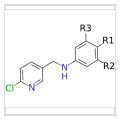

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Synthesis of fluorinated neonicotinoids

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1 Works for me dx.doi.org/10.17504/protocols.io.9h5h386

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ABSTRACT

This is a general protocol for synthesis of fluorinated neonicotinoid analogues by interaction of amines with 2-chloro-5-(chloromethyl)pyridine.

EXTERNAL LINK

https://doi.org/10.1371/journal.pone.0227811

THIS PROTOCOL ACCOMPANIES THE FOLLOWING PUBLICATION

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MATERIALS

NAME	CATALOG #	VENDOR
2-chloro-5-(chloromethyl)pyridine	516910	
anhydrous potassium carbonate	60109	
anhydrous acetonitrile	271004	
Dichloromethane	320269	Sigma Aldrich
Silica gel	645524	Sigma Aldrich
Ethyl acetate	319902	Sigma Aldrich
Hexane	208752	Sigma Aldrich

MATERIALS TEXT

Substituted aniline, obtained according to literature sources.

1 Dissolve **2 mmol** of corresponding substituted aniline in **5 ml** of anhydrous acetonitrile in round-bottom flask, add **0.324 g** (**1.9 mmol**) of 2-chloro-5-(chloromethyl)pyridine and **0.828 g** (**6 mmol**) of anhydrous potassium carbonate.

2 Put a water-cooled backflow condenser on top of the flask and heat the flask under vigorous stirring until the solution starts to boil.

3 Continue refluxing for several hours. Monitor reaction progress via thin layer chromatography (TLC).

4 After the reaction is complete, filter off inorganic salts and rigorously wash the sediment with dichloromethane.

5 Combine all liquid fraction and remove the solvents by evaporation under reduced pressure.

6 Purify the residue by preparative (TLC) on SiO_2 (eluent EtOAc/hexane 1:2).