

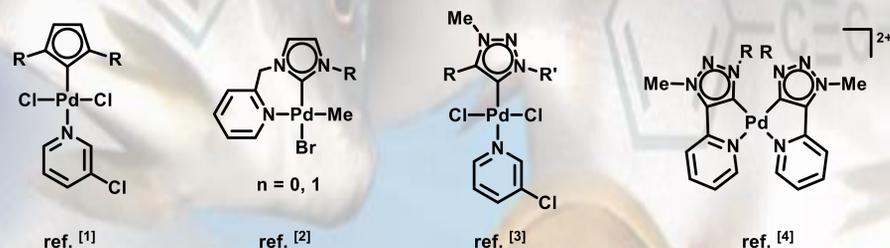
SINTEZA NOVIH MEZOIONSКИH PALADIJEVIH(II) KOMPLEKSOV IN APLIKACIJA ZA KATALIZO SONOGASHIRA REAKCIJE V VODNEM MEDIJU

Miha Virant, Martin Gazvoda, Andrej Pevec and Janez Košmrlj

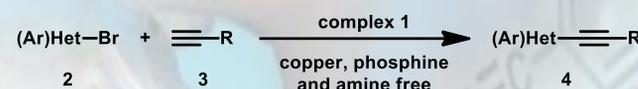


Faculty of Chemistry and Chemical Technology, University of Ljubljana,
Večna pot 113, SI-1000 Ljubljana, Slovenia, e-mail: miha.virant@fkkt.uni-lj.si

Representative examples of evolution of the Pd-NHC complex series:



Copper-free Sonogashira coupling under green reaction conditions:

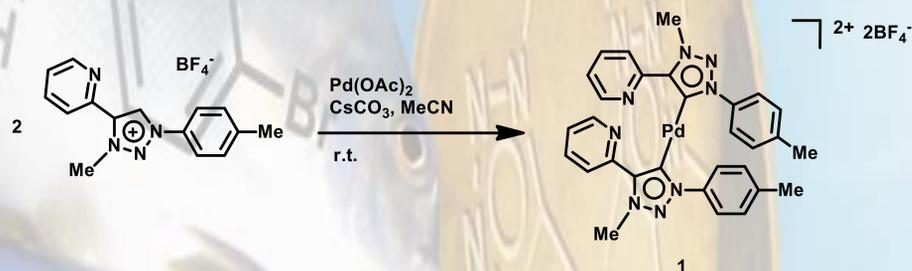


Substrate scope screening:

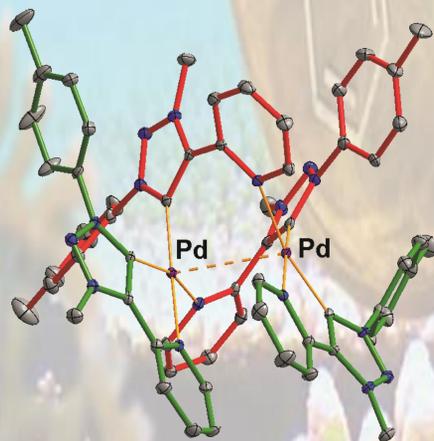
entry	2	3	cond. ^a	4	conv. ^b (yield) ^c
1	2a	3a	A ^d		100
2		3a	B		82 (75)
3	2b	3a	A		100
4		3a	B		100 (92)
5	2c	3a	B		68 (65)
6		3a	A ^d		100 (91)
7	2d	3a	B ^d		36
8	2e	3a	B		100 (95)
9	2f	3a	A		60 (58)
10		3a	A ^d		100 (86)
11	2g	3a	B		100 (97)
12	2h	3a	B		66
13	2a	3b	B		100 (91)
14	2f	3c	B		100 (95)
15	2b	3d	B		100 (89)
16	2i	3e	A		90 (87)

^a Conditions A: bromide 2 (0.25 mmol), acetylene 3 (0.5 mmol), Cs₂CO₃ (0.5 mmol), complex 1 (1 mol%), water (2 mL), 100 °C in an ACE tube, 1 h. Conditions B: as for conditions A, but with K₂CO₃ (0.5 mmol) as a base, at 140 °C, 4 h. ^b Conversion determined from ¹H NMR for at least two consecutive runs. ^c Percent yield of the isolated pure product. ^d DMF/H₂O (2/1) as the reaction solvent.

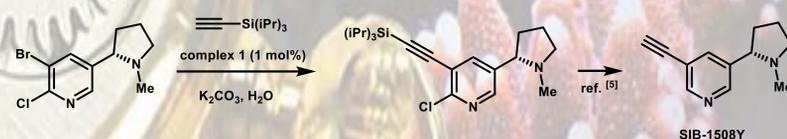
Synthesis of the complex:



X-Ray diffraction analysis model of a solid-state crystal



Application for the synthesis of Altinicline[®] precursor:



References:

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