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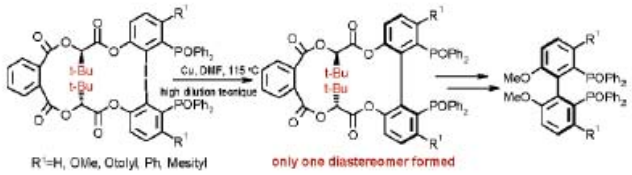
Citation:

Diastereospecific Intramolecular Ullmann Couplings: Unique Chiral Auxiliary for the Preparation of 3,3'-Disubstituted MeO-BIPHEP Derivatives
E. Gorobets, R. McDonald, and B. A. Keay
pp 1483 – 1485.

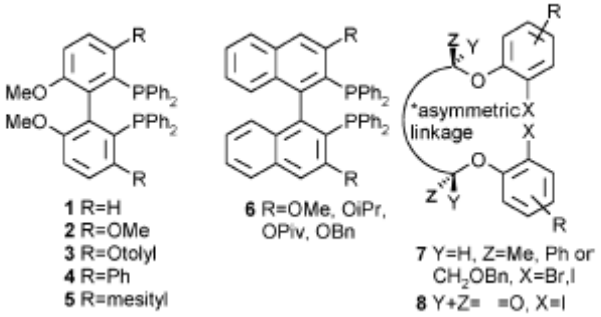
Abstract:



A chiral auxiliary is described that provides only one diastereomer during *intra*molecular Ullmann couplings. Treatment of five Ullmann coupling precursors with Cu powder in DMF at 115 °C provides 2,2′,3,3′,6,6′-hexasubstituted 1,1′-biphenyls as single diastereomers in yields ranging from 66% to 91%.

Schemes:

Scheme 1

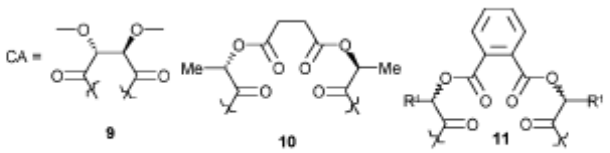


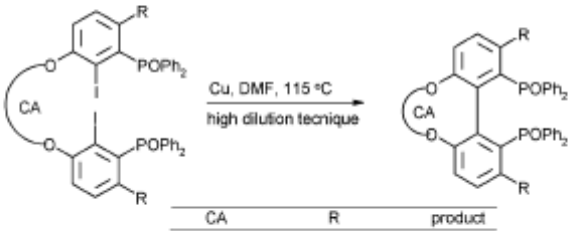
1 R=H
2 R=OMe
3 R=Otolyl
4 R=Ph
5 R=mesityl

6 R=OMe, OiPr, OPiv, OBn

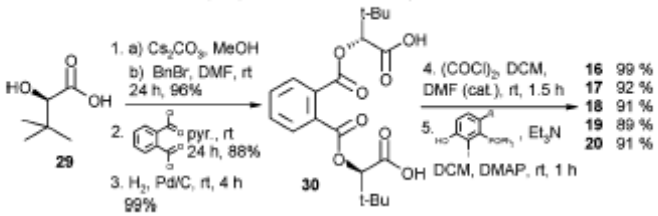
7 Y=H, Z=Me, Ph or CH_2OBn, X=Br,I
8 Y+Z= =O, X=I

Scheme 2

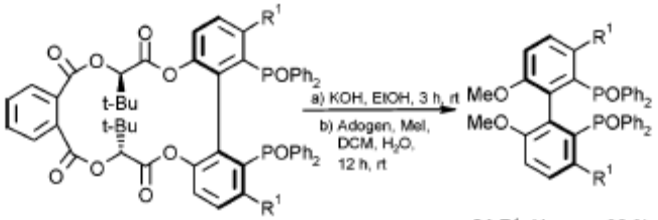




	CA	R	product
13	**9**	H	**21**
14	**10**	H	**22**
15	(*S,S*)-**11** R¹=Me	H	**23**
16	(*R,R*)-**11** R¹=*t*-Bu	H	**24**
17	(*R,R*)-**11** R¹=*t*-Bu	OMe	**25**
18	(*R,R*)-**11** R¹=*t*-Bu	OTol	**26**
19	(*R,R*)-**11** R¹=*t*-Bu	Ph	**27**
20	(*R,R*)-**11** R¹=*t*-Bu	Mesityl	**28**



Scheme 3



24 R¹=H
25 R¹=OMe
26 R¹=Otolyl
27 R¹=Ph
28 R¹=Mesityl

31 R¹=H 93 %
32 R¹=OMe 91 %
33 R¹=Otolyl 93 %
34 R¹=Ph 89 %
35 R¹=mesityl 91 %

Figures:

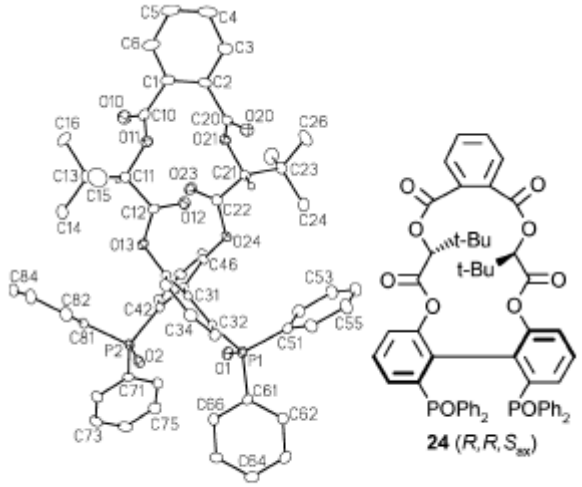


Figure 1. X-ray crystal structure of (R,R,S_{ax})-**24**. The hydrogen atoms have been removed for clarity.

Tables:

Table 1. Products Ratios from the Intramolecular UC of Compounds **9**−**17**

sm	deiodized sm/UC product (config)	UC product (% isolated yield)	% de
(*R*,*R*)-**13**	95:5 (nd)	**21** (nd)	
(*S*,*S*)-**14**	23:77 (R_{ax})	**22** (69)	71
(*S*,*S*)-**15**	10:90 (R_{ax})	**23** (81)	95
(*R*,*R*)-**16**	9:91 (S_{ax})	**24** (88)	>99
(*R*,*R*)-**17**	4:96 (S_{ax})	**25** (91)	>99
(*R*,*R*)-**18**	17:83 (S_{ax})	**26** (79)	>99
(*R*,*R*)-**19**	6:94 (S_{ax})	**27** (84)	>99
(*R*,*R*)-**20**	21:79 (S_{ax})	**28** (66)	>99

DOI: 10.1021/ol060484p