

# Review: JACS, 1997

Ruben Eckermann

Group seminar Kalesse & Gaich  
16.11.2011



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Universität  
Hannover

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## Publication Record

- 12987 pages
- 2754 papers in total
- 40 „Total Syntheses“ as research topic

## Notable events

- Tony Blair got Prime Minister & Bill Clinton began his 2nd term
- Funeral of Diana, Princess of Wales
- clone sheep Dolly got presented
- Fuck parade in Berlin was established as protest against the commercialization of the Love parade
- Mutter Teresa died

## Most cited papers

- 1) (1336) Epitaxial Growth of Highly Luminescent CdSe/CdS Core/Shell Nanocrystals with Photostability and Electronic Accessibility  
By Peng, Xiaogang; Schlamp, Michael C.; Kadavanich, Andreas V.; Alivisatos, A. P. From Journal of the American Chemical Society (1997), 119(30), 7019-7029.
- 2) (790) How Easily Oxidizable Is DNA? One-Electron Reduction Potentials of Adenosine and Guanosine Radicals in Aqueous Solution  
By Steenken, Steen; Jovanovic, Slobodan V. From Journal of the American Chemical Society (1997), 119(3), 617-618.
- 3) (738) Controlled//“Living” Radical Polymerization. Kinetics of the Homogeneous Atom Transfer Radical Polymerization of Styrene  
By Matyjaszewski, Krzysztof; Patten, Timothy E.; Xia, Jianhui From Journal of the American Chemical Society (1997), 119(4), 674-680.

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**Detailed Syntheses**

( $\pm$ )-Gelsemine  
S. Atarashi, 6226-6241

Neotripterifordin  
E. J. Corey, 9929-9930

( $\pm$ )-Scopadulic acid B  
L. E. Overman, 12031-12040

( $\pm$ )-K252a  
J. L. Wood, 9641-9651

**Concise Syntheses**

Hispidospermidin  
S. J. Danishefsky, 6686-6687

Roseophilin  
A. Fürstner, 2944-2945

(-)-Mesembrine  
S. E. Denmark, 1675-1686

(+)-Epoxydictyamene  
L. A. Paquette, 8438-8450

(+)-Isolaurepinnacin  
L. E. Overman, 2446-2452

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## Detailed Syntheses

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**Synthesis of Neotripterifordin by E. J. Corey, K. Liu; 9929-9930**

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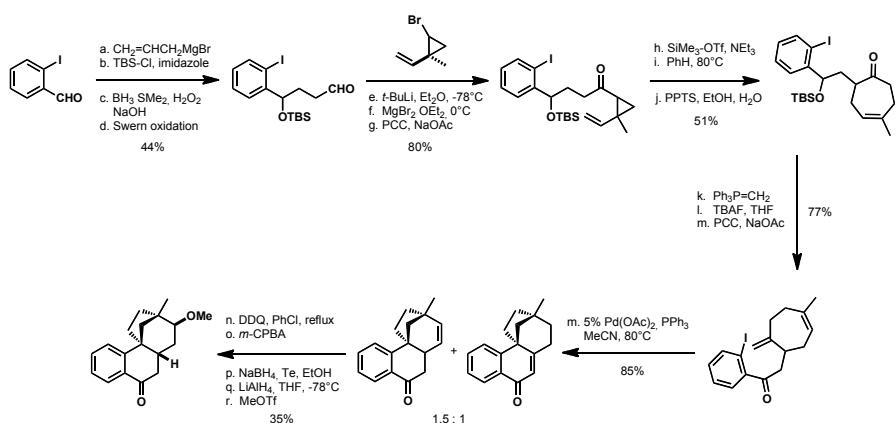
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Synthesis of ( $\pm$ )-Scopadulcic Acid B by L. E. Overman, D. J. Ricca, V. D. Tran; 12031-12040Leibniz  
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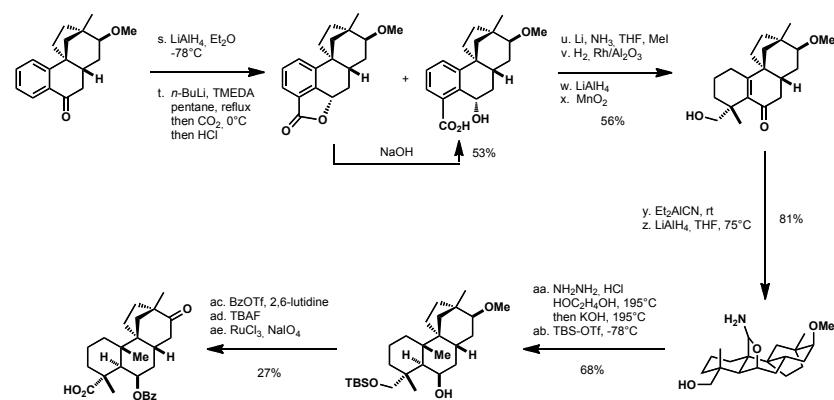
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**Synthesis of ( $\pm$ )-Gelsemine by S. Atarashi, J.-K. Choi, D.-C. Ha, D. J. Hart, D. Kuzmich, C.-S. Lee, S. Ramesh, S. C. Wu; 6226-6241**

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**Synthesis of (-)-Salsolene Oxide** by L. A. Paquette, L.-Q. Sun, T. J. N. Watson, D. Friedrich, B. T. Freeman; 2767-2768

a. DIBAL-H,  $\text{CH}_2\text{Cl}_2$ ,  $-78^\circ\text{C}$   
b. PhSH,  $\text{TiCl}_4$ ,  $\text{NEt}_3$ ,  $\text{DME}$   
c.  $\text{NaI}$ , ac,  $\Delta$   
d. LDA,  $\text{THF}$ ,  $\text{HMPA}$ ,  $-78^\circ\text{C}$ ,  $\text{MeCO}_2\text{C}_2\text{H}_5$   
e. 14M  $\text{KOH}$ ,  $\text{MeOH}$ ,  $\Delta$  then  $\text{H}_3\text{O}^+$   
f.  $(\text{COCl})_2$ ,  $\text{PhH}$  then  $\text{NEt}_3$ ,  $\Delta$   
g.  $\text{H}_2\text{C}=\text{CHLi}$ ,  $\text{THF}$ ,  $-78^\circ\text{C}$

88% 65% 57%

87% 78% 78%

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**Synthesis of (+)-K252** by J. L. Wood, B. M. Stoltz, H.-J. Dietrich, D. A. Pflum, D. T. Petsch; 9641-9651

a.  $\text{KOt-Bu}$ ,  $300^\circ\text{C}$   
b.  $\text{NH}_4\text{Cl}$   
c.  $\text{HO}_2\text{CCH}_2\text{CO}_2\text{Et}$ ,  $\text{DCC}$ ,  $\text{DMAP}$   
d.  $\text{NaOEt}$ ,  $\text{EtOH}$   
e.  $\text{CH}_3\text{CN}$ ,  $\text{H}_2\text{O}$ ,  $\Delta$   
f.  $\text{MsN}_3$ ,  $\text{NEt}_3$ ,  $0^\circ\text{C}$   
g.  $\text{Rh}_2(\text{OAc})_4$ ,  $120^\circ\text{C}$   
h.  $\text{Rh}_2(\text{OAc})_4$ ,  $\text{PhH}$   
i.  $\text{BF}_3 \cdot \text{OEt}_2$   
j.  $\text{O}_2$ ,  $\text{SMe}_2$   
k.  $\text{MeOH}$ ,  $p\text{-TSA}$   
l.  $\text{CSA}$ ,  $48\text{h}$ ,  $\text{C}_2\text{H}_4\text{Cl}_2$

80% 72% 62% 77% 80% 83% 5 : 1 80%

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# Concise Syntheses

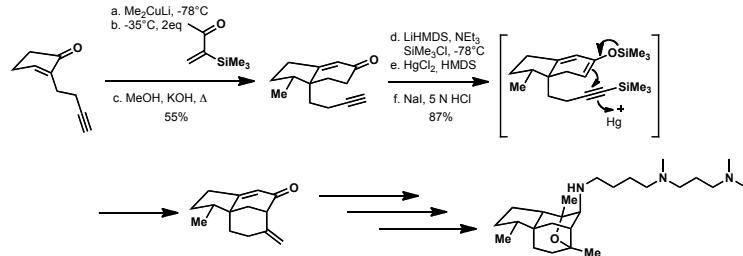
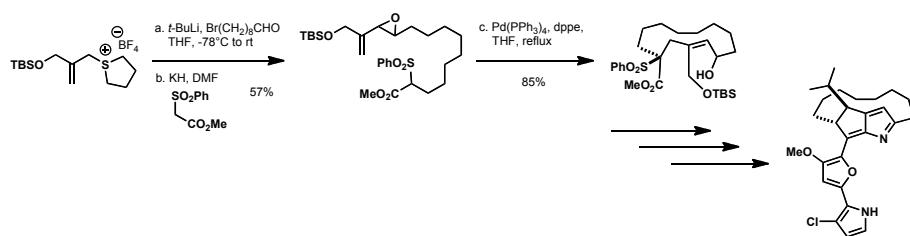
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**Synthesis of Hispidospermidin** by A. J. Frontier, S. Raghavan, S. J. Danishefsky; 6686-6687
**Synthesis of Roseophilin** by A. Fürstner, H. Weinritt; 2944-2945

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**Synthesis of (+)-Isolaurepinnacin by D. Berger, L. E. Overman, P. A. Renhowe; 2446-2452**

a.  $\text{BCl}_3, \text{CH}_2\text{Cl}_2$ , -78°C to 0°C  
b. TBAF, THF  
86%

**Synthesis of Epoxidictymene by L. A. Paquette, L.-Q. Sun, D. Friedrich, P. B. Savage; 8438-8450**

a.  $\text{Cp}_2\text{Ti}^{\text{H}_2}\text{Cl}^{\text{AlMe}_2}$   
b.  $i\text{-Bu}_3\text{Al}, \text{CH}_2\text{Cl}_2, -78^\circ\text{C}$   
c.  $\text{PCC}$   
86%

c.  $\text{I}_2, \text{PhI}(\text{OAc})_2$ , cyclohexane  
 $h\nu, 50^\circ\text{C}$   
95%

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**Synthesis of (-)-Mesembrine by S. E. Denmark, L. R. Marcin; 1675-1686**

a. 1) 1eq MAD, 5min  
2) 3eq olefin, 1h  
3) 1eq MAD, 1h  
PhMe, -10°C

b. PhH,  $\Delta$   
66%  
 $dr = 30/1$

c. Ra-Ni,  $\text{H}_2$   
MeOH  
74%

Mesembrine

Ar = 3,4-dimethoxyphenyl  
MAD:  
olefin:

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# Methods



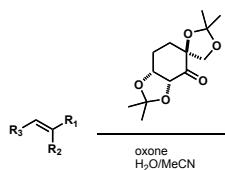
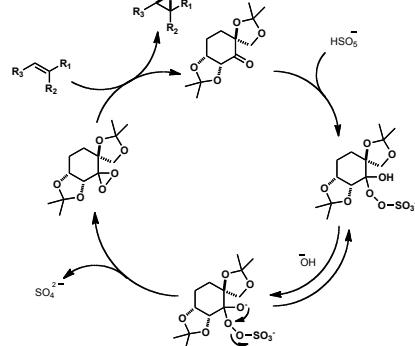
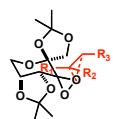
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**Asymmetric Epoxidation** by Z.-X. Wang, Y. Tu, M. Frohn, J.-R. Zhang, Y. Shi; 11224-11235Catalytic Cycle:Transition state:

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**Pseudoephedrine as a chiral auxiliary** by A. G. Myers, B. H. Yang, H. Chen, L. McKinstry, D. J. Kopecky, J. L. Gleason; 6496-6511

X = RCH<sub>2</sub>CO<sub>2</sub>, Cl, t-BuCO<sub>2</sub>, CH<sub>3</sub>O  
R = Me, Bn, n-Bu, Ph, i-Pr, i-Bu, Cl  
CH<sub>2</sub>Bn, 3-pyridyl, 2-thiophene

R' = Bn, n-Bu, BOM, CH<sub>2</sub>Bn, Et  
CH<sub>2</sub>=CHCH<sub>2</sub>, (CH<sub>2</sub>)<sub>2</sub>OTBS

**Transformations:**

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**Pd-catalyzed additions of terminal alkynes to acceptor alkynes** by B. M. Trost, M. T. Sorum, C. Chan, A. E. Harms, G. Rühter; 698-708

TDMPP = tris(2,6-dimethoxyphenyl)phosphine

R' = Me, Ph, Alkyl  
EWG = CO<sub>2</sub>Me, SO<sub>2</sub>Ph, COMe  
R = TMS, Ph, HOCH<sub>2</sub>, PhSO<sub>2</sub>CH<sub>2</sub>  
other donor substituents

**Catalytic Cycle:**

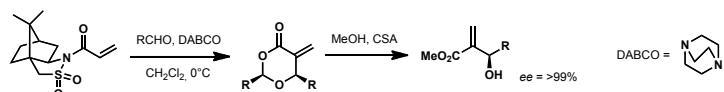
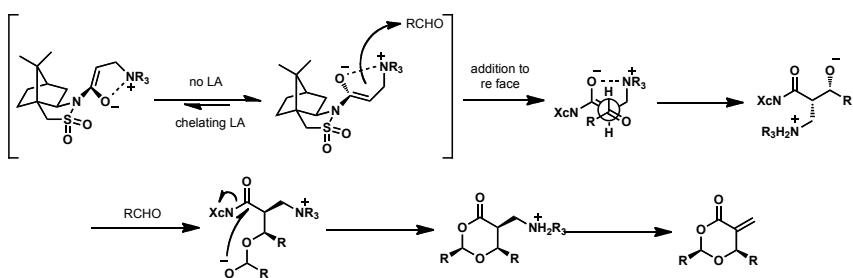
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**Asymmetric Baylis-Hillman reaction** by B. L. J. Brzezinski, S. Rafel, J. W. Leahy; 4317-4318Stereochemical induction:

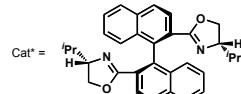
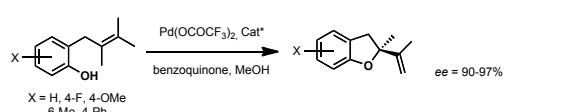
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**Asymmetric Wacker-type cyclization** by Y. Uozumi, K. Kato, T. Hayashi; 5063-5064

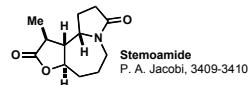
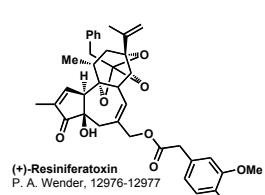
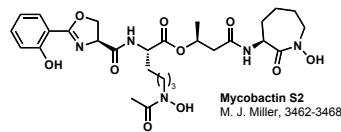
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## Syntheses not covered

