C2-STS-51

Date: 2018-06-14
Tags: *C2-STS-51*
Created by: Sebastian Steiner

Synthesis of Rufinamide on Rig 2

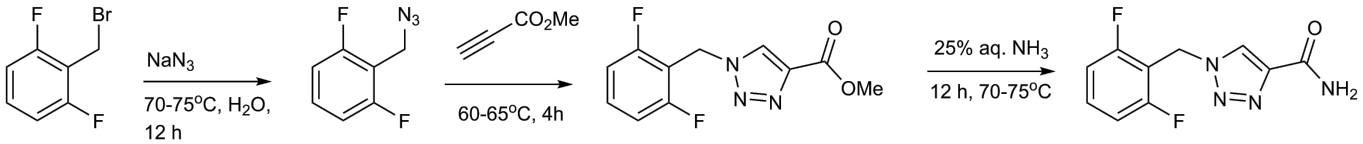
Experiment code: C2-STS-51

Date: 14/06/2018

Code repo and commit: Chempiler [f2a06e2]

ATTENTION! Attach initial GraphML and ChASM file separately!

Reaction scheme



Experimental

The reactor flask was charged manually with 2,6-difluorobenzylbromide (7.5 G. 36 mmol) and

solution of sodium azide was (2.54 G, 39 mmol) in water (75 mL) was automatically added.

The reaction mixture was stirred in reactor (400 rpm) and heated at 75 oC for 12 h.

The progress of the reaction can be monitored by TLC, eluent hexene. One can observe dissapearnace

of the starting material. After the 12 h the reaction mixture was cooled to room temperature,

and transferred to the fitration module. Then methyl propiolate was added (3.21 mL, 36 mmol) to the filtration module.

The reaction mixture was stirred at 400 rpm and heated at 60-65oC using chiller.

After 4h the reaction mixture was cooled to room temperature, and aq. soln. of 25 % ammonia was added (60 mL),

C2-STS-51

Date: 2018-06-14
Tags: *C2-STS-51*
Created by: Sebastian Steiner

and the reaction mixture was stirred at 75oC with stirring. During this time, the product precipitate as white solid.

The reaction mixture was cooled to room temperature and and the product was filtred off, washed with water, and dried.

Giving the Rufinamide as off-white solid (X.XX G, % yield).

Reagents:

[reagent name], [purity as stated], [vendor and container], [Lot number], [opened (date)]

2,6-Difluorobenzyl bromide, 97%, Sigma-Aldrich, 25g glass bottle, Lot #BCBR7827V, opened 22/05/18
Methyl propiolate
Sodium azide
25 % Ammonia

Automation notes:

ChASM file as provided by Jarek is run on Rig 2 without changes.

Calculations

Reagent	Mass [g]	Volume [mL]	Amount [mol]	Equivalvents	Molar mass [g/mol]	Density [g/mL]
2,6-Difluorobenzyl bromide	7.45		0.036	1	207.02	
Sodium azide	2.54		0.039	1.1	65.00	
Methyl propiolate	3.02	3.20	0.036	1	84.07	0.945
Rufinamide	8.575 (theor.)		0.036	1	238.194	

Result

???

C2-STS-51

Date: 2018-06-14
Tags: *C2-STS-51*
Created by: Sebastian Steiner

Comments:

On 2018-06-14 12:06:18 Sebastian Steiner wrote :
Sodium azide: 10.12 g in 240 mL of water. Because reasons.

On 2018-06-19 17:22:45 Sebastian Steiner wrote :
m(NaN3) = 10.1297g

On 2018-06-19 18:36:55 Sebastian Steiner wrote :
m(difluorobenzylbromide) = 7.454g

On 2018-06-21 10:51:27 Sebastian Steiner wrote :
Automated sequence proceeded uneventfully.

On 2018-06-21 13:00:27 Sebastian Steiner wrote :
m(rufinamide) = 3.7617g (44%) which is within one standard deviation. Done!

elabid : 20180614-9c5e3a73349b68c5fe7c5c96e9d51ae6646c97b5
link : https://datalore.chem.gla.ac.uk:88/experiments.php?mode=view&id=258