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BIMODAL ETHYLEN-COPOLYMERIZATION in a CONTINUOUS 2-STAGE MINI PLANT FOR OLEFIN COPOLYMERIZATION

Questions? Please contact us!

EXAMPLE: **TOHO THC Catalyst**
Internal PRT data file #119

Ahaus, April 2017
by Günter Weickert and Eric-Jan Prinsen

SHORT INTRODUCTION

This document describes an example experiment carried out in a scaled-down reactor system. The smallest possible reactor size was targeted under the condition that product quality is the same as in industrial systems. Such product properties can be reached with this plant at significantly lower costs compared to standard continuous pilot plants.

The experiment was done with quasi-online control of

- MFR, (melt flow rate at 190°C, 2.5kg and 190°C/21.6kg)
- solid content

and

- 'blending ratio'.

Samples were taken from both reactor 1 (R1) and reactor 2 (R2). Solid content data was available about 10min after sampling, and MFR-data was available after 20min.
Pre-activated THC catalyst from TOHO TITANIUM was used.
From the first beginning, feed parameters have been set to constant values, especially

⇒ LS=15s "time constant for pumping slurry from R1 to R2"
kept constant from t=0

⇒ CAT=60s "time constant for catalyst slurry feed to R1",
kept constant from t=3.6min

⇒ GLS=100s "time constant for level control pump in R2"
kept constant from t=0

Other operation parameters are given below.

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This short report is made for experts who are familiar with catalytic olefin polymerizations. It should be understood that "continuous" polymer qualities cannot be produced in batch operations.

Catalyst storage preparation

- 1000 mg TOHO THC catalyst
- 150mL 1m TEA solution
- 1500 mL C6

Catalyst was pre-activated by mixing these components.

SAMPLING

Sample #	Time min	Polymer %	MFR(190/2.16)	Comment
S1_1	26.5	6.04		
S1_2	40	10.37		
S1_3	60	14.44	523.53	
S1_4	82	19.00	600.86	
S1_5	100	20.72	398.07	
S1_6	130	23.04	414.19	
S1_7	160	25.53	470.59	
S1_8	200	28.34	389.03	
S1_9	218	29.05	385.22	
R1	229.5	25.19	375.82	
			379.05	
				MFR(190/21.6)
S2_1	64	6.06	0.0394	1.99
S2_2	96	11.68	0.0466	2.38
				2.56
S2_3	126	17.15	0.0495	2.61
S2_4	166	21.20	0.0410	3.06
				2.58
				2.98
S2_5	203	24.14	0.1032	4.18
				4.19
S2_6	229	21.47	0.0820	3.97
				3.83
R2	229.5	25.00	0.1093	3.64
			0.0900	3.57
			0.0970	3.94

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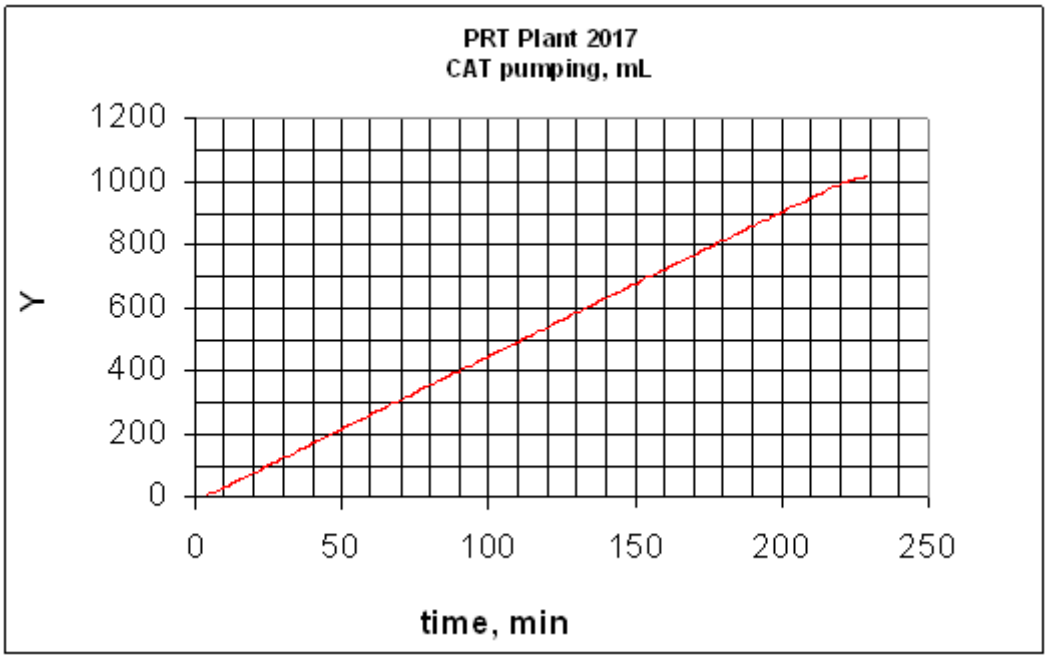
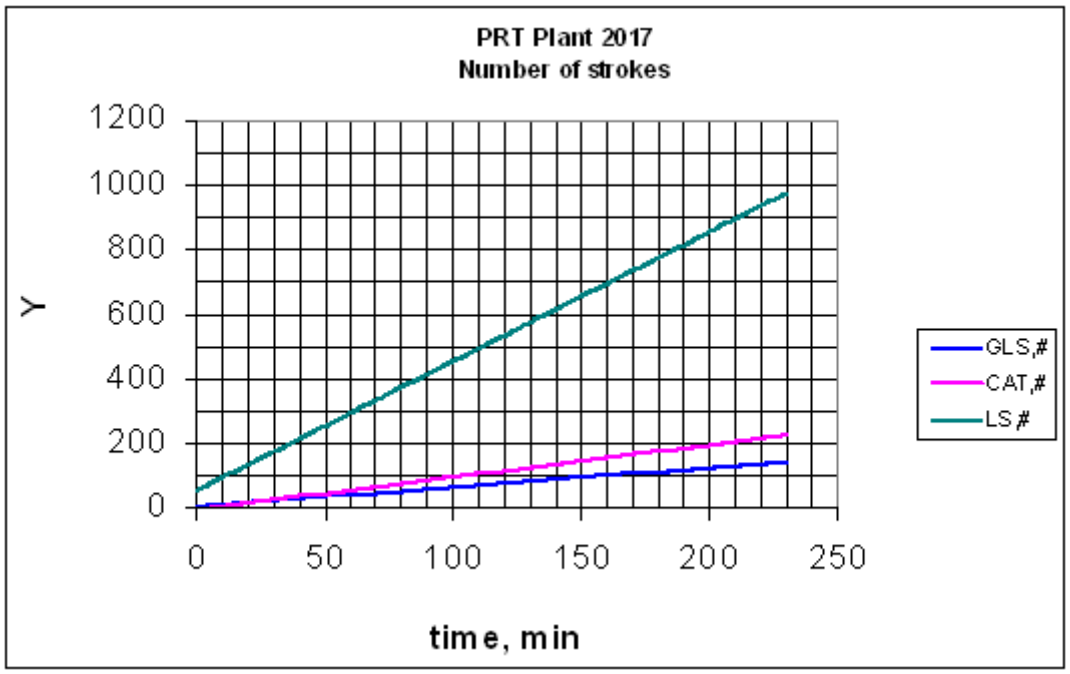
DATA ACQUISITION

Our SPS – PC combined data acquisition system allows plant control as shown in the graphs below:

Pumping



Catalyst slurry feed is constant. Starting with 1500mL filling, catalyst is consumed after 220min at CAT=60



LS =15; CAT=60; GLS=100

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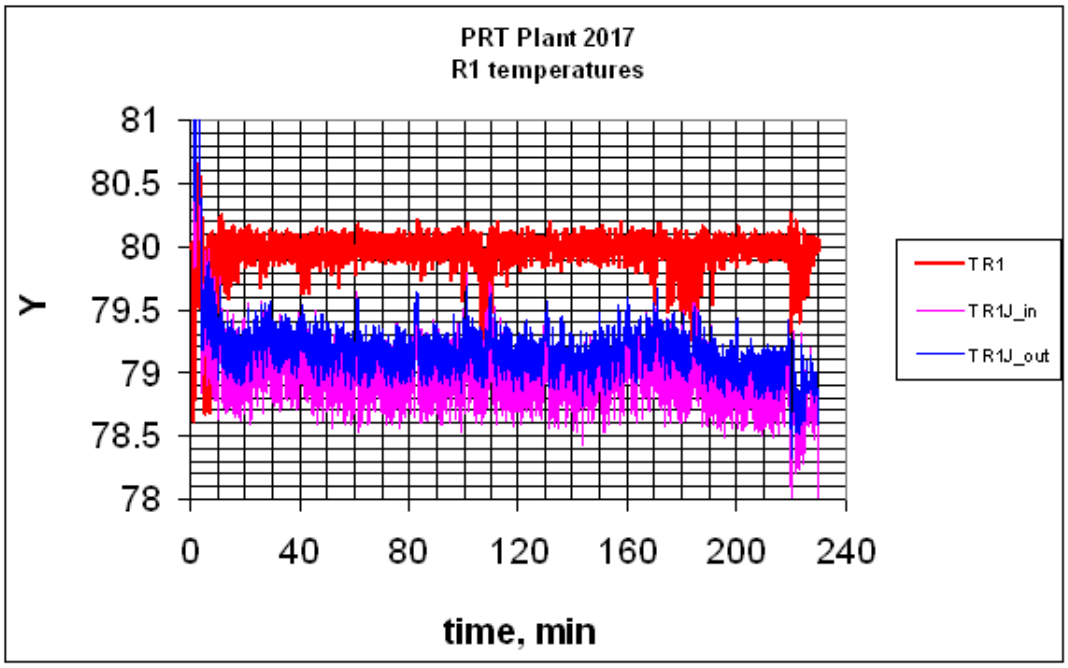
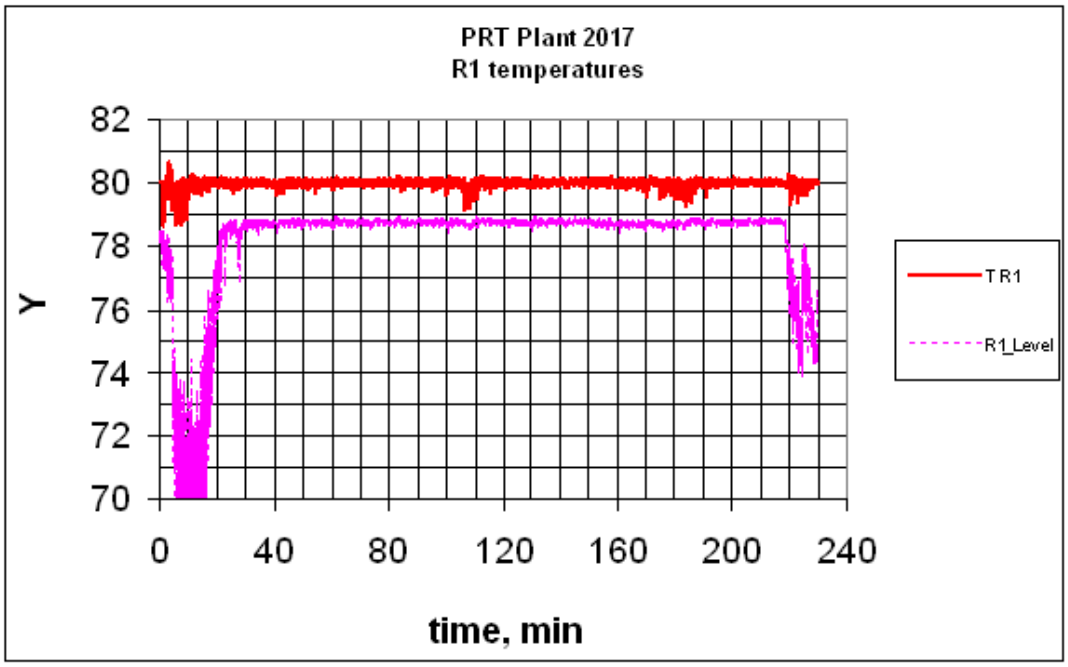
R1-Temperatures



Very constant reaction temperature TR1. Temperature difference between reactor content TR1 and cooling water temperature TR1J_in and TR1J_out represents polymerization rate.
About 100mg catalyst was injected before feeding C2, therefore reaction starts immediately with feeding C2. Increasing temperature difference until steady state if all relevant parameters are kept constant.



The temperature difference between TR1 and "level temperature" indicates the slurry level in R1.

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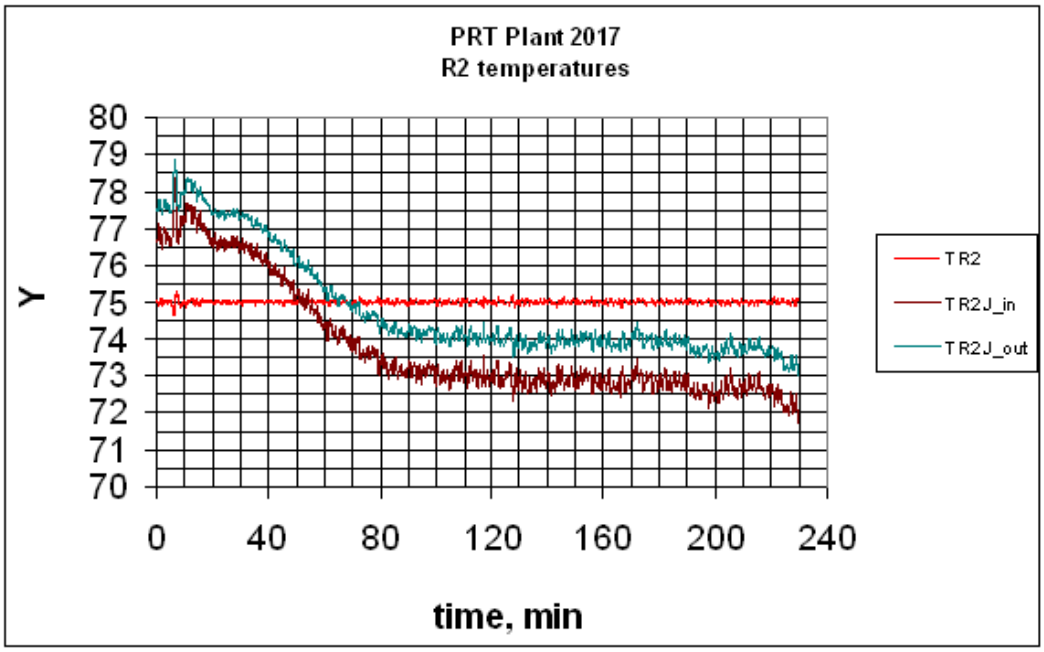
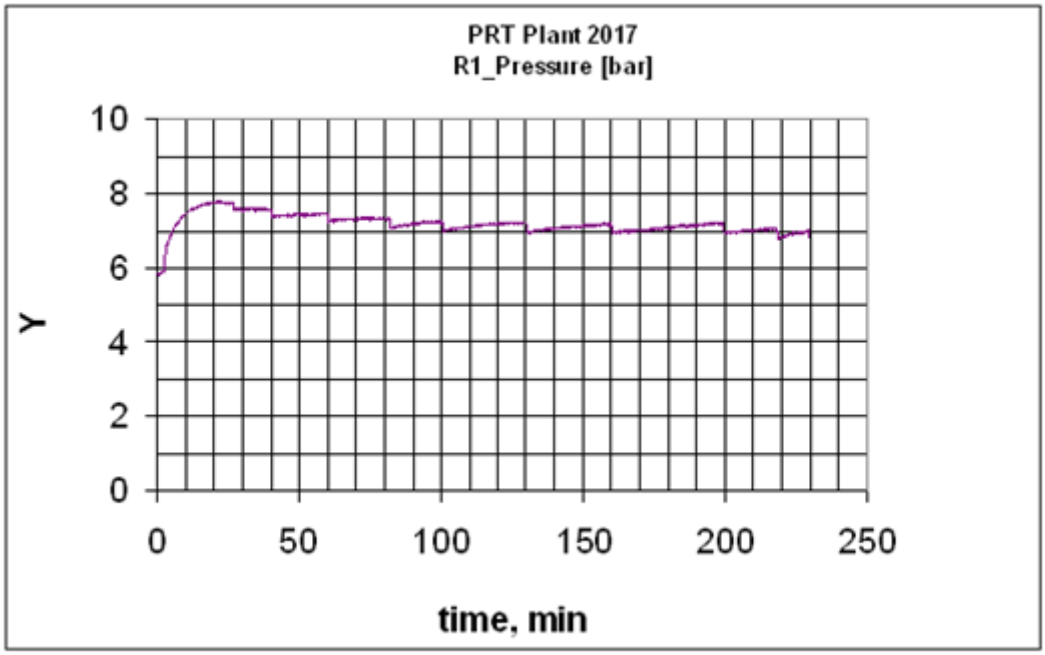
R2-Temperatures



Very constant reaction temperature in R2. Decreasing cooling shows increasing polymerization rate.

R1-Pressure



The chosen process control mode allows quick finding of steady state.

Sampling causes quick pressure decrease: 9 samples have been taken.

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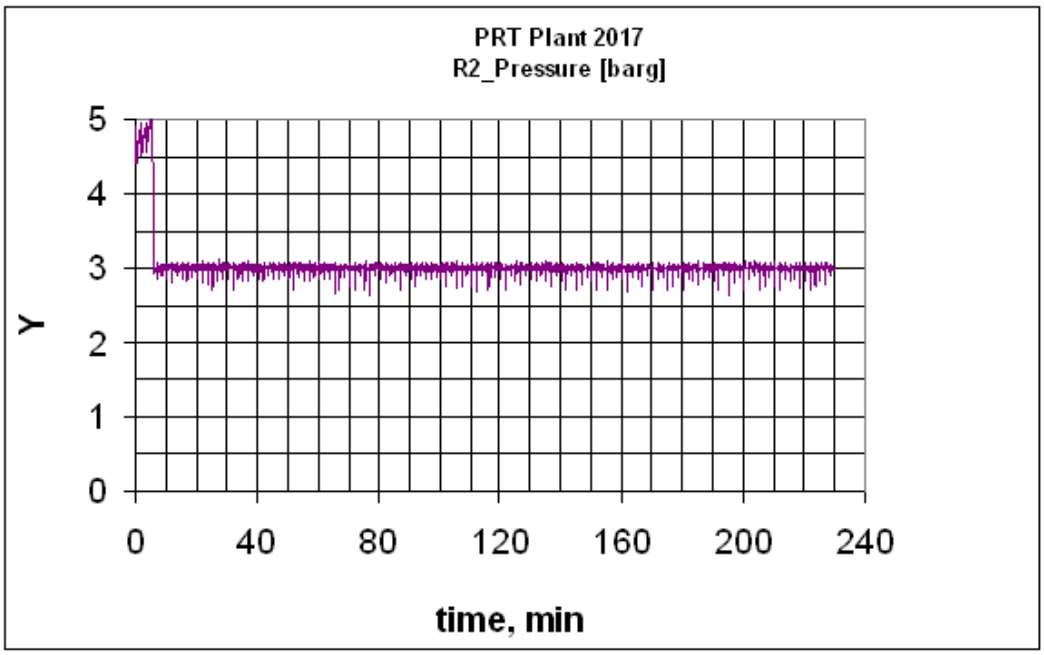
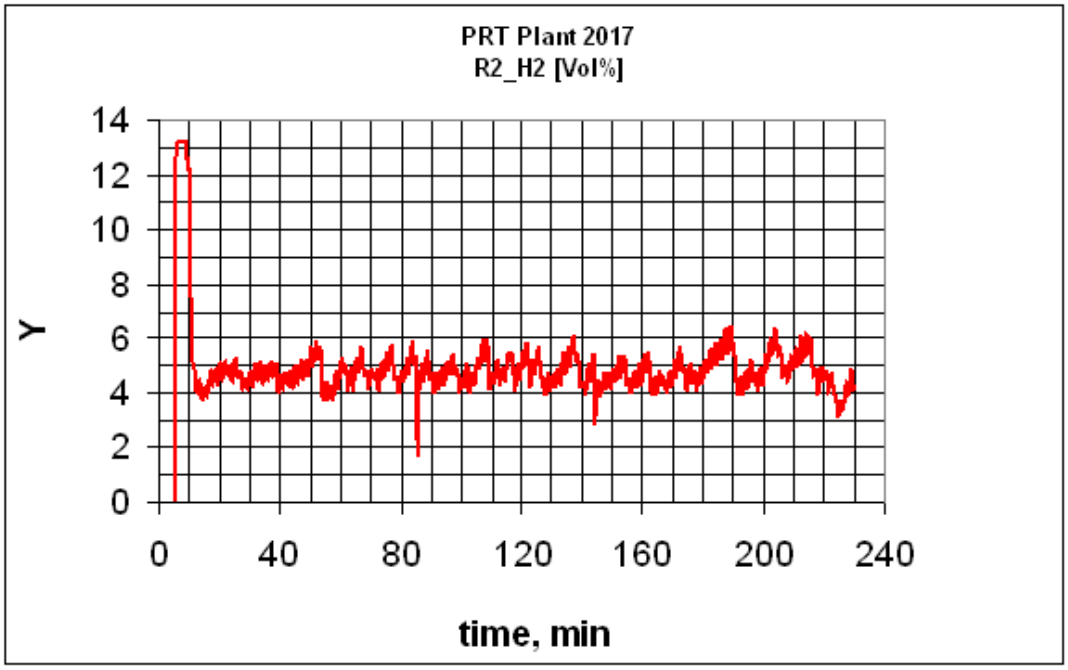
R2-Pressure



R2 pressure was kept constant at 3 barg.

LS and LSG pumping cause neglectible small oscillations.

R2-H2 Concentration



The desired value was 4.5%.

In this reactor configuration purge the excess H2 out of R2 by means of an extra C2 purge.

Meanwhile PRT has developed a combined "outlet vessel – pump" system that allows gas composition changing between R1 and R2 – the extra C2 purging is not required anymore.

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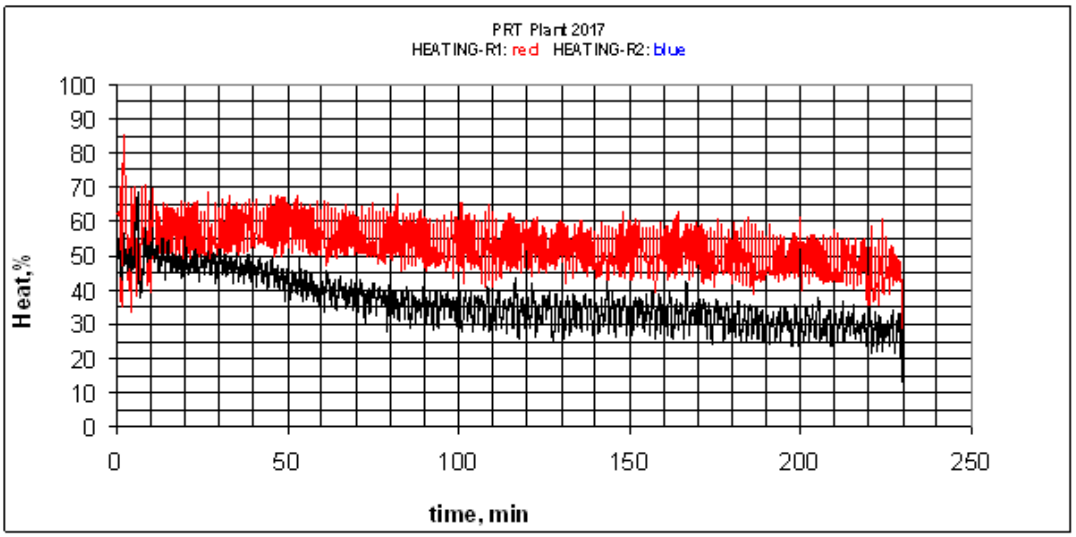
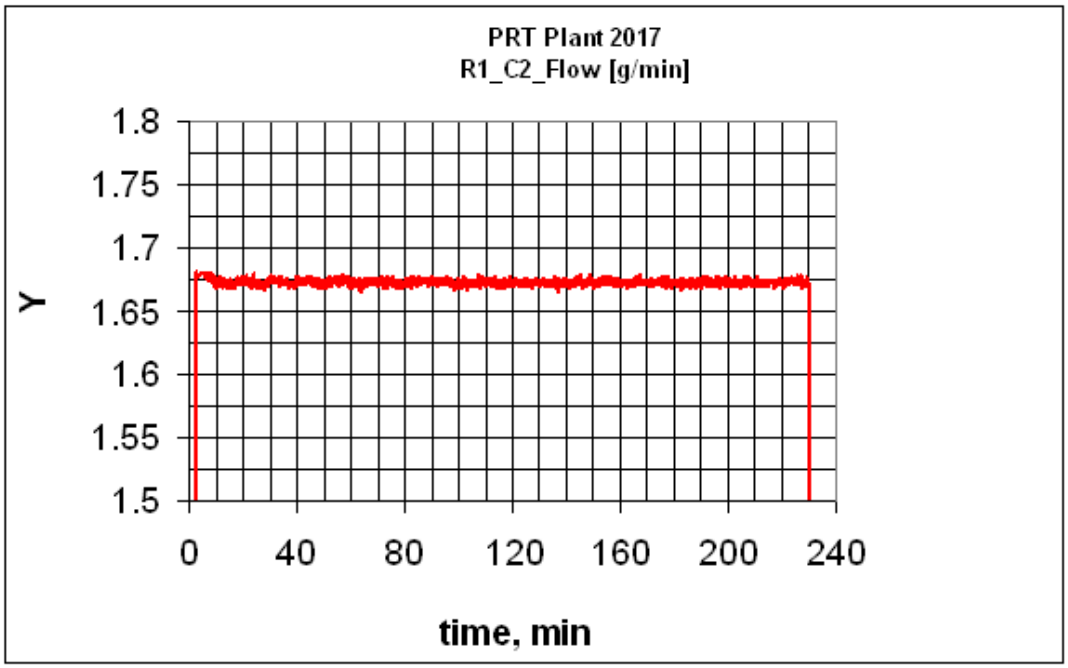
Reactor Heating-Cooling



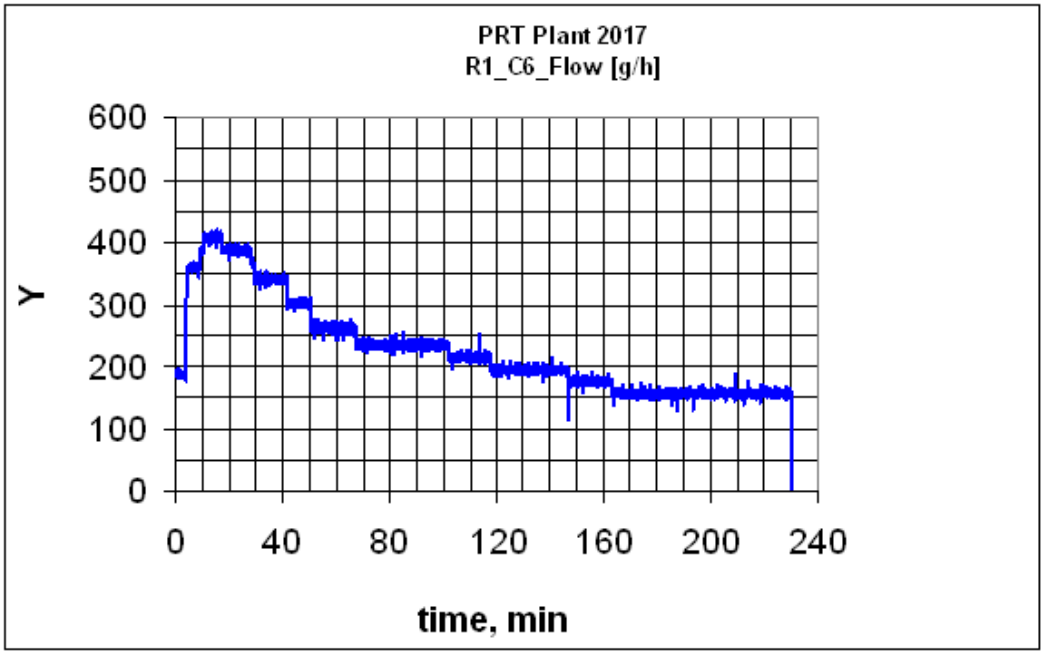
Decreasing heating capacity indicates increasing polymerization heat.

On-line measuring of the polymerization rate via this calorimetric principle is possible.

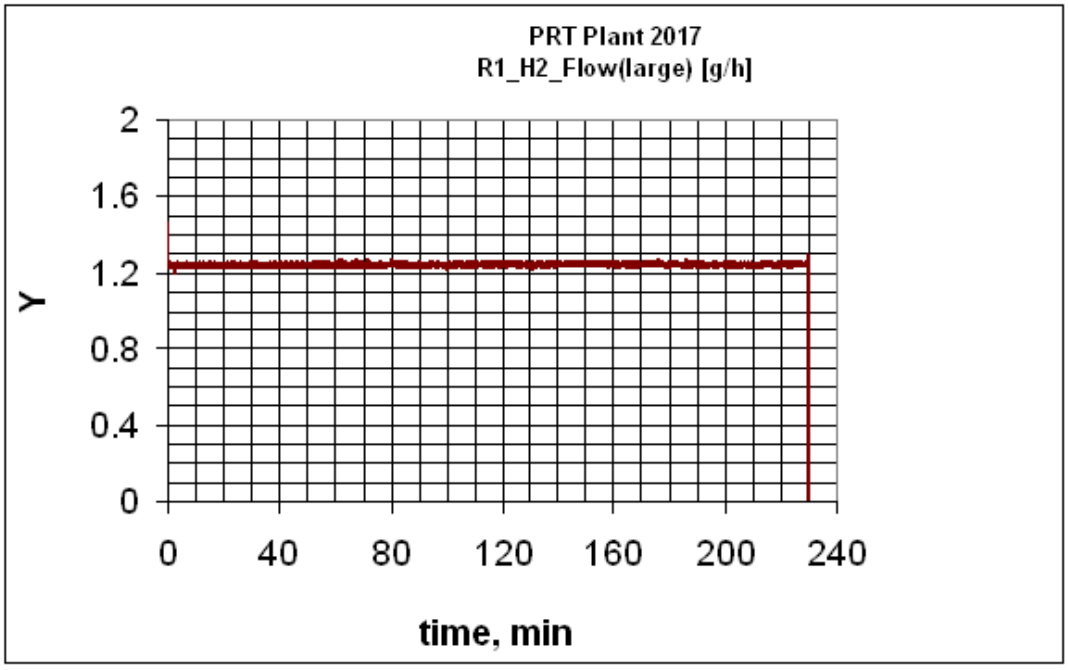
R1-Feed



C2 feed to R1 was set constant at 1.675g/min = 100.5g/h. This – together with the overall C6 feed to R1 (C6 feed + catalyst feed) – determines the R1 solid content. The final solid content in steady state can be "designed" at the beginning of the experiment.

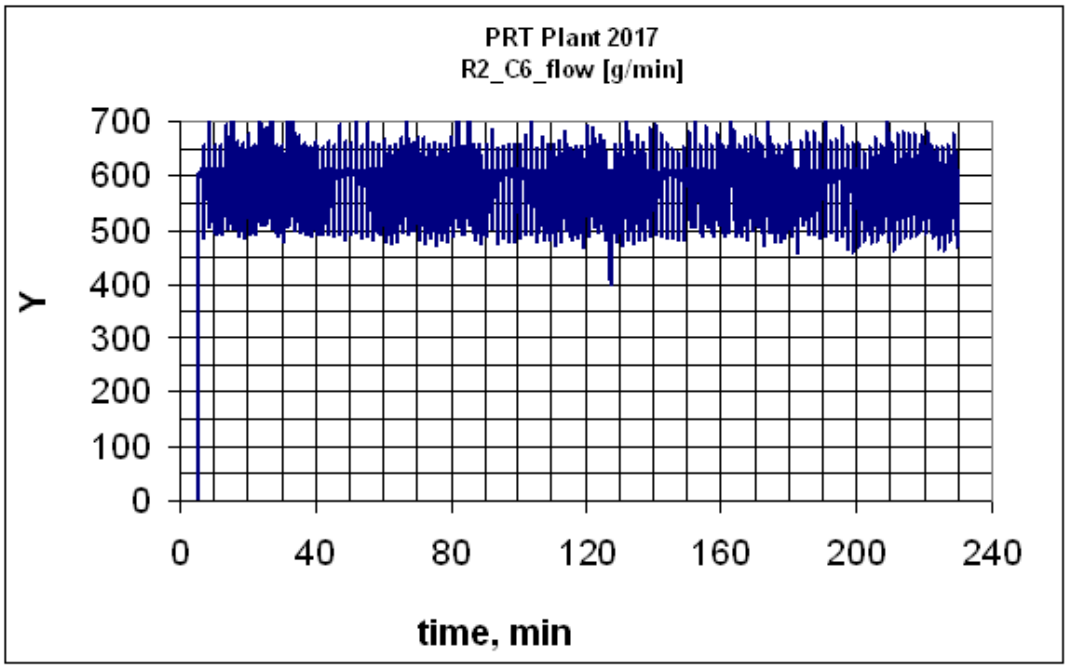


R1-C6 was decreased stepwise to get the R1 level controlled – the final value is 157g/h.
Together with C6 from the catalyst feed this gives 320g C6/h.
Taking 100% C2 feed conversion into account, we get a theoretical maximum polymer content of 24% in steady state R1, which is in quite good agreement with the SAMPLING results, see table above.

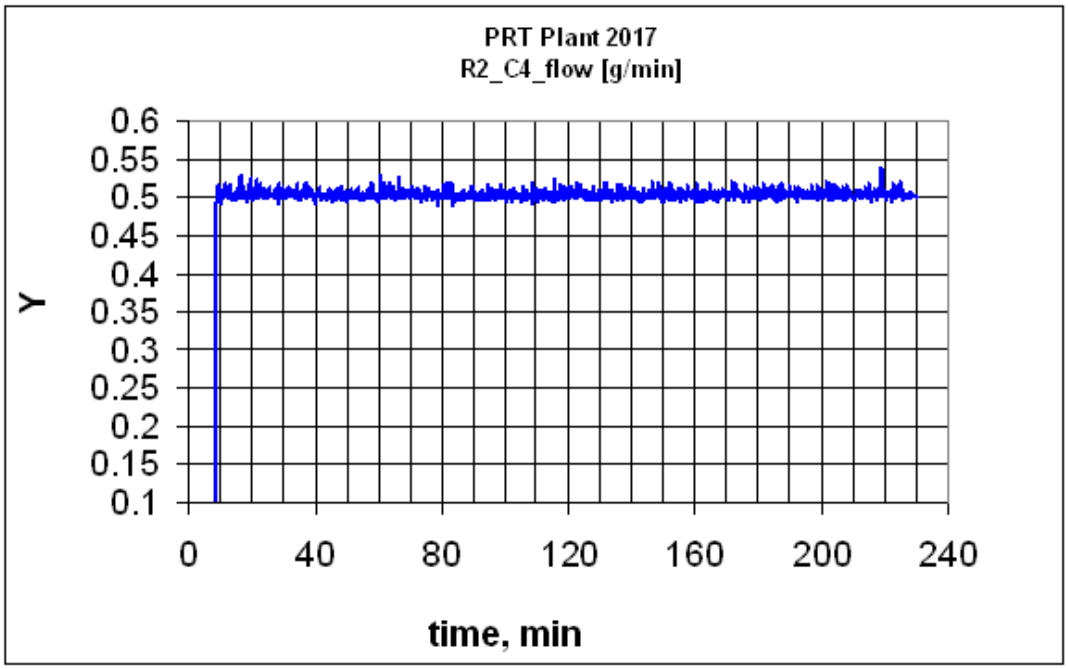


The R1-H2 feed was constant at 1.245 g/h during the whole experiment.

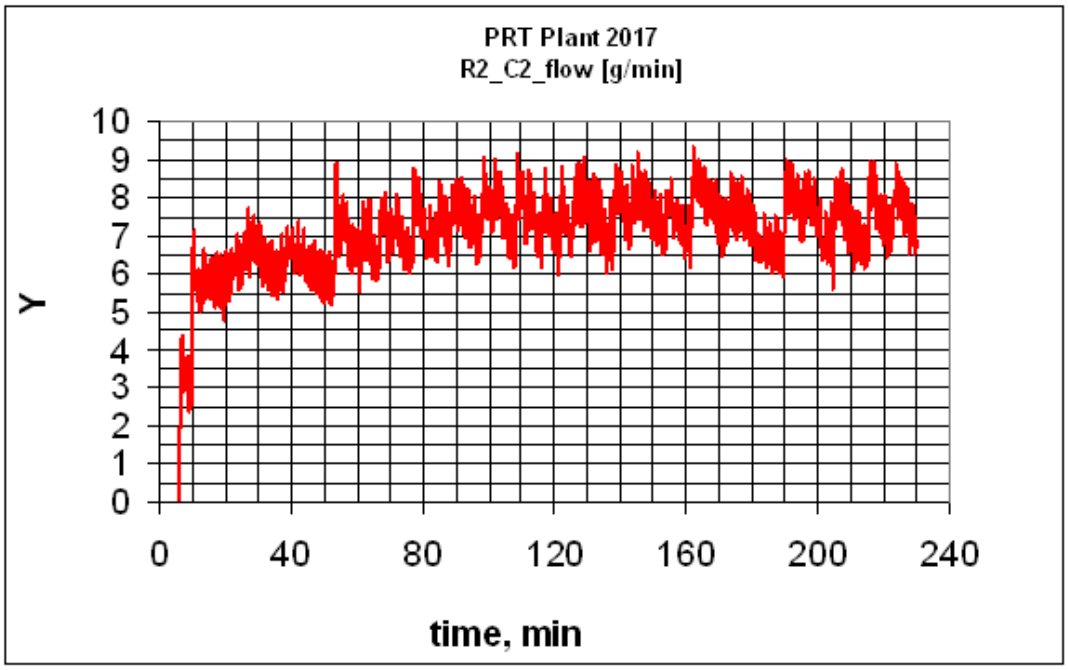
R2-Feed



The average (constant) C6 feed to R2 was 590g/h.



The co-monomer (1-butene = "C4") feed to R2 was kept constant at 30g/h.



The overall C2 feed to R2 contains currently the amount of C2 that is required for purging the H2 concentration down (to 4.5%, see above). Near to steady state the value was 450g/h.

The overall production of polymer was 290g/h with 100g/h produced in R1 and 190g/h produced in R2, therefore the blending ratio is R1 : R2 = 0.52 g/g.