Ir-catalysed allylic amination followed by ring closing metathesis and racemisation-free double bond reduction.

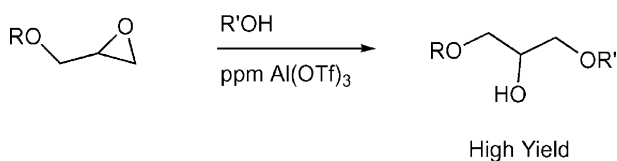


3269

**Aluminium triflate: a remarkable Lewis acid catalyst for the ring opening of epoxides by alcohols**

D. Bradley G. Williams\* and Michelle Lawton

$\text{Al}(\text{OTf})_3$  was found to be an extremely effective catalyst (at ppm levels) for ring opening reactions of epoxides using a range of alcohols.



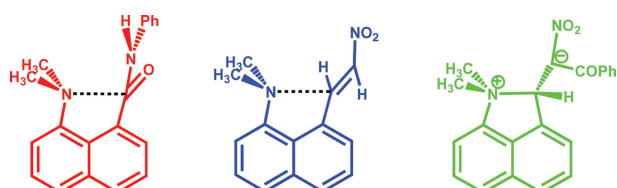
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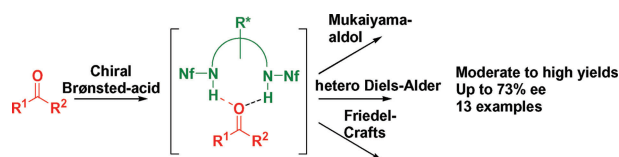
**Structural studies of *peri*-interactions and bond formation between electron-rich atomic centres and *N*-phenylcarboxamides or nitroalkenyl groups**

Jane O'Leary, Xavier Formosa, Wolfgang Skranc and John D. Wallis\*

Dimethylamino groups either make *peri*-interactions or form a bond giving a zwitterion depending on the adjacent electrophilic group.



3284

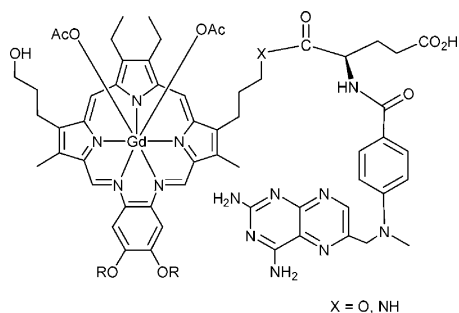


### A versatile catalyst for asymmetric reactions of carbonyl groups working purely by activation through hydrogen bonding: Mukaiyama-aldol, hetero Diels–Alder and Friedel–Crafts reactions

Wei Zhuang, Thomas B. Poulsen and Karl Anker Jørgensen\*

$C_2$ -Symmetric chiral bis-sulfonamides are demonstrated to be effective Brønsted-acid catalysts leading to the optically active products in moderate to excellent yields and with moderate enantioinduction.

3290

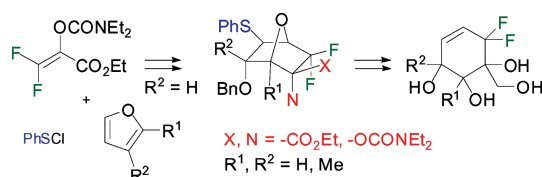


### Gadolinium texaphyrin–methotrexate conjugates. Towards improved cancer chemotherapeutic agents

Wen-Hao Wei, Mark Fountain, Darren Magda,\* Zhong Wang, Phil Lecane, Mimi Mesfin, Dale Miles and Jonathan L. Sessler\*

Ester linked methotrexate–motexafin gadolinium conjugates showed enhanced *in vitro* activity (A549 cells) at early incubation times relative to methotrexate alone.

3297

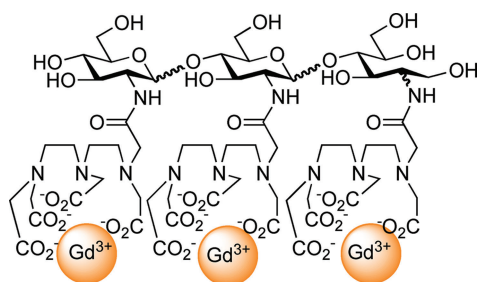


### Highly-functionalised difluorinated cyclohexane polyols *via* the Diels–Alder reaction: regiochemical control *via* the phenylsulfonyl group

Patrick J. Crowley, John Fawcett, Gerry A. Griffith, Andrew C. Moralee, Jonathan M. Percy\* and Vittoria Salafia

A difluorinated dienophile underwent cycloaddition reactions with a range of furans to afford cycloadducts which could be processed regio- and stereoselectively *via* episulfonium ions. The oxabicyclic products were ring opened *via*  $E1_C B$  or reductive desulfonative pathways to afford, ultimately, difluorinated cyclohexene or cyclohexane polyols.

3311

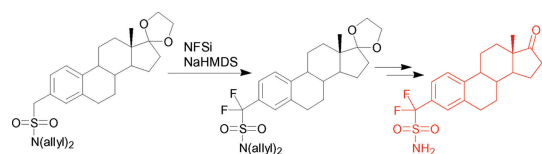


### Synthesis of DTPA-conjugated (1,4)-linked 2-aminoglycosides varying in the anomeric configuration and their MRI contrast effect

Hiroshi Tanaka, Yoshio Ando, Masatoshi Wada and Takashi Takahashi\*

The efficient synthesis of DTPA-conjugated oligosaccharides composed of  $\alpha$ - and/or  $\beta$ -linked tri- to monoglucosamines and MRI contrast effects of their Gd(III) complexes are presented.

3329



### Synthesis of a non-hydrolyzable estrone sulfate analogue bearing the difluoromethanesulfonamide group and its evaluation as a steroid sulfatase inhibitor

Yong Liu, Vanessa Ahmed, Bryan Hill and Scott D. Taylor\*

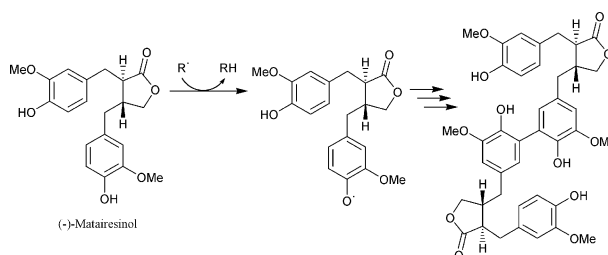
A non-hydrolyzable estrone sulfate analogue bearing an  $\alpha, \alpha$ -difluorosulfonamide moiety at the 3-position on the A-ring was synthesized and reversibly inhibited steroid sulfatase (STS) with a pH dependence opposite to that of other reversible STS inhibitors.

3336

### Chemical studies on antioxidant mechanisms and free radical scavenging properties of lignans

Patrik C. Eklund,\* Otto K. Långvik, Johan P. Wärnå, Tapio O. Salmi, Stefan M. Willför and Rainer E. Sjöholm\*

The radical scavenging mechanism of lignans, in terms of their radical donating ability, was investigated. The mechanism is proposed to proceed *via* the formation of phenoxyl radical, followed by inter- or intramolecular termination reactions.

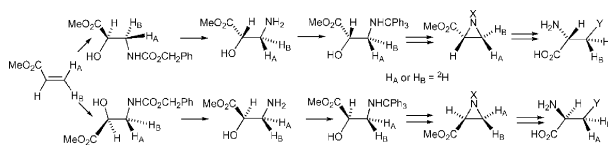


3348

### A short, versatile chemical synthesis of L- and D-amino acids stereoselectively labelled solely in the *beta* position

Kreingkrai Lowpetch and Douglas W. Young\*

A synthesis of L and D amino acids is reported which relies on the conversion of specifically labelled acrylates into optically pure aziridines. The aziridines were treated with a range of nucleophiles to give the title amino acids after hydrolysis.

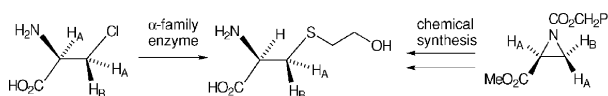


3357

### Stereochemistry of reactions of the inhibitor/substrates L- and D- $\beta$ -chloroalanine with $\beta$ -mercaptoethanol catalysed by L-aspartate aminotransferase and D-amino acid aminotransferase respectively

Benjamin Adams, Kreingkrai Lowpetch, Faye Thorndycroft, Sheena M. Whyte and Douglas W. Young\*

L-Aspartate aminotransferase, a member of the  $\alpha$ -family of PLP-dependent enzymes, catalyses  $\beta$ -substitution of L- $\beta$ -chloroalanine with retention of stereochemistry. This is the stereochemical outcome typical of reactions catalysed by enzymes of the  $\beta$ -family of PLP-dependent enzymes.

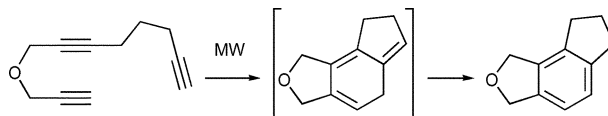


3365

### Non-metal-catalysed intramolecular alkyne cyclotrimerization reactions promoted by focussed microwave heating in batch and flow modes

Steen Saaby, Ian R. Baxendale and Steven V. Ley\*

A number of oligomeric alkynes underwent [2 + 2 + 2] intramolecular trimerization to afford arenes under metal-free conditions using focussed microwave heating.

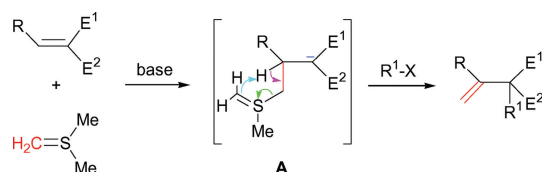


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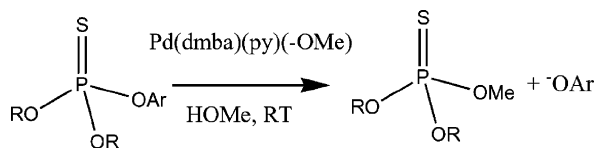
### The unprecedented reaction of dimethylsulfonium methylide with Michael acceptors: synthesis of 1-substituted vinyl silanes and styrenes

Sonali M. Date, Rekha Singh and Sunil K. Ghosh\*

An unprecedented cycloelimination from the adduct of dimethylsulfonium methylide and an activated olefin *via* A has been established leading to a functionalized 1-substituted alkene.



3379

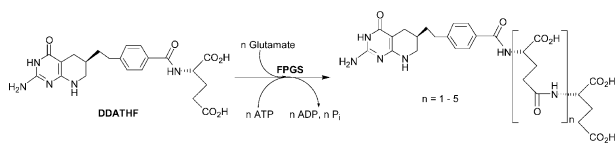


**An *ortho*-palladated dimethylbenzylamine complex as a highly efficient turnover catalyst for the decomposition of P=S insecticides. Mechanistic studies of the methanolysis of some P=S-containing phosphorothioate triesters**

Zhong-Lin Lu, Alexei A. Neverov and R. Stan Brown\*

The Pd(*N,N*-dimethylbenzylamine)(pyridine)(<sup>-</sup>OCH<sub>3</sub>) palladacycle catalyzes the methanolysis of the P=S pesticides fenitrothion (**3**), diazinon (**4**), quinalphos (**5**), coumaphos (**10**) and dichlofenthion (**11**).

3388

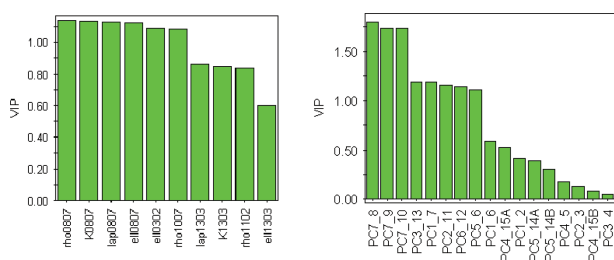


**Synthesis of (6*R*)- and (6*S*)-5,10-dideazatetrahydrofolate oligo- $\gamma$ -glutamates: Kinetics of multiple glutamate ligations catalyzed by folylpoly- $\gamma$ -glutamate synthetase**

John W. Tomsho, John J. McGuire and James K. Coward\*

Synthesis of two 5,10-dideazatetrahydrofolate diastereomers and poly- $\gamma$ -glutamate derivatives provides steady-state kinetics data as substrates for folylpoly- $\gamma$ -glutamate synthetase.

3399

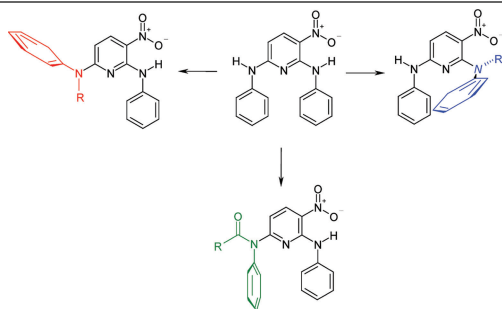


**Quantum chemical topology (QCT) descriptors as substitutes for appropriate Hammett constants**

P. J. Smith and P. L. A. Popelier\*

Quantum topological molecular similarity (QTMS) is used to construct a variety of medicinal, ecological and physical organic quantitative structure–activity relationships and quantitative structure–property relationships.

3408

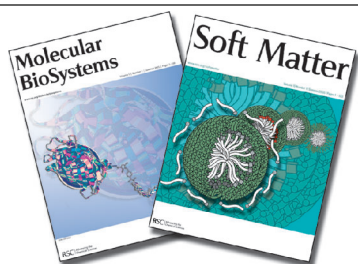


**Synthesis and conformational properties of 2,6-bis-anilino-3-nitropyridines**

Sylvia Schmid, Martin Röttgen, Ulf Thewalt and Volkhard Austel\*

Characteristic changes in conformational preferences are associated with substitution at the anilino nitrogen atoms of 2,6-bis-anilino-3-nitropyridines.

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**Synthetic applications of aliphatic unsaturated  $\alpha$ -H- $\alpha$ -amino acids**

Jasper Kaiser, Sape S. Kinderman, Bart C. J. van Esseveldt, Floris L. van Delft, Hans E. Schoemaker, Richard H. Blaauw and Floris P. J. T. Rutjes (DOI: 10.1039/b507973j)

**Glycosylation with *in situ* separation: carbohydrate chemistry on a TLC plate**

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