AEI-047: Synthesis of 2,6-Bis(P,P-di(tertbutyl)phosphinomethyl)pyridine

Date: 2022-11-30
Tags: PNP AEI P(tBu)N(py)P(tBu)
Status: Done
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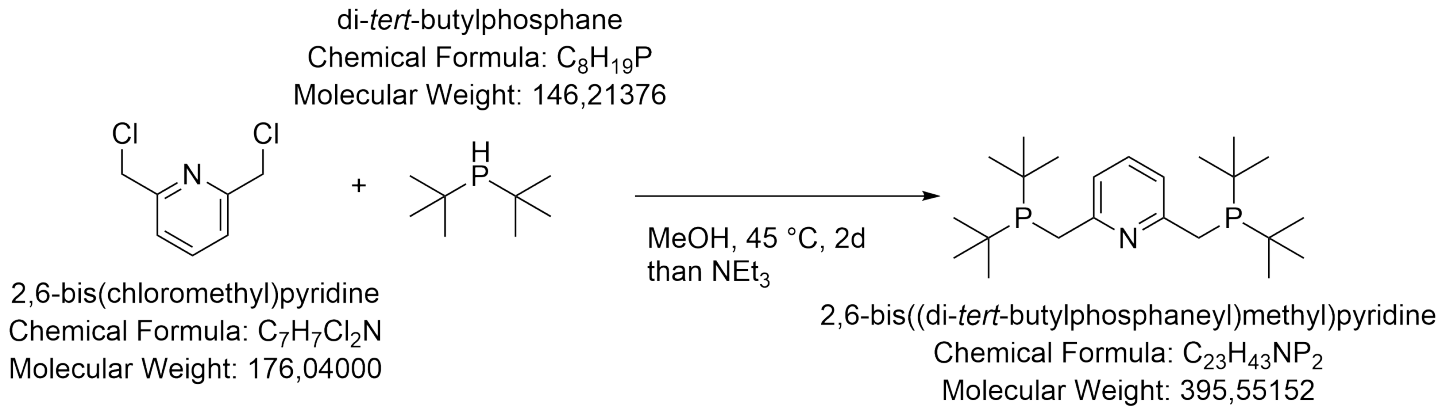
Reaction scheme/sample structure



Reagents

Name	Amount [mmol]	Equivalents	$Mass_{theo}$ [g]	$Mass_{exp}$ [g]	Molar mass [g/mol]	$Volume_{exp}$ [mL]	$Volume_{theo}$ [mL]	Density [g/mL]
2,6-bis(chloromethyl)pyridine	3.2	1	0.568	0.5695	176.04	/	/	/
MeOH	/	/	/	/	/	2.4	/	/
di-(tertbutyl)phosphine	6.88	2.15	1.0000	1.0022	146.21	/	/	/
triethylamine	7.21	2.25	0.730	/	101.2	1.0	1.0	0.73

Procedure/observations

All steps (unless stated otherwise) were carried out under argon using standard Schlenk technique.

Synthesis similar to https://pubs.acs.org/doi/10.1021/om800425p and https://pubs.acs.org/doi/10.1021/om010719v

Date	Time	Step	Observations
29.11	13:00	A schlenk flask (10 mL) and a 25 mL-Schlenk flask set under Argon.	
	15:20	In the big glovebox tBu2PH was weighed in the 10 mL flask	clear liquid.
	16:20	2,6-bis(chloromethyl)pyridine was weigehd in air to the 25 mL flask and the atmosphere was changed to argon	2,6-bis(chloromethyl)pyridine white solid

	16:40	MeOH (2.4 mL) were added to the 10 mL flask	
	16:50	The tBu2PH solution was added to the 25 mL flask	
	16:55 - 17:00	The reaction mixture was stirred at room temperature.	After a few minutes everything dissolved.
	17:00 -	The reaction mixture was heated to 45 °C	30.11, 10:00: turbid solution. The heating was turned off between 9:40 and 10:10 01.12, 9:00: more turbid solution
02.12	- 9:10	The heating was turned of and the reaction mixture was cooled down to rt	
	9:49 - 9:52	NEt3 was added dropwise	Formation of white solid. Temperature increased about 3 °C. (measured with IR-thermometer against rt), see after reaction.jpg
	10:00 - 10:40	The obtained mixture was dried under reduced pressure	White solid. raw product.jpg remainder: clean colling trap good, to avoid the smell of phosphines in the lab
	10:45	Toluene (15 mL) was added to the solid	Part of the solid dissolved
	10:45 - 10:55	The mixture was stirred	
	10:55 - 11:20	The mixture was allowed to settel	
	11:20 - 11:45	The mixture was filtered according to [Protocol] Cannula Filtration/Transfer	
	11:45 - 12:05	The residue was washed with toluene (3 * 1 mL)	
	13:00 - 13:40	The solution was dried under reduced pressure	white solid
	13:50 -13:55	Et2O (15 mL) was added to the solid	some white residue remained
	13:55 -14:05	The mixture was allowed to settle	
	14:05 -14:30	The mixture was filtered according to [Protocol] Cannula Filtration/Transfer and washed with Et2O (3 * 1 mL)	clear solution and white solid, some solids formed in the filtrate, most likely due to partiall evaporation of Et2O solid.jpg
	14:35 - 14:50	The solvents were removed under reduced pressure	During the evaporation needles formed, which were redissolved
05.12	10:20 - 11:50	The white solid was dried under reduced pressure	

	13:00	A NMR sample was prepared in DCM-d2 according to [Protocol] Preparation of NMR Sample	

Analysis

Date	Time	Sample name	Analysis method	Solvent	File name	Interpretation
05.12	13:33	AEI-047	1H (-30 - 20 ppm), 31P	DCM-d2	AEI-047_10	All signals as expected in 1H and 31P. In 1H only 1 relevent impurity with approx. 6 % can be seen at 4.58 ppm. In the 31 P one mayor species with 96.5 % is observed at 35.5 ppm. Main impurity with with 2 % at 36.5 ppm. Minor impurities: 58 and 64 ppm each less than 1 %

Product characterization

		m [mg]	purity	m_{pure} [mg]	n [mmol]	Y [%]
AEI-047	fine white powder	954.18	96.5 %	920,78	2.3278	72.7

Linked resources

- Protocol - Preparation of NMR Sample
- Protocol - Cannula Filtration/Transfer
- Protocol - Schlenk Technique

Attached files

AEI-047_10-1.nmrium (for MA)
sha256: 99d85a2754bcaad47b18812b9aba0b3fc3a476c0a4ee14c9b0244b1d3c7724fb

AEI-047.zip
sha256: 6d419f47ed57754d91a0f65a9be5ced6932fa10a021e1ad858474ecc09332ab0

AEI-047_10.nmrium
sha256: f8eec5785f6a107957a6511f6dbcc4c1f49d2af1c7c8cf5850bb276229eaa592

solid.jpg
sha256: 164595d3e9693e6c04f1621c333313f8aecae5f808d0db16abd93dad0f9fac99

raw-product.jpg
sha256: 835015e89591c7caca2c9f1fc4905dacff3559c60bd4473aec00cd63644d38f2

after-reaction.jpg
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AEI-047.png
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AEI-047.png
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AEI-047.cdxml
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Unique eLabID: 20221130-652e80a4501d4b63310207b7c5f12fa5222f94eb
Link: https://elab.water-splitting.org/experiments.php?mode=view&id=292