



AVANTIUM

# PERSONAL LABORATORY LOGBOOK

Assigned to

***Andre' van de Beek***

Logbooknumber

***ABE-P89***

Date of issue

***23-Mar-09***

Exhibit 2010

**Date/Initials** OxE-081 - Block 5 **Experiment**

10sep09  
ABF

**Experimental**  
if done make X

- Load blocks according table 12 till 16. Weighing tolerance  $\pm 0.3$  mg.
- Add stirring magnet and cap with Avantium disk 11352955. These caps leaked so old ones "Big" used.
- Close the blocks according OP-CH-100 Manual HPHQCS.
- Bring under pressure with Air till 20 Bar at RT according OP-CH-99: Pressuriser Work procedure
- Plac in preheated HTHP QCS # LR-041 of 160 or 180 °C, according settings in table 2, during 1 or 0.5 hours
- Fill in logboeknr.: HTHPQCS-E13
- Place immediately after the reaction time in an ice bath during 30 minutes.
- Open and thereby decompress reactors in a fumehood.
- Add 5.0 ml of Saccharine stock solution, table 5 and stir for 1 hour until all the products were dissolved.
- Transfere these dilutions to 7 times to 8 ml vials with Pasteur's pipettes.
- Dilute 700 times by 10  $\mu$ l of dilutions 78 times + 990  $\mu$ l of Water in HPLC vials.

Table 4: Substrate MMF solutions, in table 11 to 26

Sol A HMF (100 mg/ml)	Amount	Sol B HMF/AMF (113.34 mg/ml)	Amount
HMF [g]	0.500	AMF [g] [53.34 mg/ml]	0.263
V AcOH [ml]	5	HMF [g] [60 mg/ml]	0.302
Total V [ml]		V AcOH [ml]	5
Concentration (mg/ml)	99.96	Total V [ml]	
Date		Concentration (mg/ml)	113.04
Samples prepared with this solution.		Date	
		Samples prepared with this solution.	
Sol C HMF/AMF (120 mg/ml)	Amount	Sol D AMF (133.33 mg/ml)	Amount
AMF [g] [80 mg/ml]	0.396	AMF [g]	0.669
HMF [g] [40 mg/ml]	0.194	V AcOH [ml]	
V AcOH [ml]	5	Total V [ml]	5
Total V [ml]	5	Concentration (mg/ml)	
Concentration (mg/ml)	118.04	Date	133.88
Date		Samples prepared with this solution.	
Samples prepared with this solution.			

Table 14: Block 5 Blocknr: /Temp = 180 °C / P = 50 bar (Air) / T<sub>0</sub> = 10.25 d.d.: 10sep09 Time 1 hours

R Nr.	Substrate name	Substrate [ml]	Cat Code	V <sub>Cat</sub> [ml]	Cat concentration [mol %]	O <sub>2</sub> /MMF [mol ratio]	HPLC name
1	DMF	0.5 ml F-2	Cat-081-01	1.0	4	2.69	OxE081-V49
2	DMF	0.5 ml F-2	Cat-081-01	1.0	4	2.69	OxE081-V50
3	DMF	0.5 ml F-2	Cat-081-02	1.0	4	2.69	OxE081-V51
4	DMF	0.5 ml F-2	Cat-081-02	1.0	4	2.69	OxE081-V52
5	DMF	0.5 ml F-2	Cat-081-03	1.0	4	2.69	OxE081-V53
6	DMF	0.5 ml F-2	Cat-081-03	1.0	4	2.69	OxE081-V54
7	DMF	0.5 ml F-2	Cat-081-04	1.0	4	2.69	OxE081-V55
8	DMF	0.5 ml F-2	Cat-081-04	1.0	4	2.69	OxE081-V56
9	5-MF	0.5 ml E-2	Cat-081-01	1.0	4	2.69	OxE081-V57
10	5-MF	0.5 ml E-2	Cat-081-01	1.0	4	2.69	OxE081-V58
11	5-MF	0.5 ml E-2	Cat-081-02	1.0	4	2.69	OxE081-V59
12	5-MF	0.5 ml E-2	Cat-081-02	1.0	4	2.69	OxE081-V60

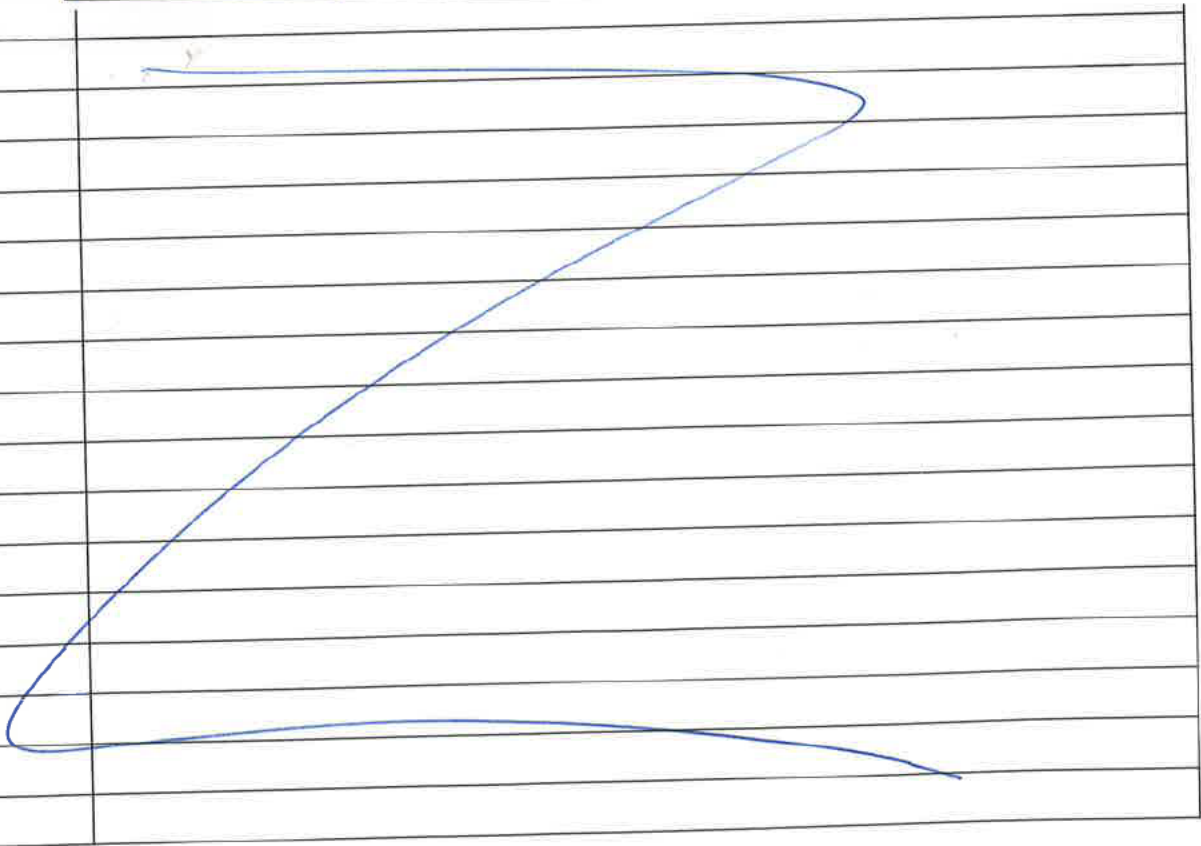
Reviewed by ADG Date 05-oct-2009

Date/Initials OxE-08-Blak S **Experiment**

10 sep 09  
ABF

Sol E 5MF (87.30 mg/ml)		Amount	Sol E 5MF (87.30 mg/ml)	
			Amount for E-1	Amount for E-2
5-MF [g]		0.430	0.4379	0.4351
V AcOH [ml]				
Total V [ml]		5	5	5
Concentration (mg/ml)		85.96	87.58	87.02
Date				09sep09
Samples prepared with this solution.			090909-OxE081-V37 till V44	090909-OxE081-V49 till V56

Sol F DMF (76.22 mg/ml)	Amount for F-1	Amount for F-2
DMF [g]	0.3814	0.3807
V AcOH [ml]		
Total V [ml]	5.0	5.0
Concentration (mg/ml)	76.28	76.14
Date		10sep09
Samples prepared with this solution.	090910-OxE081-V45 till V48	090909-OxE081-V57 till V60



Reviewed by ADG Date 05-oct-2009