Perkin 1 Abstracts: Solid Phase Organic Synthesis

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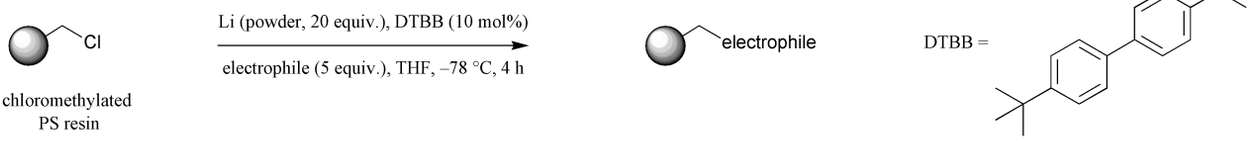
Perkin 1 Abstracts: Solid Phase Organic Synthesis are a selection of significant papers published in the recent literature covering the broad area of Solid Phase Organic Synthesis (SPOS). The abstracts cover preparation of single compounds on solid support as well as combinatorial libraries. Advances in new linker design are also covered.

Functionalised polymers *via* an organolithium reagent on a soluble support. ***Support***

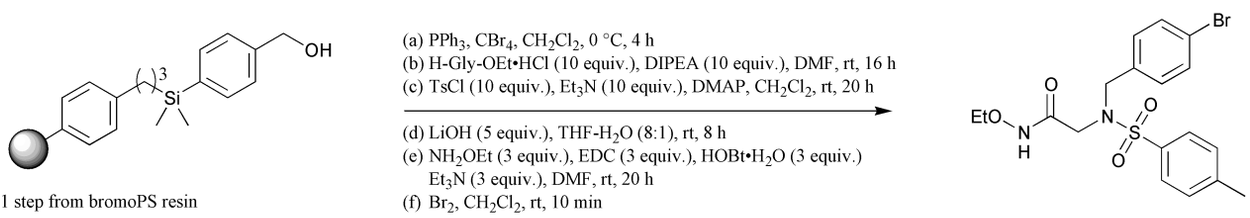


chloromethylated PS resin

M. Yus, C. Gómez and P. Candela, *Tetrahedron Lett.*, 2001, **42**, 3977.

7 examples (yields 48-100%, ^{1}H NMR purity 60-75%). Preparation of the support is also reported.

Traceless silicon-based aromatic transferring linkers. ***Linker***

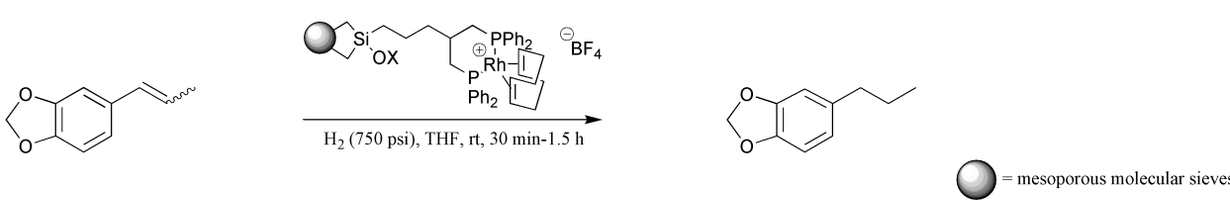


1 step from bromoPS resin

Y. Lee and R. B. Silverman, *Tetrahedron*, 2001, **57**, 5339.

1 example (yield 87%). Preparation of several other arylsilane-based linkers and their application are also reported. Cleavage *via* protiodesilylation is also possible.

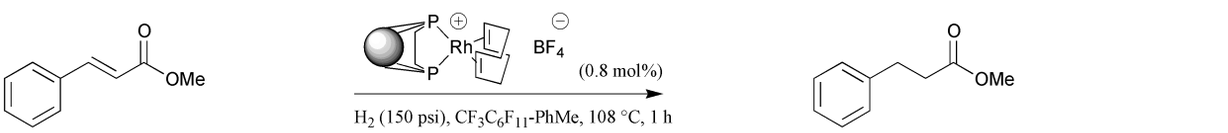
Rhodium bis-phosphine catalysts for alkene hydrogenation. ***Catalyst***



C. M. Crudden, D. Allen, M. D. Mikoluk and J. Sun, *Chem. Commun.*, 2001, 1154.

4 examples using catalysts prepared with different methods of grafting to the solid-support (TON 200-3300). TON and Rh leaching were found to be influenced by the grafting method.

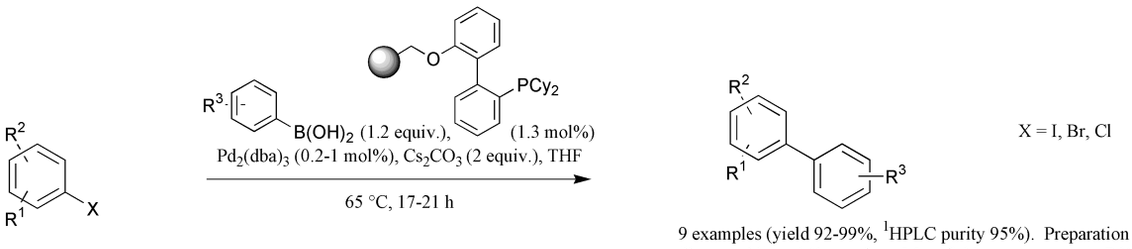
Effects of a fluorous biphase solvent system on a polymer-supported hydrogenation catalyst. ***Catalyst***



S. L. Vinson and M. R. Gagné, *Chem. Commun.*, 2001, 1130.

The illustrated reaction is used to investigate the effect of catalyst recycling and $CF_3C_6H_{11}$-PhMe ratios. An increase in catalyst activity directly correlating to fluorous content of the solvent is reported.

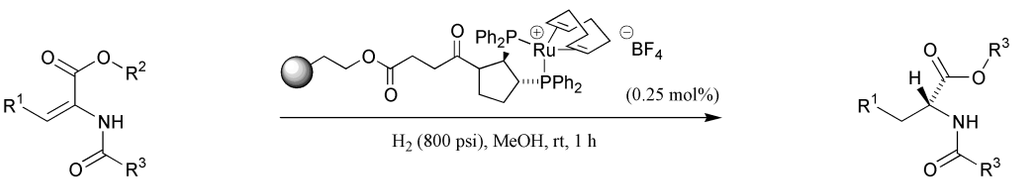
Polymer-supported dialkylphosphinobiphenyl ligands for Pd(0)-catalysed Suzuki and amination reactions. ***Ligand***



9 examples (yield 92-99%, [1]HPLC purity 95%). Preparation of the illustrated ligand (1 step from Merrifield resin), its use in Pd(0) catalysed amination of aryl halides (13 examples, yield 79-99%, HPLC purity 95%) and recycling experiments are also reported.

C. A. Parrish and S. L Buchwald, *J. Org. Chem.,* 2001, **66**, 3820.

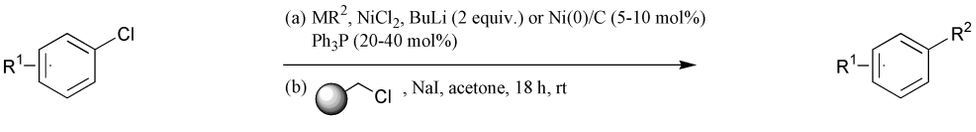
Chiral diphosphine ligands for an asymmetric hydrogenation catalyst. ***Ligand***



5 examples (%ee 93-96). Preparation of the illustrated ligand (1 step from MeO-PEG-OH), 2 other MeO-PEG supported catalysts using BINAP-type ligands and their application to asymmetric hydrogenation (4 examples, %ee 90-96) are also reported.

Q.-H. Fan, G.-J. Deng, C.-C. Lin and A. S. C. Chan, *Tetrahedron: Asymmetry*, 2001, **12**, 1241.

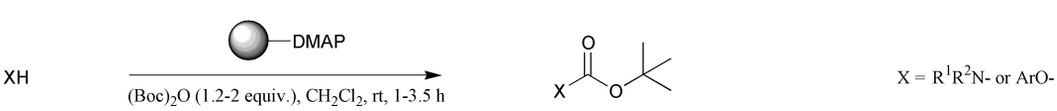
Scavenging of Ph_3P and Ph_3P=O with Merrifield resin. ***Scavenger***



6 examples (yield 82-98%). Application of the Merrifield resin scavenger to the Stille coupling (1 example, yield 93%), the Staudinger reaction (1 example, yield 42%, no reaction with the free amine is observed) and to scavenging of Ph_3P=O are also reported.

B. H. Lipshutz and P. A. Blomgren, *Org. Lett.,* 2001, **3**, 1869.

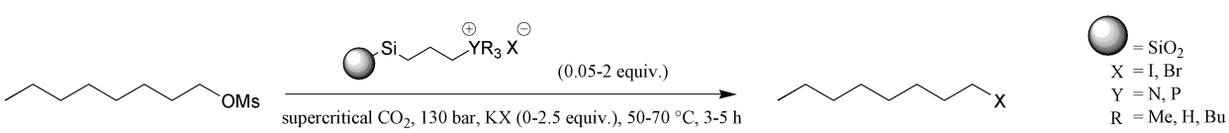
PolyDMAP as a mild reagent for the destruction of excess di-*tert*-butyl dicarbonate. ***Reagent***



3 examples (sample yield 97%). Comparison of polyDMAP with imidazole and trifluoroethanol over the removal of $(Boc)_2O$ is also reported.

Y. Basel and A. Hassner, *Synthesis*, 2001, **4**, 550.

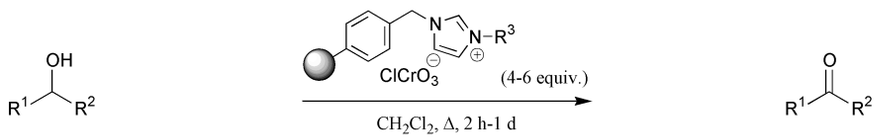
Nucleophilic displacements in supercritical carbon dioxide. ***Reagent***



10 examples (sample yield 90-95%).

J. DeSimone, M. Selva and P. Tundo, *J. Org. Chem*., 2001, **66**, 4047.

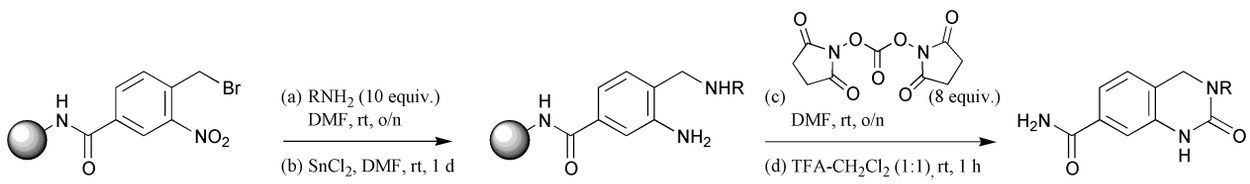
1-Aminoimidazolium chlorochromates as selective, recyclable oxidants. ***Reagent***



M. L. Linares, N. Sánchez, R. Alajarín, J. J. Vaquero and J. Alvarez-Builla, *Synthesis*, 2001, **3**, 382.

21 examples of the oxidation of benzylic and cinnamylic alcohols (yield 51-99%). Preparation (3 steps from Merrifield resin) and recycling of the polymer-supported chlorochromates are also reported.

3,4-Dihydroquinazolin-2(1*H*)-ones and 3,4-dihydro-1*H*-quinazolin-2-thiones.

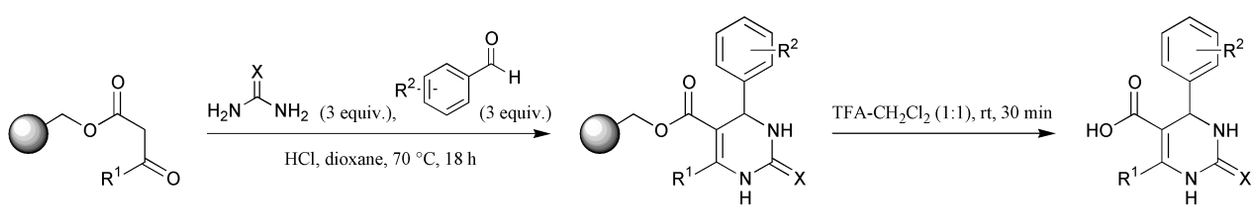


2 steps from PS Rink-NH-Fmoc resin

11 examples (yield 70-100%, HPLC purity 60-95%). Preparation of 3,4-dihydro-1*H*-quinazolin-2-thiones (11 examples, yield 72-97%, HPLC purity 60-89%) *via* a similar route is also reported.

Q. Sun, X. Zhou and D. J. Kyle, *Tetrahedron Lett.,* 2001, **42**, 4119.

Dihydropyrimidones *via N*-acyliminium ion-based α-ureidoalkylations.



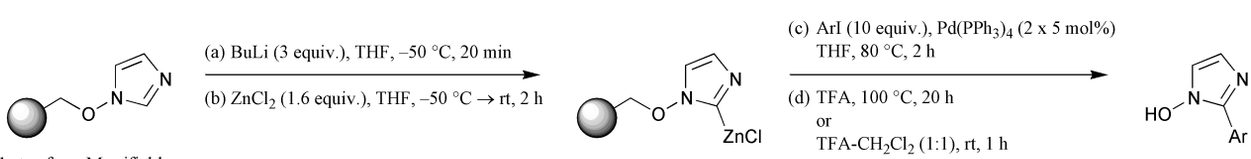
1 step from Wang resin

X = O, S

10 examples, (yield 52-88%, average ^{1}H NMR purity 95%). An orthogonal route using basic conditions (10 examples, yield 30-60%, average ^{1}H NMR purity 95%) is also reported.

M. G. Valverde, D. Dallinger and C. O. Kappe, *Synlett,* 2001, **6**, 741.

Pd(0)-catalysed arylation of polymer-supported imidazol-2-ylzinc chlorides.

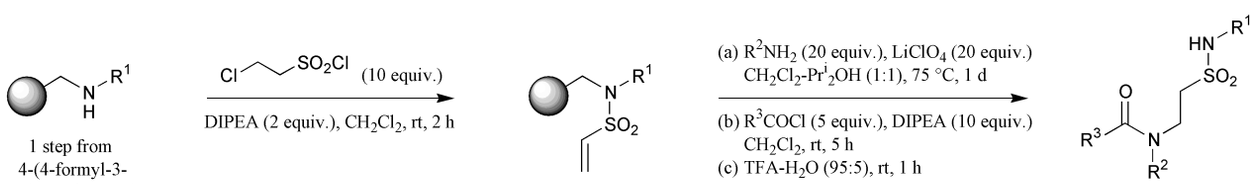


1 step from Merrifield or Wang resin

18 examples (yield 0-93%). Optimisation of the reaction conditions is also reported.

S. Havez, M. Begtrup, P. Vedsø, K. Anderson and T. Ruhland, *Synthesis*, 2001, **6**, 909.

Michael addition of amines to vinyl sulfonamides.



1 step from 4-(4-formyl-3-methoxyphenoxy)-butyryl AM resin

2 examples (HPLC purity 99%). Optimisation of the reaction conditions is also reported.

G. M. Makara and Y. Mao, *Tetrahedron Lett*., 2001, **42**, 4123.

Traceless synthesis of urea, semicarbazide and carbamate derivatives.

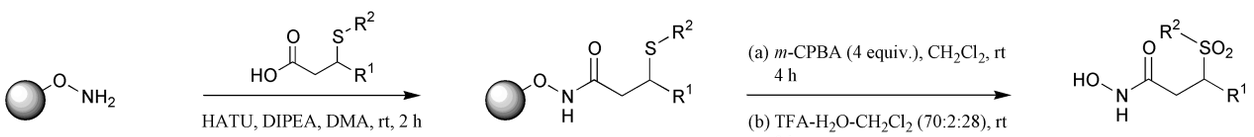
Wang Bromo resin

X = O, S

14 examples (yield 68-97%, HPLC purity 13-97%). Preparation of semicarbazide and carbamate derivatives *via* a similar route (6 examples, yield 72-92%, HPLC purity 35-93%) and use of bromo-Wang SynPhase™ Lantern resin are also reported.

C. W. Phoon and M. M. Sim, *Synlett*, 2001, **5**, 697.

β-Sulfinyl- and β-sulfonyl-hydroxamic acids.

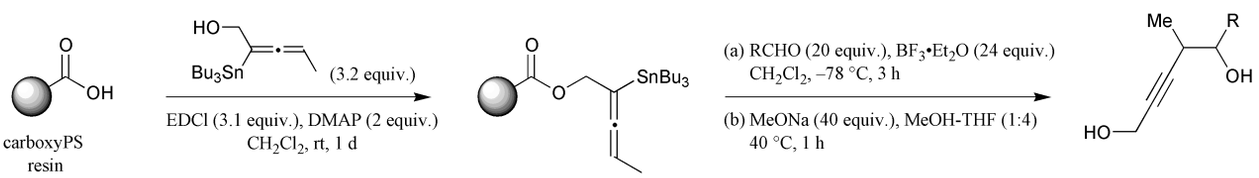


hydroxylamine Wang resin

6 examples (yield 14-29%, HPLC purity 90-95%). Preparation of sulfoxides *via* a similar route (5 examples, yield 10-71%, HPLC purity 80-95%) is also reported.

G. Rossé, F. Gerber, J.-L. Specklin and C. Hubschwerlen, *Synlett*, 2001, **4**, 538.

Diastereoselective synthesis of homopropargylic alcohols.



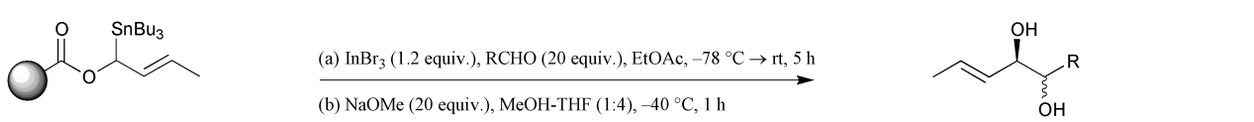
carboxyPS resin

EDCl = $(CH_2)_2N{=}C{=}N(CH_2)_3N(CH_3)_2{\bullet}HCl$

5 examples (yield 43-76%, *syn*:*anti* ratio 35:65–95:5). Preparation of homopropargylic alcohols using InBr mediation (5 examples, yield 36-80%, *syn*:*anti* ratio 40:60-98:2) is also reported.

J. Cossy, M. Defosseux and C. Meyer, *Synlett*, 2001, **6**, 815.

1,2-Diols and γ-lactones.

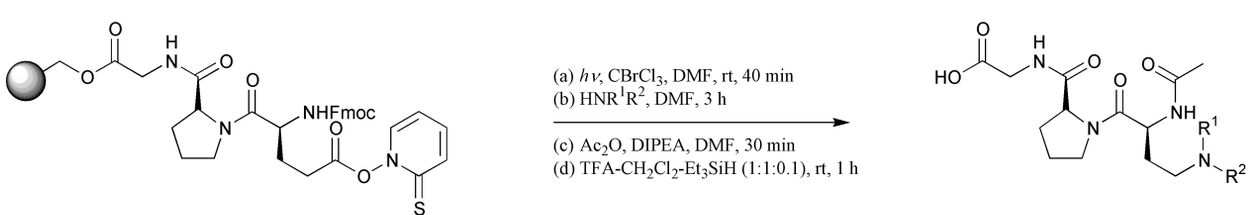


1 step from carboxylic PS resin

5 examples (yield 50-90%, %de 0-100). Preparation of γ-lactones (6 examples, yield 40-75%, %de 6-100) by warming the reaction mixture to rt prior to addition of the aldehyde is also reported.

J. Cossy, C. Rasamison, D. G. Pardo and J. A. Marshall, *Synlett*, 2001, **5**, 629.

Barton radical decarboxylation.



from Wang resin

3 examples (yield 65-75%). Fmoc deprotection and amine modification prior to photochemical radical decarboxylation, and reaction of the bromide intermediate with various nucleophiles (4 examples, yield 45-65%) are also reported.

M. E. Attardi and M. Taddei, *Tetrahedron Lett.*, 2001, **42**, 3519.