



Date/Initials

Experiment

01/05/2016 Samples are analyzed according to following JPL UPLC method:

ANALYTICAL INFORMATION

UPLC method

Column: Waters Acquity UPLC HSS C18, 2.1x100 mm, 1.8 µm

Eluent: 0.2% TFA in Water and 50:50; ACN:MeOH

Injection volume: 3.5 µl

Flow: 0.4 ml/min

Run time: 16 min

Internal standard: Saccharine (measured at 250nm)

Solvents

A 1: 0.2% TFA in Water

B 1: 50:50; ACN:MeOH

Binary Pump Program

Time (min)	Solvent A (%)	Solvent B (%)	Flow (ml/min)
Initial	98.0	2.0	0.40
4	90.0	10.0	0.40
10	85.0	15.0	0.40
12	44.0	56.0	0.40
13	2.0	98.0	0.40
14	2.0	98.0	0.40
15	98.0	2.0	0.40
16	98.0	2.0	0.40

Autosampler

Injection mode: Partial Loop with needle overflow

Weak Solvent wash 90/10 Water/MeOH

Strong Solvent wash 10/90 Water/MeOH

Column Temperature: 50°C

UV detector

Channel 1: 270 nm – Bandwidth 1.2 nm – detection of furanics

Channel 2: 250 nm – Bandwidth 1.2 nm – detection of standard saccharine

Collect 3D data

- Range 190 to 400 nm step 1.2 nm

Process and export results of sequence
 ibosay_Am1630_RUNAL.DIK
 Use processing method "Processing Monomer Oxidation"
 Export results into excel file "2016-Q2-Monomer UPLC data-area" in the folder PLYXYAnalytics\Analytics\Processed data
 Report to JPL

01/05/2016

JPL

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Experiment 3

12 MAY 16
DVH

Introduction

follow up experiments on Experiment 1 page 1

Equipment

QCS ϕ 4 (LR- ϕ 43)
QCS block 2 ϕ & 3 ϕ

Experimental procedure

12 MAY 16
DVH

Calibration for project AM1630 T: 145, 160 and 180 oc
QCS (ID)
NAME/Date
Reactor content 1.5 mL Acetic Acid 20 bar Air/N₂
Stirring rate=750rpm

chemicals	supplier	Lot No	CAS No	Avantum Nr	MW	purity
HMF	Epochen	22179013	67-47-8	0241	126.11	99%
AMF	Aldrich	1051583	10551-58-3	0055	168.15	99%
DMSO	Biosolve	1051583	67-68-3	UR-4		99.00%
Co(OAc) ₂ ·4H ₂ O	Sigma-Aldrich	54057115	8147-53-1	5079	249.08	
Mn(OAc) ₂ ·4H ₂ O	Aldrich	420400039	6156-78-1	0056	245.00	99%
NaBr	Sigma	1051583	7647-15-0	5432	102.89	
Acetic Acid	Biosolve	1051583	64-10-2	0031	00.05	99.00%

SOLUTIONS	needed (g)	HMF	actual	needed (g)	AMF	actual	Acetic Acid (mL)	needed	actual
A	2.5	5.0005	0	2.5	2.65378	25	50		
B	1.5	2.94856	1.333	2.000	3.95863	25	50		
C	1	1.86407							

Co(OAc) ₂ ·4H ₂ O	needed (mg)	actual	Mn(OAc) ₂ ·4H ₂ O	needed (mg)	actual	NaBr	needed (g)	actual	AcOH	ml to mark
CA1	228.943	266.64	223.012	202.37	225.0715	187.243	200.20	187.612	85	100

conditions: Air 20 bar, T = 315 °C, 1 hr reaction time

QCS (ID)	amount (ml)	cat (ml)	pressure	solution actual	cat actual
AM1630 R1-13	A	0.5	1	20	4349.7
AM1630 R1-14	B	0.5	1	20	4499.9
AM1630 R1-15	C	0.5	1	20	4830.0
AM1630 R1-16	C	0.5	1	20	4364.2
AM1630 R1-17	B	0.5	1	20	4255.0
AM1630 R1-18	C	0.5	1	20	4319.5
AM1630 R1-19	A	0.5	1	20	4537.8
AM1630 R1-20	A	0.5	1	20	4259.8
AM1630 R1-21	C	0.5	1	20	4305.2
AM1630 R1-22	A	0.5	1	20	4350.9
AM1630 R1-23	B	0.5	1	20	434.86
AM1630 R1-24	B	0.5	1	20	4365.7

A	B	C	C
B	C	A	A
C	A	B	B

reactor	1	2	3	4
B	5	6	7	8
C	9	10	11	12

clean blocks with sleeves
add stirrer and weigh blocks
close and pressurize
place in pre-heated block according to settings
fill in log book
place immediately after reaction time (1hr) in ice (30 minutes)
decompress and open in fumehood
if possible weigh reactors
make ES 70 mg/ml solution (350 ml)
add sacharine stock solution and stir until dissolved
take solutions and add them to sample vials containing H₂O

0 mg/mL
step 1: add 5 mL ES
step 2: take 10 μ L and add to 3 mL water

24.5 gram
24500 mg sacharine in 350 mL (to mark 350)

calculations:

sample work up:	1.5 mL	step 1: Add ES	new concentrations:	mg/mL	step 2	add to:	final concentration
reactor content:	70 mg/mL	5	sacharine	53.84615385	use	H ₂ O (ml)	0.178890877
ES concentration:	70 mg/mL		luka	9.520892052	0.01	3	0.031631203
estimated feraricins in res:	61.88644834 mg						

recipe:
step 1: Add ES (5 mL) directly to the reactor
ES amount: 70 mg/mL, 5 mL, 35 g, 500 mL, 35.1365 gram, 70.273 mg/mL
step 2: stir until all is dissolved
step 3: dilute
take 10 μ L of solution
add to 3 mL of water in a vial
step 4: take 1 mL of the resulting solution and submit for UPLC analysis

before exp (mg)
after exp (mg)

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