

Supporting Information

Pd-imidate complexes as recyclable catalysts for the synthesis of C5-alkenylated pyrimidine nucleosides via Heck cross-coupling

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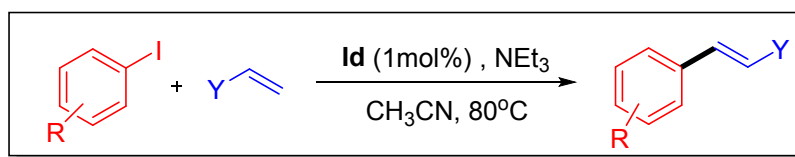
Fax: 00912233611020; E-mail: ar.kapdi@ictmumbai.edu.in

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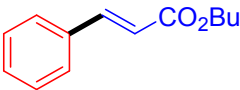
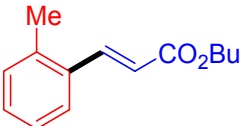
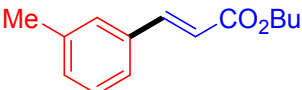
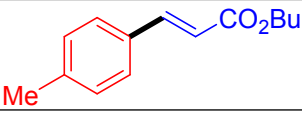
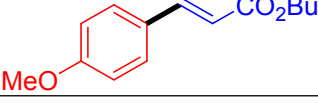
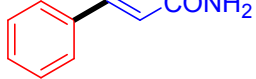
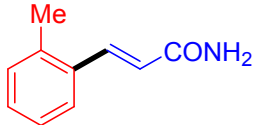
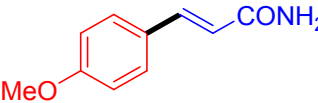
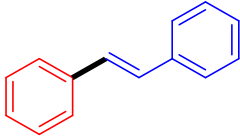
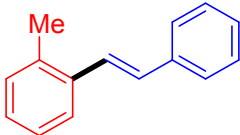
- ¹H and ¹³C NMR for unknown molecules 3c and 3i

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Table 1: Heck alkenylation of aryl iodides with alkenic nucleophiles.



Y = Ph, CO₂Bu, CONH₂

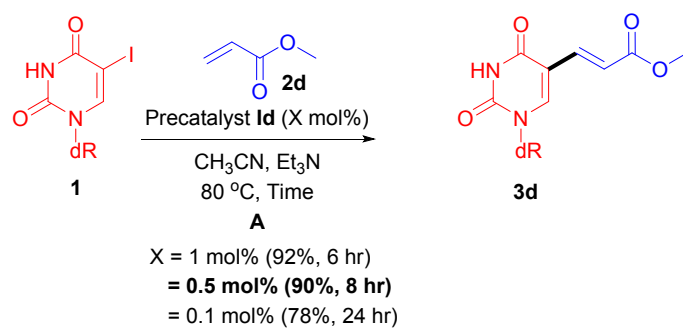
Entry	R	Y	Time/hr	Product	% Yield	Ref.
1.	H	CO ₂ Bu	15		85	1
2.	2-Me	CO ₂ Bu	24		68	1
3.	3-Me	CO ₂ Bu	18		85	1
4.	4-Me	CO ₂ Bu	24		72	1
5.	4-Meo	CO ₂ Bu	24		75	1
6.	H	CONH ₂	24		65	2
7.	2-Me	CONH ₂	24		62	2
8.	4-MeO	CONH ₂	24		64	3
9.	H	Ph	18		88	2
10.	2-Me	Ph	20		78	4

- 1) T. Tu, J. Malineni, K. H. Dęta, *Adv. Synth. Catal.*, 2008, **350**, 1791.
- 2) G. An, X. Ji, J. Han, Y. Pan, *Synth. Commun.*, 2011, **41**, 1464.
- 3) R. Imashiro, M. Seki, *J. Org. Chem.*, 2004, **69**, 4216.
- 4) L. Wang, H. Li, P. Li, *Tetrahedron*, 2009, **65**, 364.

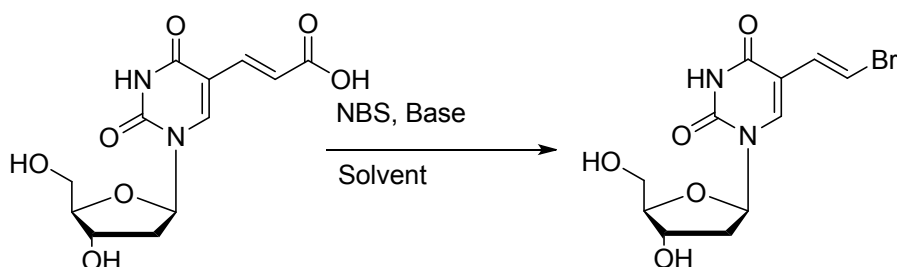
General procedure for Heck Alkenylation of aryl iodides: A solution of precatalyst **Id** (1.0 mol%) in dry CH₃CN (1.0 mL) was stirred for 5 min at ambient temperature under N₂. Then, aryl iodide (0.5 mmol) was added and the solution stirred for 5 min at 80 °C. Thereafter, Et₃N (1.0 mmol) and alkene substrate (0.5 mmol) were also added with CH₃CN (1.0 mL). The resulting solution was then stirred at 80 °C for required amount of time. After the completion of reaction the solvent was removed under vacuo and the resultant residue obtained was purified using column chromatography in Pet ether: EtOAc solvent system (96:4) to afford the desired product.

Optimisation studies for BVDU synthesis:

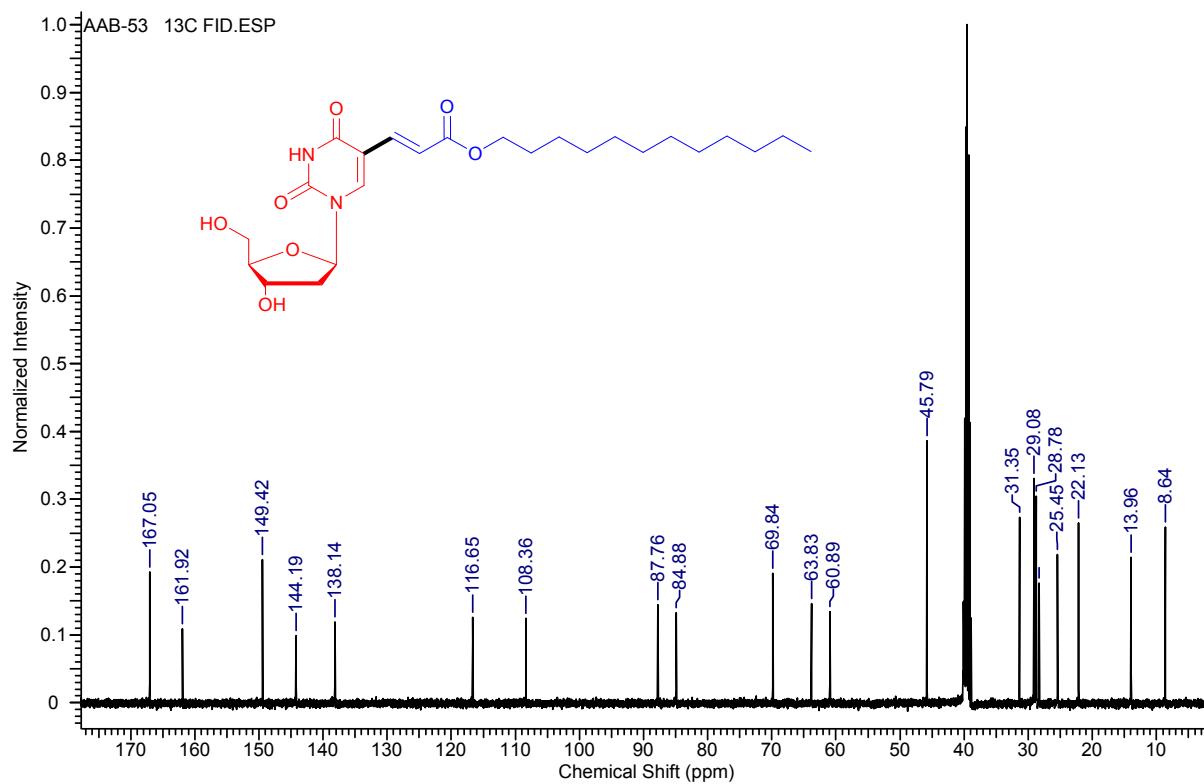
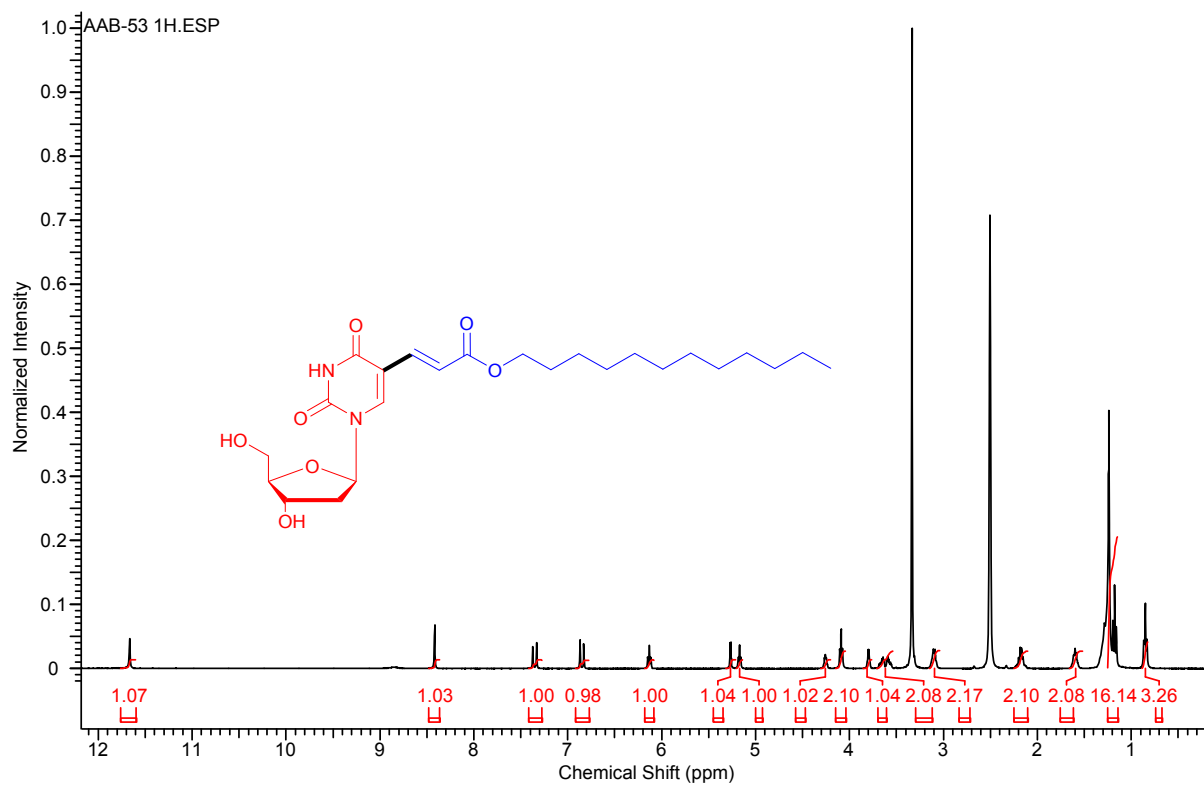
Step 1: Catalyst Loading



Step 3:



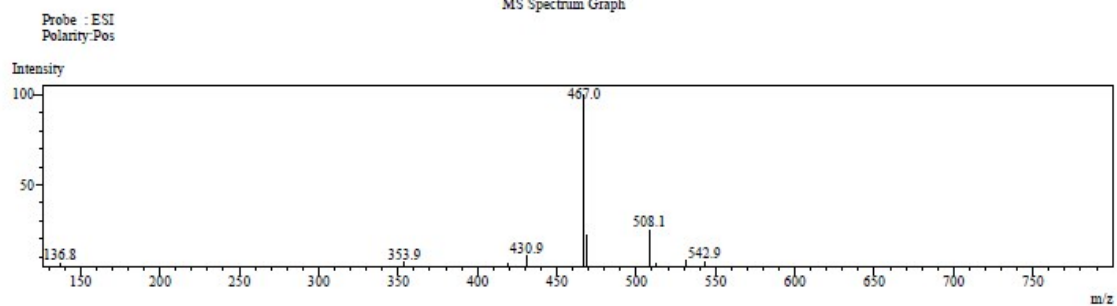
Sr.No.	CVU (eq.)	NBS(eq.)	Base(eq.)	Solvent(ml)	Temp.	Time(min.)	% Yield
1	1	2	NaOAc(2)	CH ₃ CN(3 ml)	R.T.	10	40
2	1	2	5 mol% NEt ₃	CHCl ₃	R.T.	30	30
3	1	1	-----	THF:H ₂ O,3:2(5ml)	R.T.	30	35
4 ^a	1	1.5	-----	THF:H ₂ O,3:2(5ml)	R.T.	30	30
5 ^a	1	2	-----	CH ₃ CN (3ml)	R.T.	30	25
6 ^a	1	1		H ₂ O	R.T.	30	30
7	1	2	K ₂ CO ₃ (2)	THF:H ₂ O,3:2, (5ml)	0-5°C	40	55
8	1	1	K ₂ CO ₃ (2)	DMF	R.T.	30	89

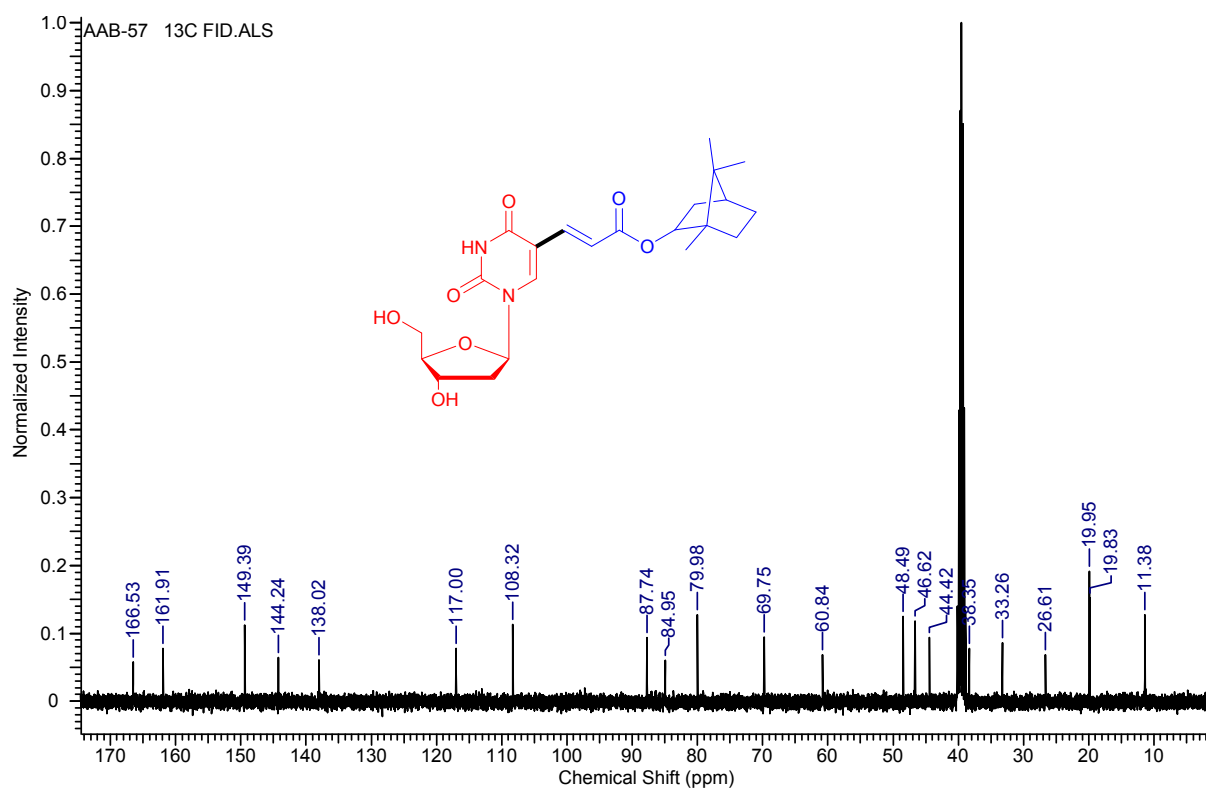
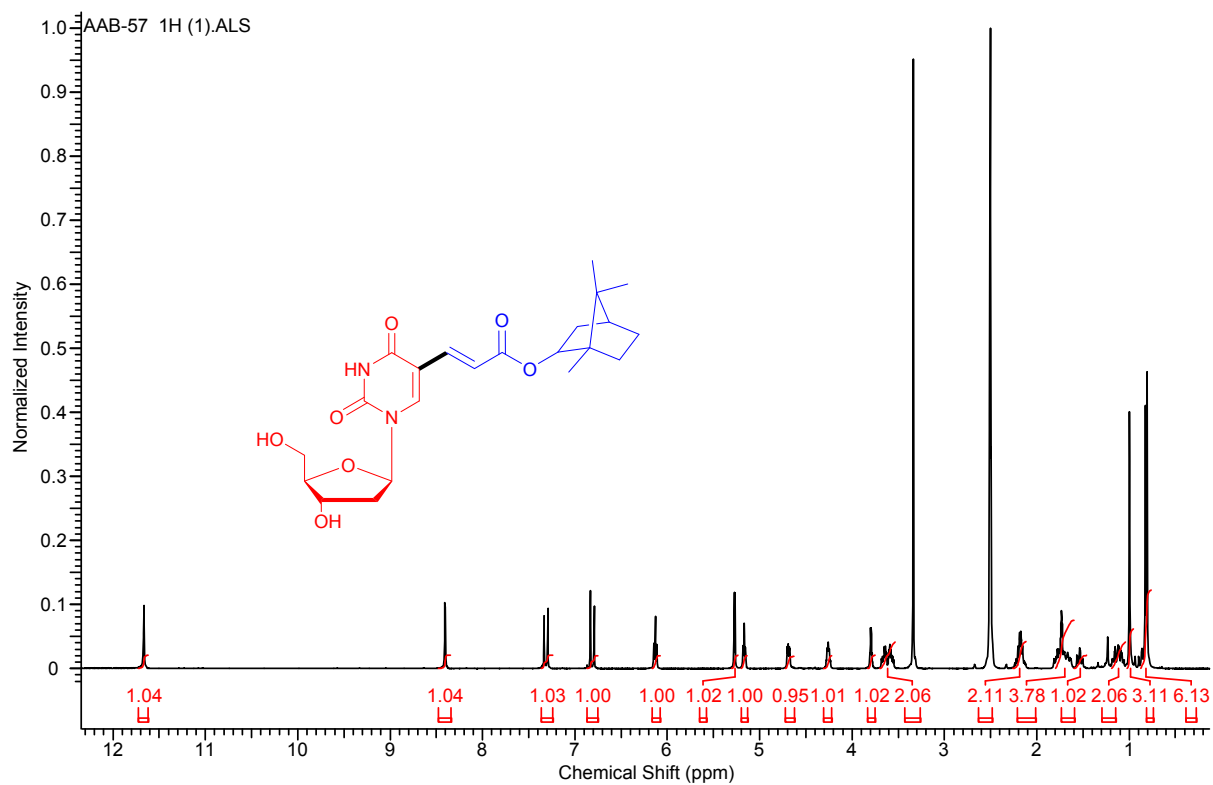


SAPALA ORGANICS PVT LTD

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Date Acquired : 6/16/2014 18:01:51

MS Spectrum Graph





SAPALA ORGANICS PVT LTD

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MS Spectrum Graph

