



Date/Initials

Experiment 1

12 MAY 16

DVK

12 MAY 16
DVK

Calibration for project AM1630 T: 145, 160 and 190 oc
 QCS (ID)
 NAME/Date
 Reactor content 1.5 mL Acetic Acid 20 bar A1/N2
 Stirring rate=750rpm

chemicals	supplier	Lot No	CAS No	Avantium No	MW	purity
HMF	Epochm	20111800	97-97-0		8311	129.11 99%
AMF	Aldrich	57001000V	10551-88-3		6886	105.15 99%
DMSO	Biosolve	0001103	67-26-5		58.12	99.00%
Co(OAc) ₂ ·4H ₂ O	Sigma Aldrich	AR05271-11V	6147-53-1		90.04	249.08
Mn(OAc) ₂ ·4H ₂ O	Aldrich	AKAR000033V	6150-78-1		6886	245.09 99%
NaBr	Sigma	HT001000V	7647-15-6		109.92	102.89
Acetic Acid	Biosolve	0072381	64-19-7		58.12	60.05 99.80%

SOLUTIONS	needed (g)	HMF actual	needed (g)	AMF actual	Acetic Acid (wt. %)	needed	actual
A	2.5	6.09065	0	0	25	25	50
B	1.5	2.04856	1.333	1.333	25	25	50
C	1	1.96407	2.000	2.000	25	25	50

	Co(AcO) ₂ ·4H ₂ O	Mn(AcO) ₂ ·4H ₂ O	NaBr	AcOH
needed (mg)	226.643	223.012	187.243	187.612
actual	206.64	205.79	204.78	220.29
needed (g)	0.20664	0.20579	0.20478	0.22029
actual	0.20664	0.20579	0.20478	0.22029

conditions: Air 20 bar, T = 155 oc, 1.5 hr reaction time	QCS (ID)	amount (ml)	Cat (ml)	pressure	empty (mg)	Solution (mg)	Cat (mg)
AM1633 R1-13	A	0.5	1	20	43016	533.8	1048.09
AM1633 R1-14	B	0.5	1	20	44209.6	533.34	1049.32
AM1633 R1-15	C	0.5	1	20	43499.4	534.32	1048.03
AM1633 R1-16	C	0.5	1	20	43262	534.14	1047.92
AM1633 R1-17	B	0.5	1	20	43064	533.67	1046.79
AM1633 R1-18	C	0.5	1	20	42636.7	534.37	1048.2
AM1633 R1-19	A	0.5	1	20	43184.9	534.16	1048.36
AM1633 R1-20	A	0.5	1	20	43368.4	534.09	1047.89
AM1633 R1-21	C	0.5	1	20	43675.9	536.99	1048.07
AM1633 R1-22	A	0.5	1	20	43595	534.56	1048.21
AM1633 R1-23	B	0.5	1	20	43467.8	534.66	1048.05
AM1633 R1-24	B	0.5	1	20	43358	533.54	1048.19

QCS block: location as last time please! -->

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

reactors	1	2	3
	9	6	7
	9	10	11

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 (>50 mL)
 add sacharine stock solution and stir until dissolved
 take solutions and add them to sample vials containing H2O

24.5 gram
 24500 mg sacharine in 350 mL (to mark 350)
 u mg/mL
 step 1: add 5 mL ES
 step 2: take 10uL and add to 3 mL water

calculations:	sample work up:	reactor content:	ES concentration:	estimated furanics in re:	step 1: Add ES	new concentrations:	mg/mL	Step 2	add to:	final concentration
	1.5 mL	70 mg/mL	61.88444834 mg	5	sacharin	53.64615385	0.01	3	0.178890677	
					folca	9.520992052			0.031611203	

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 uL 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

12 MAY 16

DVK

12 MAY 16
DVK

