



AVANTIUM

PERSONAL LABORATORY LOGBOOK

Assigned to

Andre' van de Beek

Logbooknumber

ABE-P89

Date of issue

23-Mar-09

Exhibit 2017

Date/Initials *OxE-073* Experiment
08 Jun 09
ABE Experiment: OxE-073 *12 May09* ABE



The objective of this experiment is to study the stability of the final compounds (FDCAR) in the oxidation of MMF.

Main objective: **Stability of products**

Table 1: Chemicals

Name	HMF	MMF	MeOH
Supplier			
Cas nr			
Lot nr			
Mw (g/mol)			
info			
Name	Saccharine	AcOH	
Supplier	Aldrich		
Cas nr	81-07-2		
Lot nr	S37371-017		
Mw (g/mol)	183.19		
info	98+%		

Table 2: Apparatus

Apparatus	HPLC06
Brand / type	Agilent
Guard Column	none
Column	Sunfire C18, 4.6*10 cm, 3.5 um, part no. 186002553
Flow (ml/min)	1.0
Oven temp (°C)	40.0
Mobile phase	Gradient: 0.2% TFA with MeOH : ACN (50:50)
Method name analyzing	080313-Oxe-gradient-35min.met
Apparatus	HTHP QCS
Stirring speed (rpm)	750
QCS Heating T (°C)	247 (results in 220 °C inside the reactor)
Huber T (°C)	175

Table 3: Catalysts

Cat	Cas nr.	Avantium nr.	MW	Purity
Co(OAc) ₂ ·4H ₂ O	6147-53-1	N7019	249.08	98-102
Mn(OAc) ₂ ·4H ₂ O	6156-78-1	105A	245.09	99+
NaBr	7647-15-6	143	102.89	99+

Experimental REMARK:

Date weighing, dissolving and adding Cat and substrate : *12 may 09*
 Date starting reaction of experiment : "
 Date weighing, dissolving and adding Saccharine stock solution : "
 Date of analyzing with the HPLC : *13 may 09* *his Paul*
 HPLC-analyzing according HPLC-6 logboek :

Experimental

Load blocks according table1 till 16. Weighing tolerance **± 0.3 mg**.

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Date/Initials OxE-073 **Experiment**

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Add stirring magnet.
Close with teflon/rubber cap.
Bring under pressure with Air till 60 Bar at RT
Placed in preheated HTHP QCS of 180 °C, according settings in table 2, during 1 hours. } 12 May 09 by ABE
Placed immediately after the reaction time in an ice bath during 35 minutes.
Open and thereby decompress reactors.
Add 1.0 ml of DMSO. } 45 for 20 minute
Add 5.0 ml of Saccharine stock solution, table 5.
These dilutions where stirred for 10 minutes.
These dilutions to 7 times were transferred to 10 ml vials. } stirring a hole night.
Dilutions to 700 times were made by 10 µl of dilutions 8 times + 990 µl of **Water** in HPLC vials. } read bis C.M.N

Table 4: Substrate MMF solutions, in table 11 to 12 called A, and B

Sol A (111.12 mg/ml)	Amount	Sol B (222.24 mg/ml)	Amount
MMF [g]		MMF [g]	
V AcOH [ml]		V AcOH [ml]	
Total V [ml]		Total V [ml]	
Concentration (mg/ml)		Concentration (mg/ml)	
Date		Date	
Samples prepared with this solution.		Samples prepared with this solution.	

Table 5: Saccharine stock solution (11.04 mg/ml)

	amount
m Saccharine [g]	
V MeOH [ml]	
Total V [ml]	
Concentration (mg/ml)	
Date	
Samples prepared with this solution.	

same as
OxE-065

Co/Mn/Br-15 rollat. from see OxE-69. Hg. 65

Table 11: Block 1 /Temp = 220 °C / P= 60 bar (Air) / T₀= 1427 Time 1 hours

R Nr.	Substrate name	Substrate	Cat Code	V _{AcOH} [ml]	V _{Cat} [ml]	Cat concentration [mol %]	Solvent	HPLC name
1	FDCA	50 499	---	1.0	---	---	AcOH	OxE073-V1
2	FDCA	50 499	---	1.0	---	---	AcOH	OxE073-V2
3	FDCA1/2Me	50 503	---	1.0	---	---	AcOH	OxE073-V3
4	FDCA1/2Me	50 499	---	1.0	---	---	AcOH	OxE073-V4
5	FDCA	50 503	Co/Mn/Br-15	---	1.0	---	AcOH	OxE073-V5
6	FDCA	50 503	Co/Mn/Br-15	---	1.0	---	AcOH	OxE073-V6
7	FDCA1/2Me	50 489	Co/Mn/Br-15	---	1.0	---	AcOH	OxE073-V7
8	FDCA1/2Me	50 503	Co/Mn/Br-15	---	1.0	---	AcOH	OxE073-V8

not done

Reviewed by ADG Date 22 June '09