

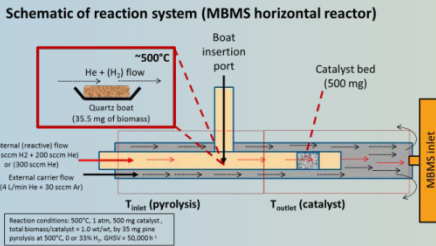

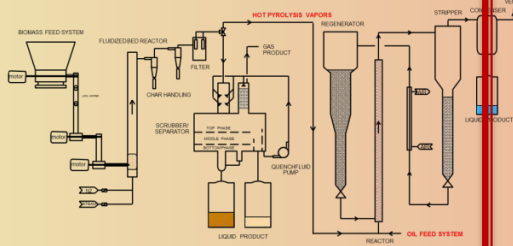

# Pilot-scale CFP Commissioning: *Creative Problem Solving and Lessons Learned*

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Rosemont, Illinois  
October 8, 2019



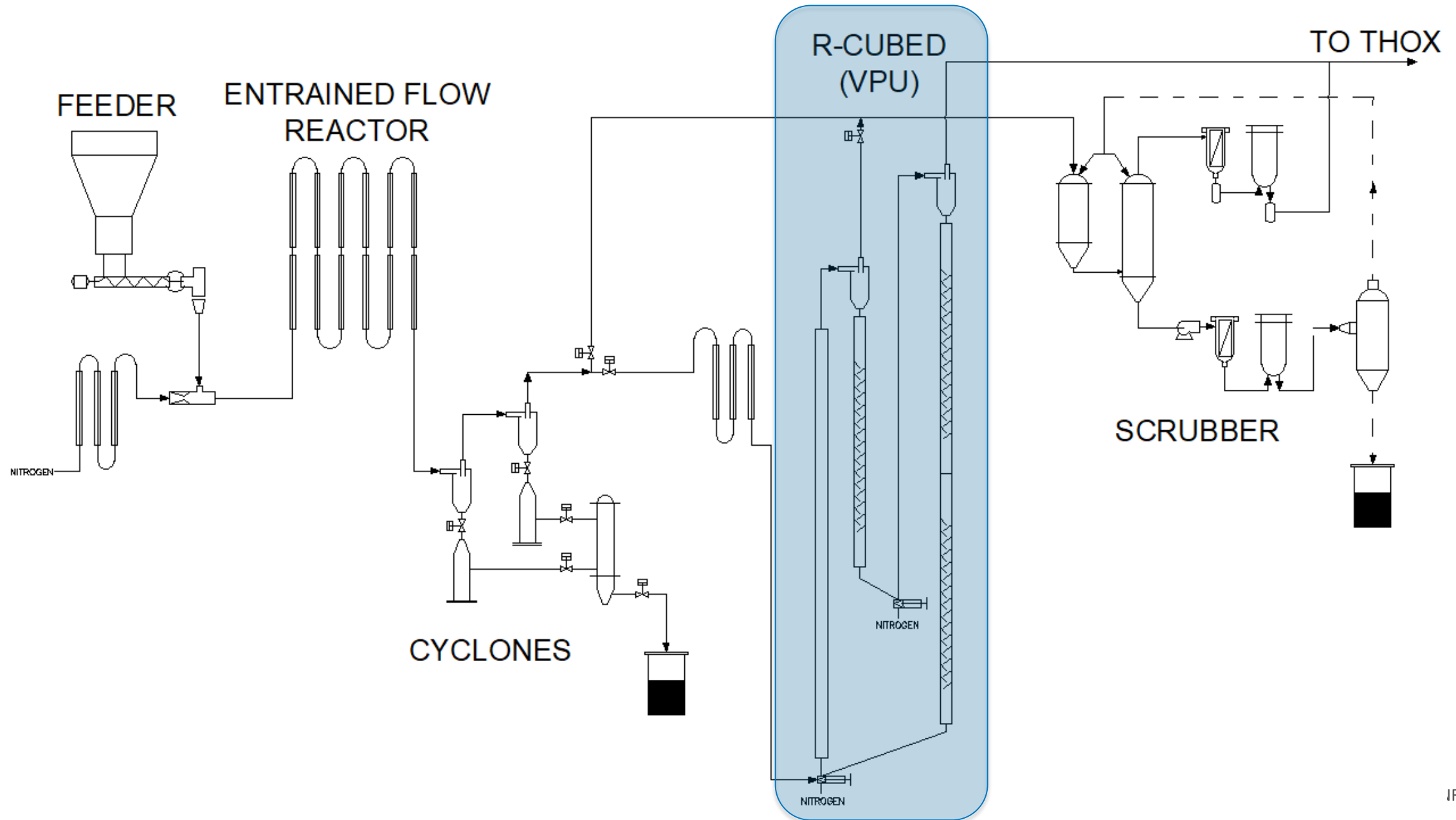
# Staged Multi-Scale Evaluation Improves Research Efficiency

<h2>Microscale Reactor</h2>  <p><b>Catalyst:</b> 1g</p>	<h2>Laboratory Scale Fluid/ Fixed Bed Reactor</h2>  <p>500g</p>	<h2>DCR (Small Pilot Scale)</h2>  <p>2kg</p>	<h2>R-Cubed (Pilot Scale)</h2>  <p>100kg</p>
<p><b>Biomass:</b> 25 mg/run <b>Uses/Purpose:</b></p> <ul style="list-style-type: none"> <li>• Rapid catalyst screening</li> <li>• Preliminary product analysis (no condensed oil)</li> <li>• Batch experiments</li> <li>• Mechanistic insight</li> </ul>	<p>0.5 kg/h</p> <ul style="list-style-type: none"> <li>• Catalyst evaluation with continuous biomass feed</li> <li>• Assess operating conditions</li> <li>• Full product/yield analysis</li> <li>• Extended time on stream</li> <li>• Fixed/Fluidized bed (not representative of riser)</li> </ul>	<p>3 kg/h</p> <ul style="list-style-type: none"> <li>• Process evaluation / integration with industrially-relevant riser</li> <li>• Assess operating conditions compared to lab-scale</li> <li>• Co-processing of biomass and petroleum feeds (liq. and gas)</li> </ul>	<p>20 kg/h</p> <ul style="list-style-type: none"> <li>• Evaluation of process operability / uptime</li> <li>• Identification &amp; assessment of scale-up challenges &amp; impacts</li> <li>• Generate significant product quantities</li> </ul>

## Process and catalyst evaluation at multiple scales:

- Improves research efficiency, thus reducing cost
- Provides data that is directly transferrable to industry partners
- Allows for a tiered catalyst and process development approach

# TCPDU Process Flow Diagram





## Challenges / Creative Problem Solving

- Design Constraints
- Measuring & controlling catalyst flow rate
- Pressure & level control
- Plugging in exit lines
- Air regeneration – complete coke combustion for catalyst efficiency

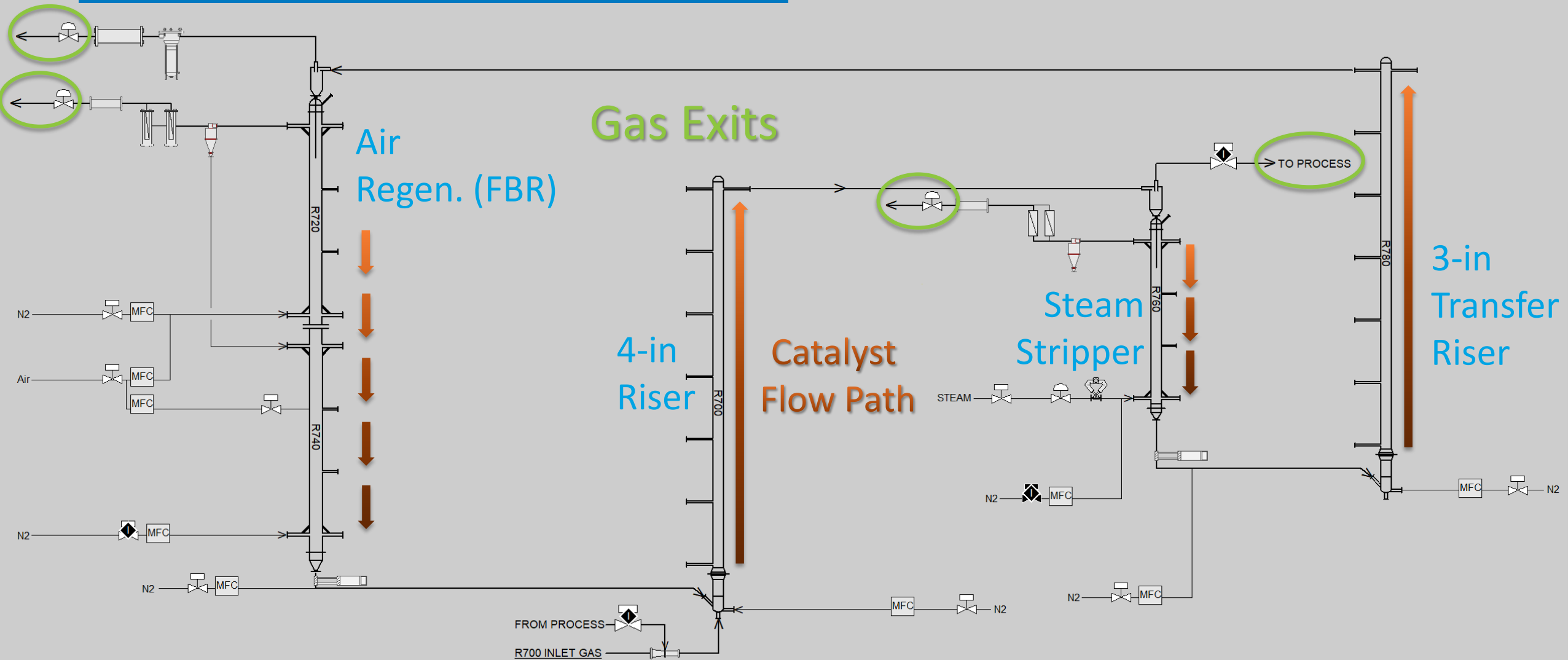
# Design Constraints

- Must have built-in flexibility
- Ceiling height limit
  - Must account for thermal expansion
- Floor loading limit (Techlok flanges)
- BPVC: limited to 6-in. diameter pipe
- Highly fluidizable catalyst (Zeolite)

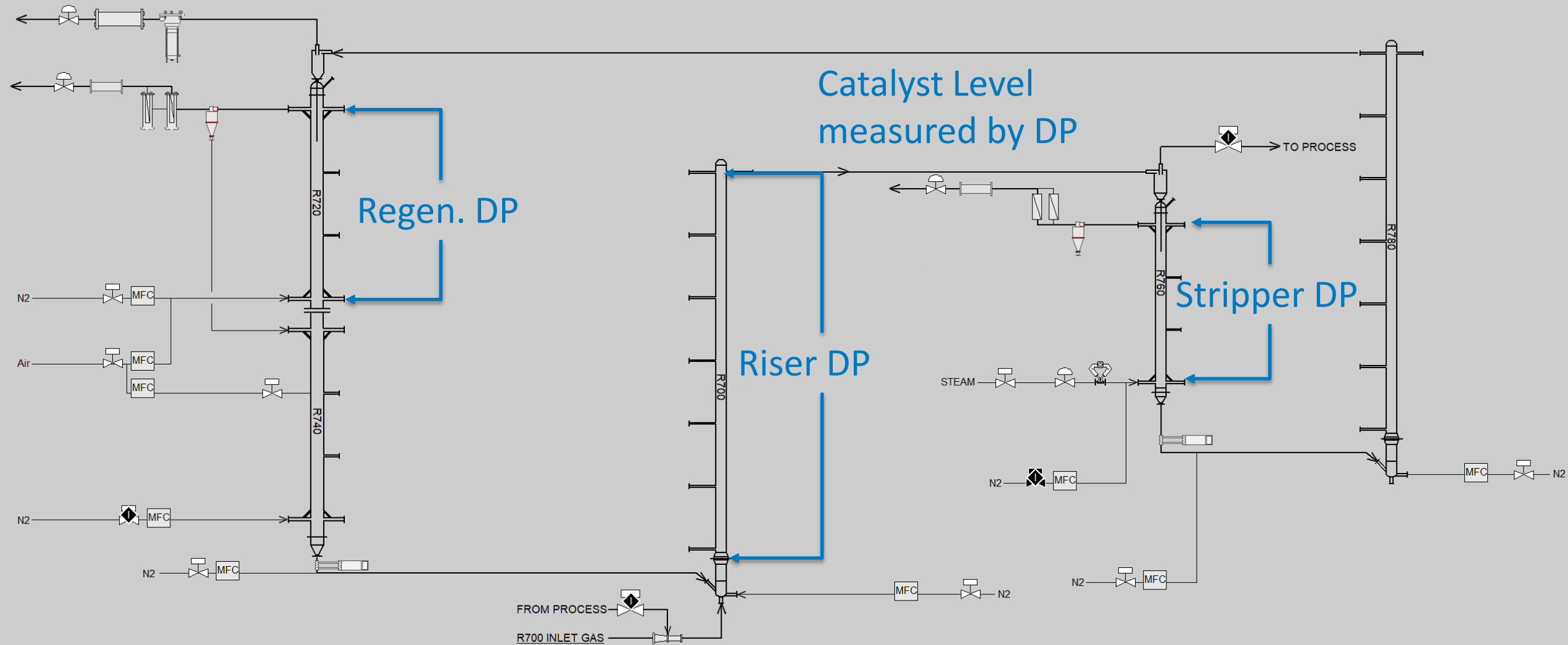




# R<sup>3</sup> Process Flow Diagram

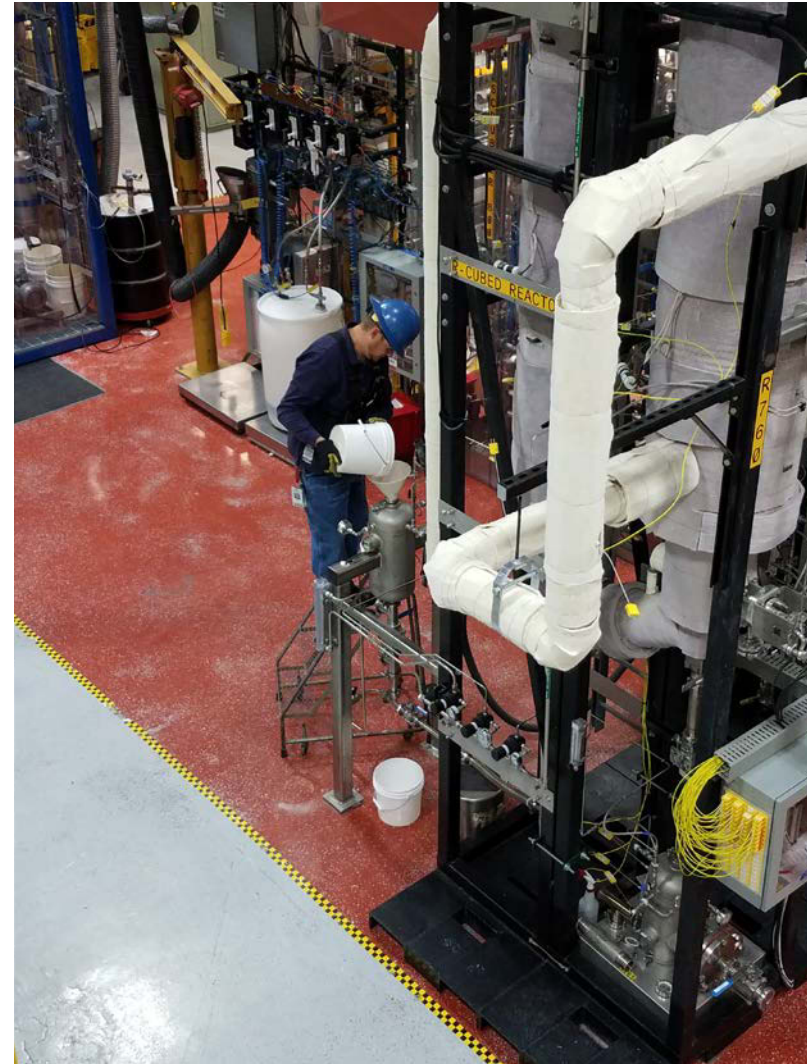
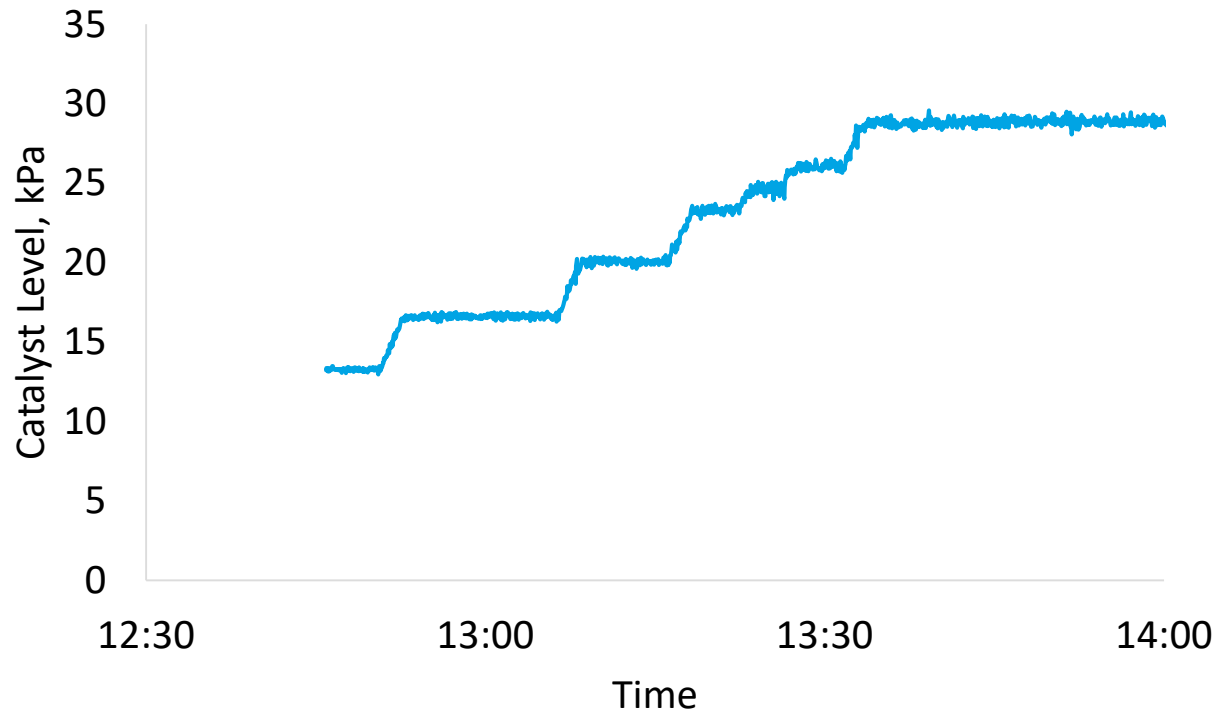


# R<sup>3</sup> Process Flow Diagram



# How to Measure Catalyst Mass Flow

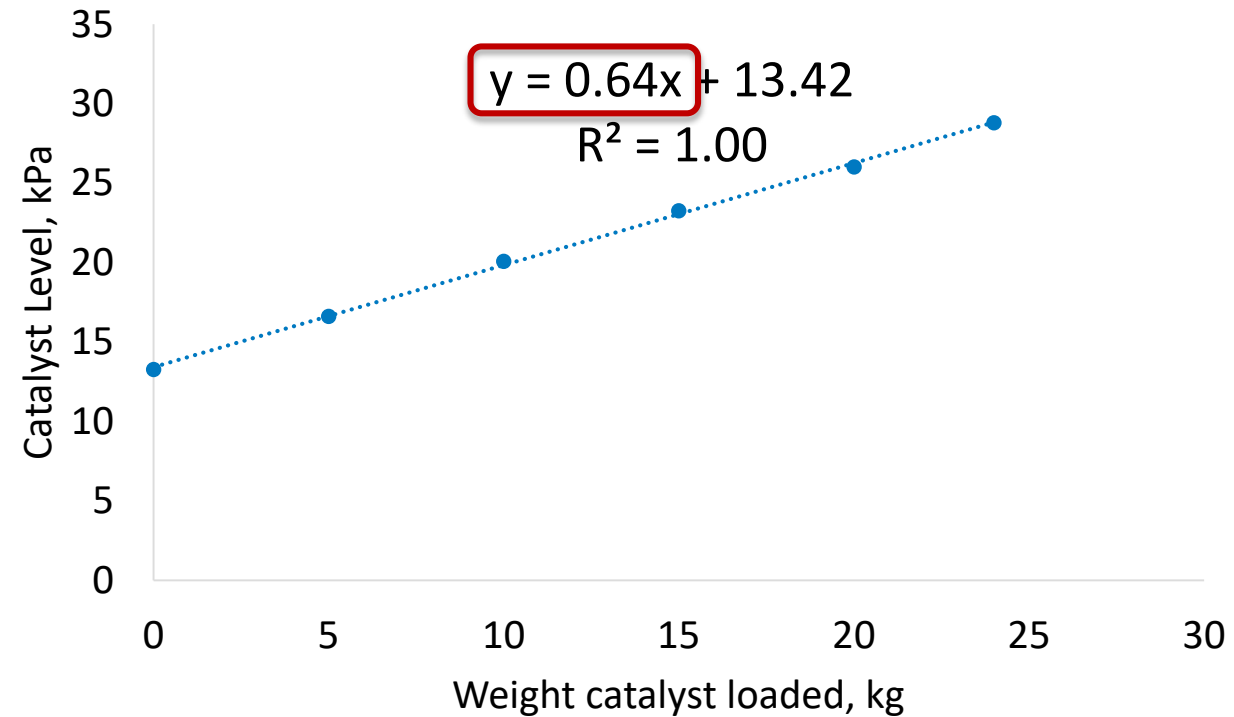
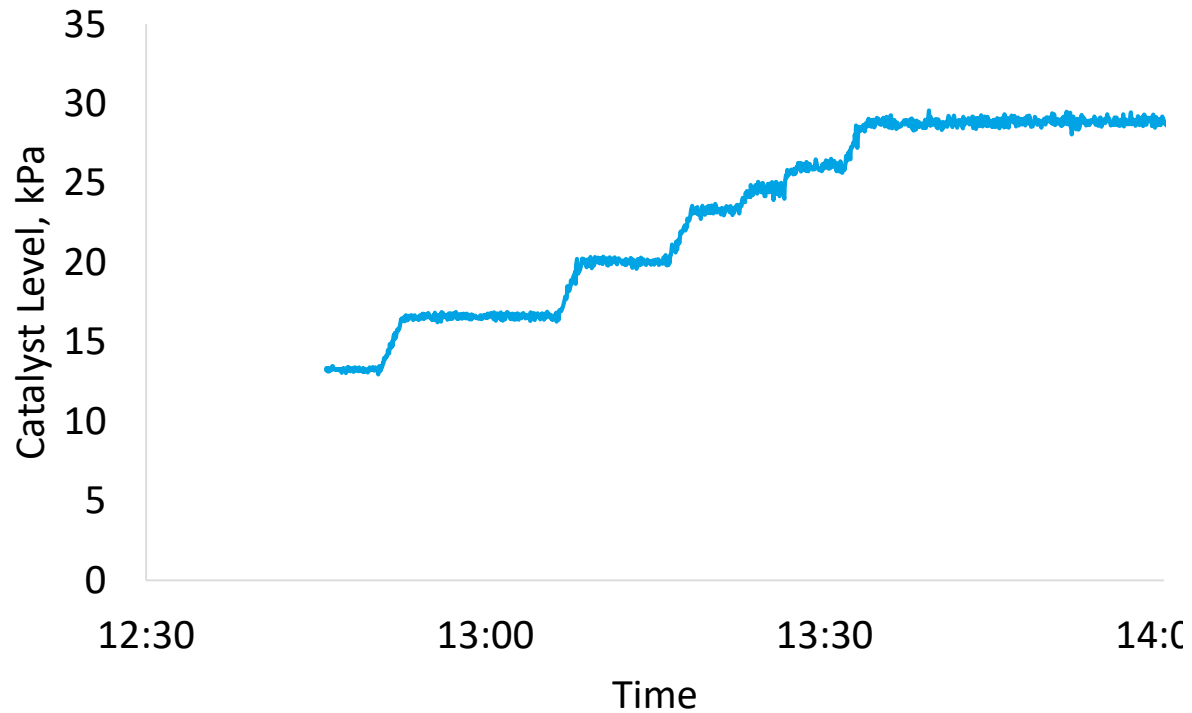
- Loaded 5 kg shots of catalyst
- $1 \text{ kg} = 0.64 \text{ kPa}$  at 60 kPa





# How to Measure Catalyst Mass Flow

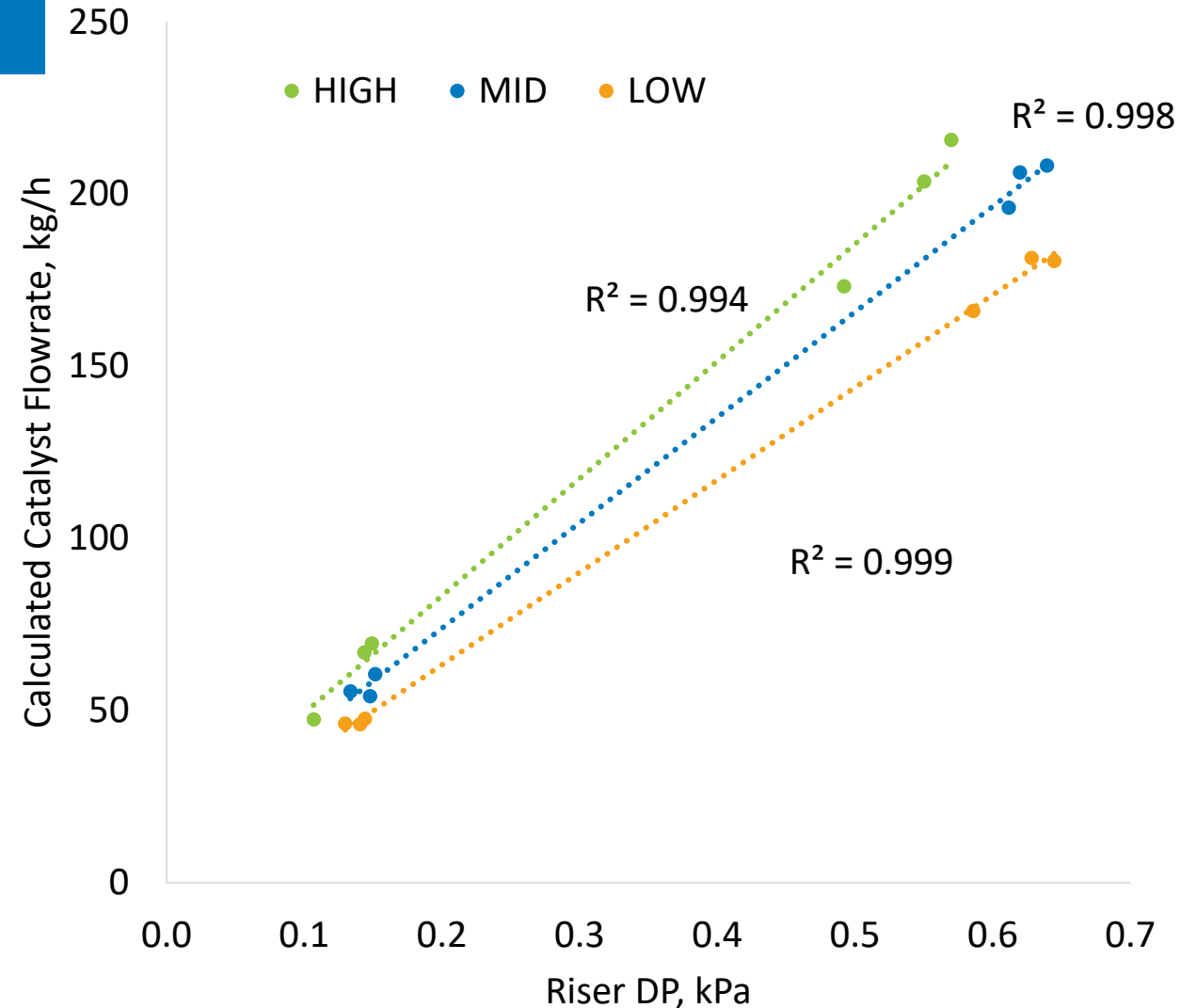
- Loaded 5 kg shots of catalyst
- 1 kg = 0.64 kPa at 60 kPa



# Catalyst mass flow required for kinetics

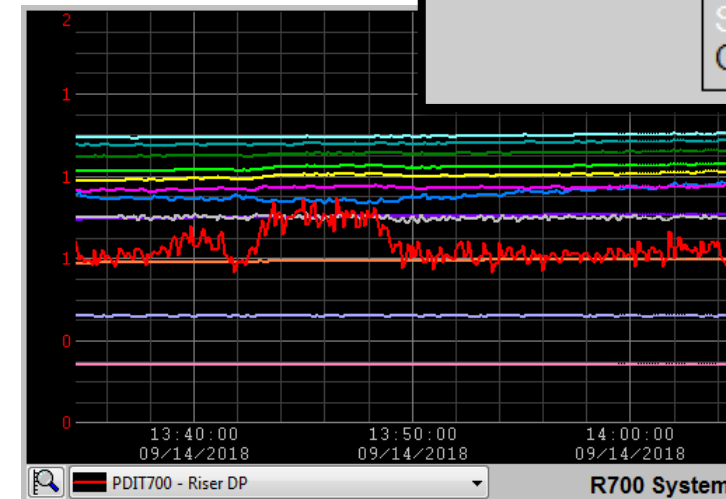
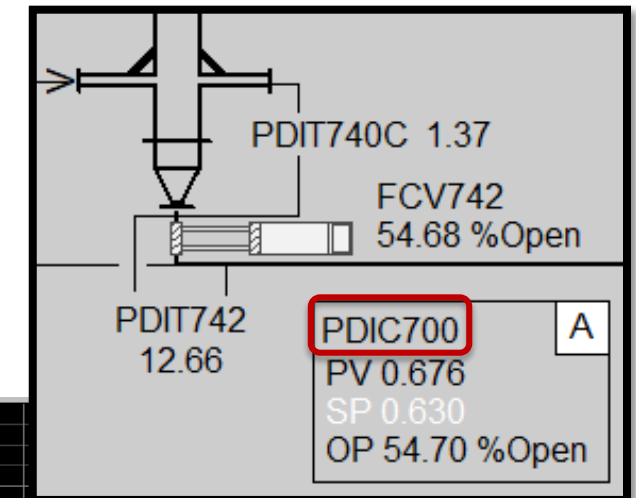
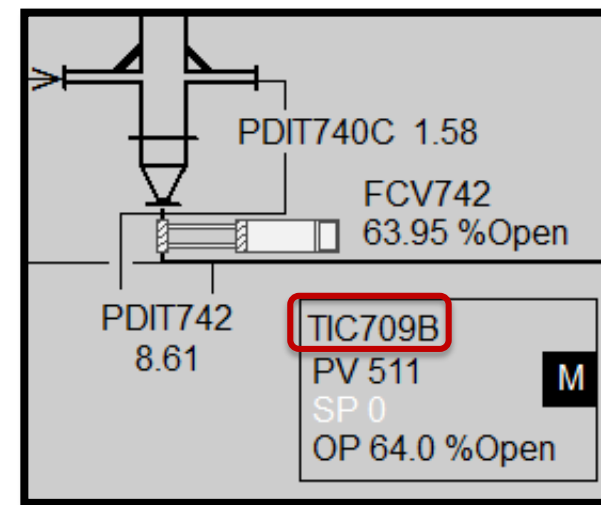
- Timed the transfer of catalyst from one FBR to other
- Simulated high/mid/low process gas flows with N<sub>2</sub>

$$\dot{M} = (\Delta P_{start} - \Delta P_{end}) \times \frac{1 \text{ kg}}{0.64 \text{ kPa}} \times \frac{1}{\Delta t}$$



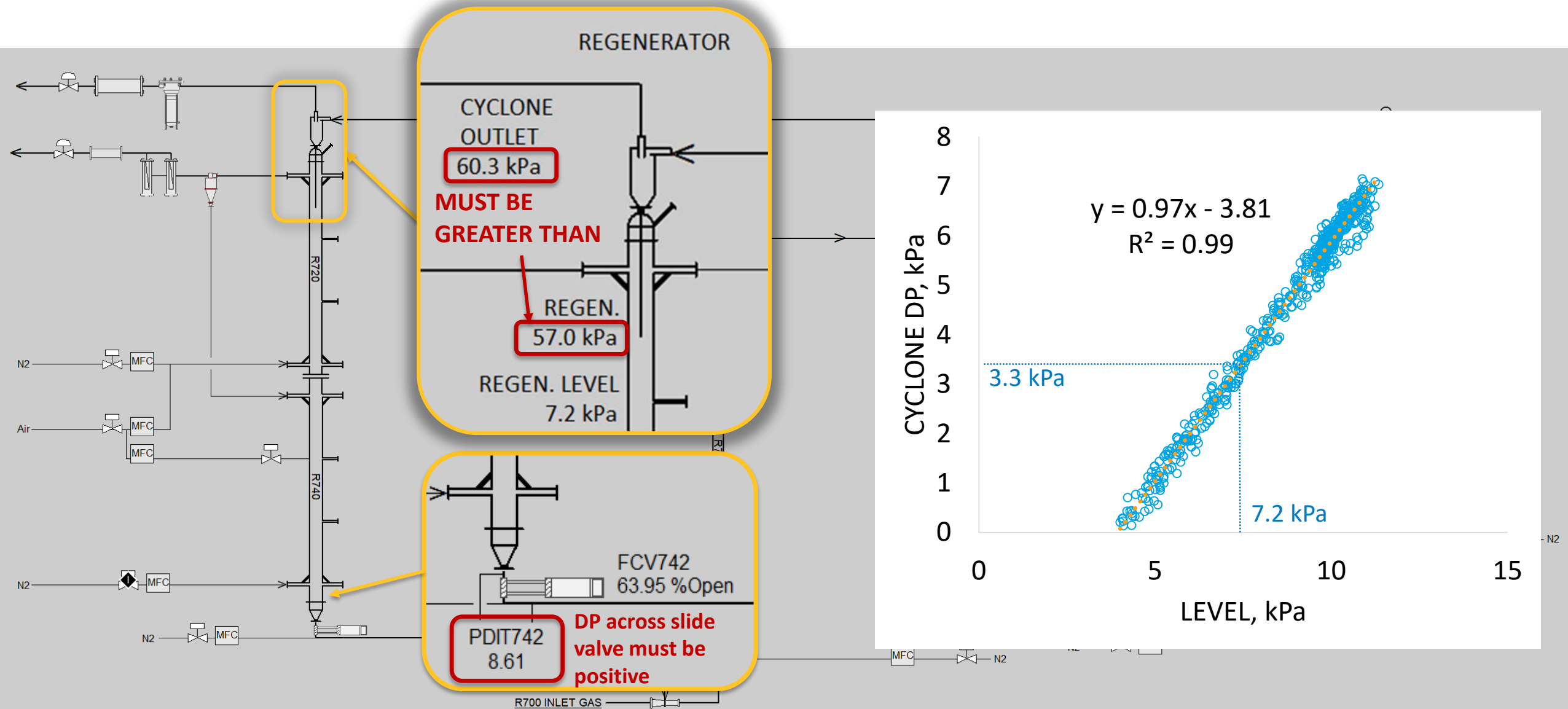
# Controlling catalyst flow via slide valves

- First generation: temperature at Riser exit (TE709B) controlled catalyst flow
  - Difficult due to thermal mass of riser & external heaters (thermocouple not sensitive to catalyst flow)
- Next generation: keeps catalyst flowrate constant, as measured by DP across riser (PDIT700)

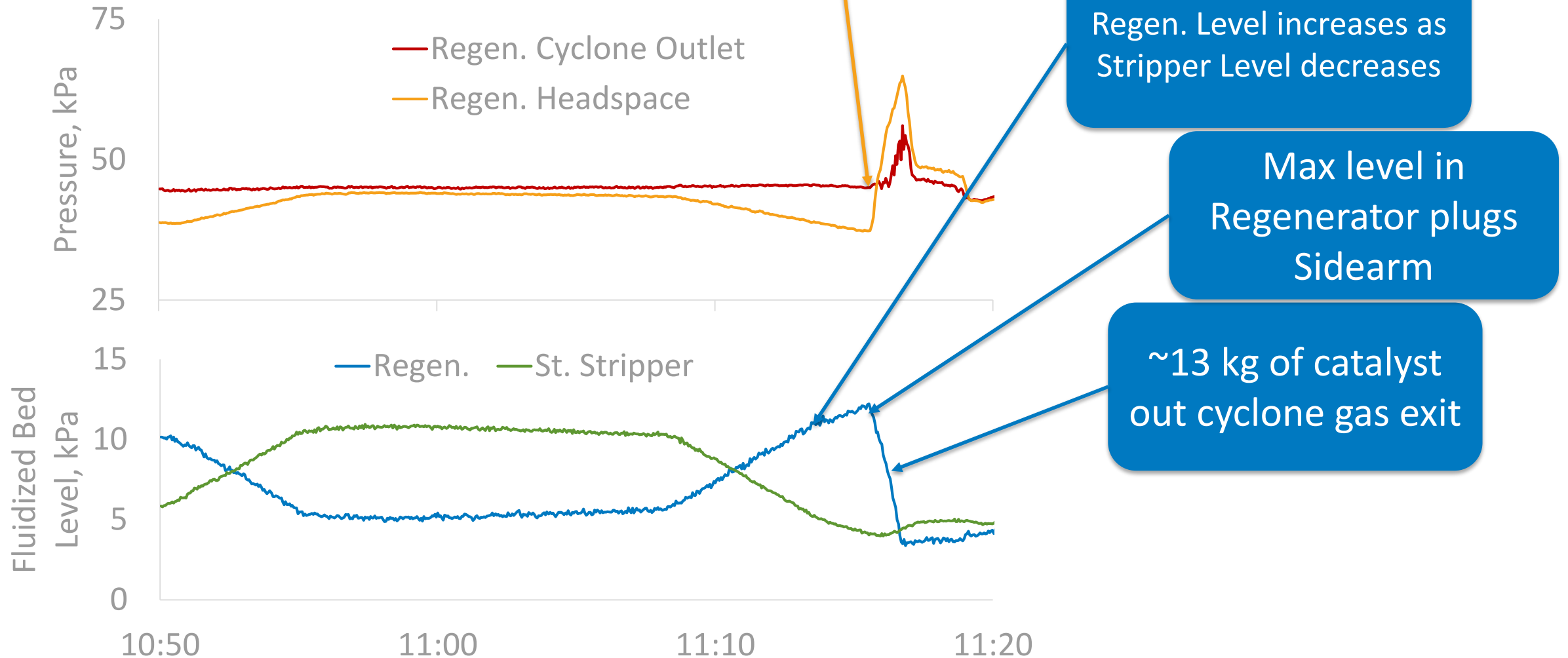




# Pressure Control in Fluidized Beds (Regen/Stripper)



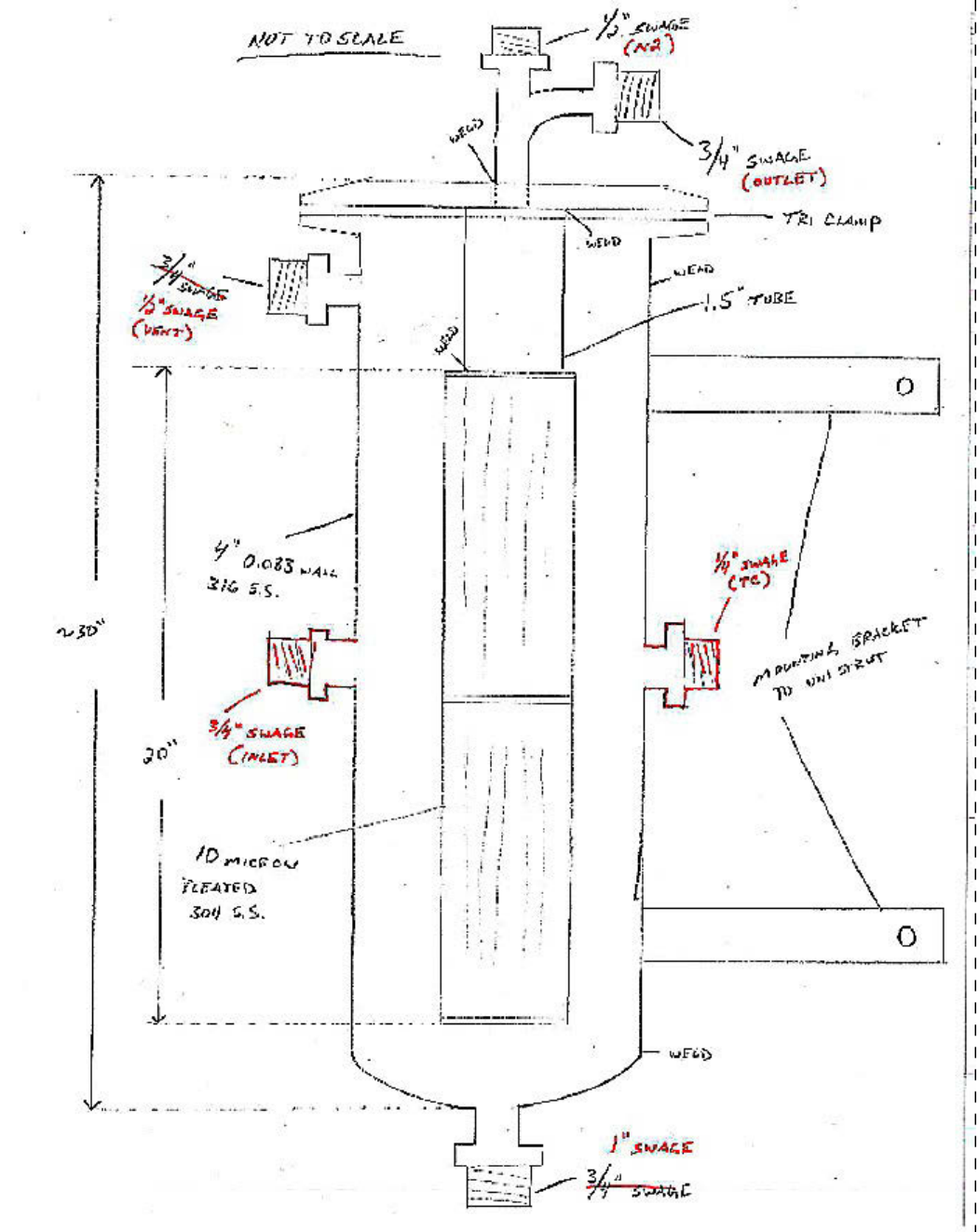
# Level Control & Maximum Level in FBRs



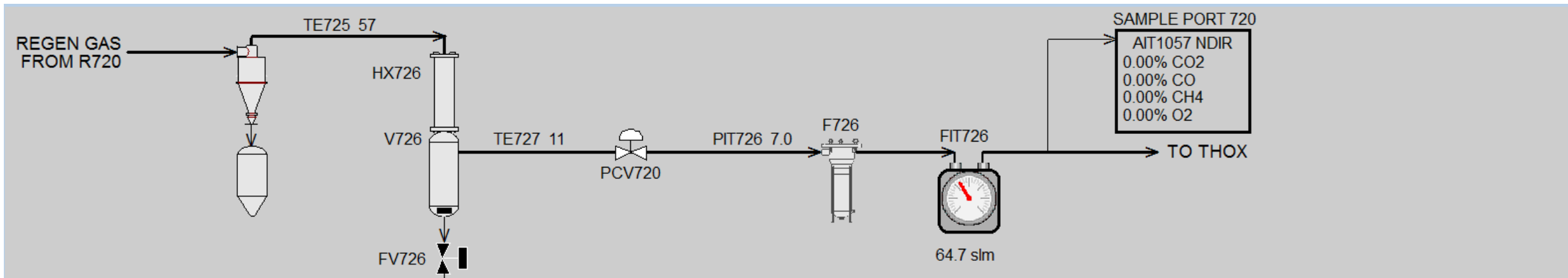
# Hot Gas Filtration

- Catalyst + water = Mud =  
Plugged Sidearms = Catalyst out
- Hot Catalyst + water vapor = ok
- Required on ALL exit streams

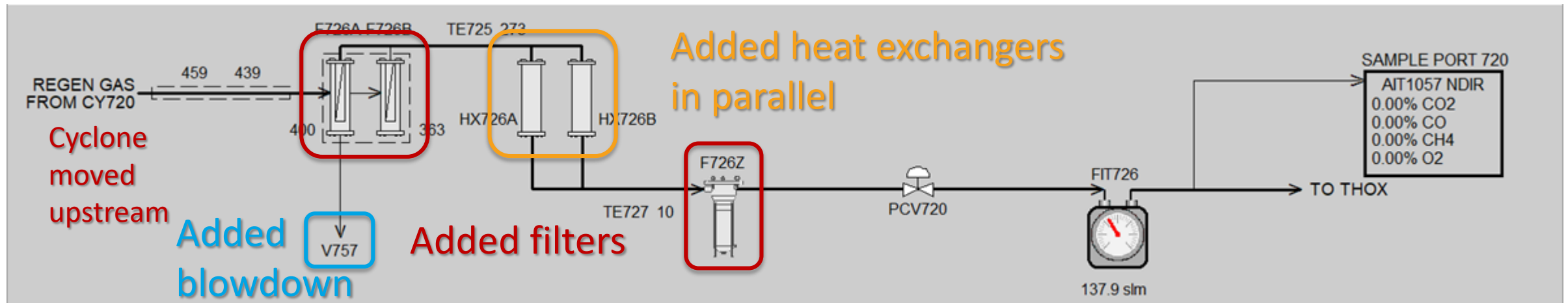
**CRITICAL TO SUCCESS OF OPERATION**







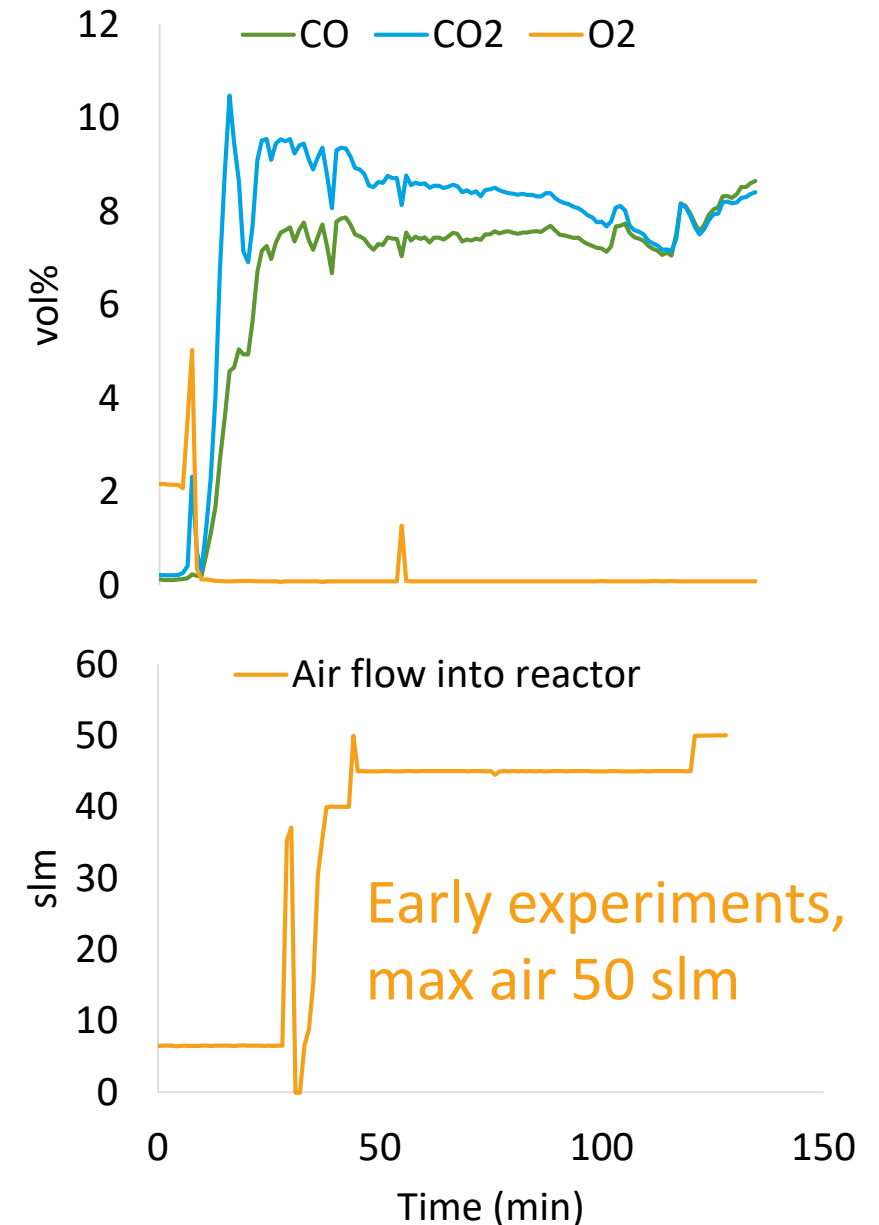
## Upgraded exit lines to remove catalyst particles while hot



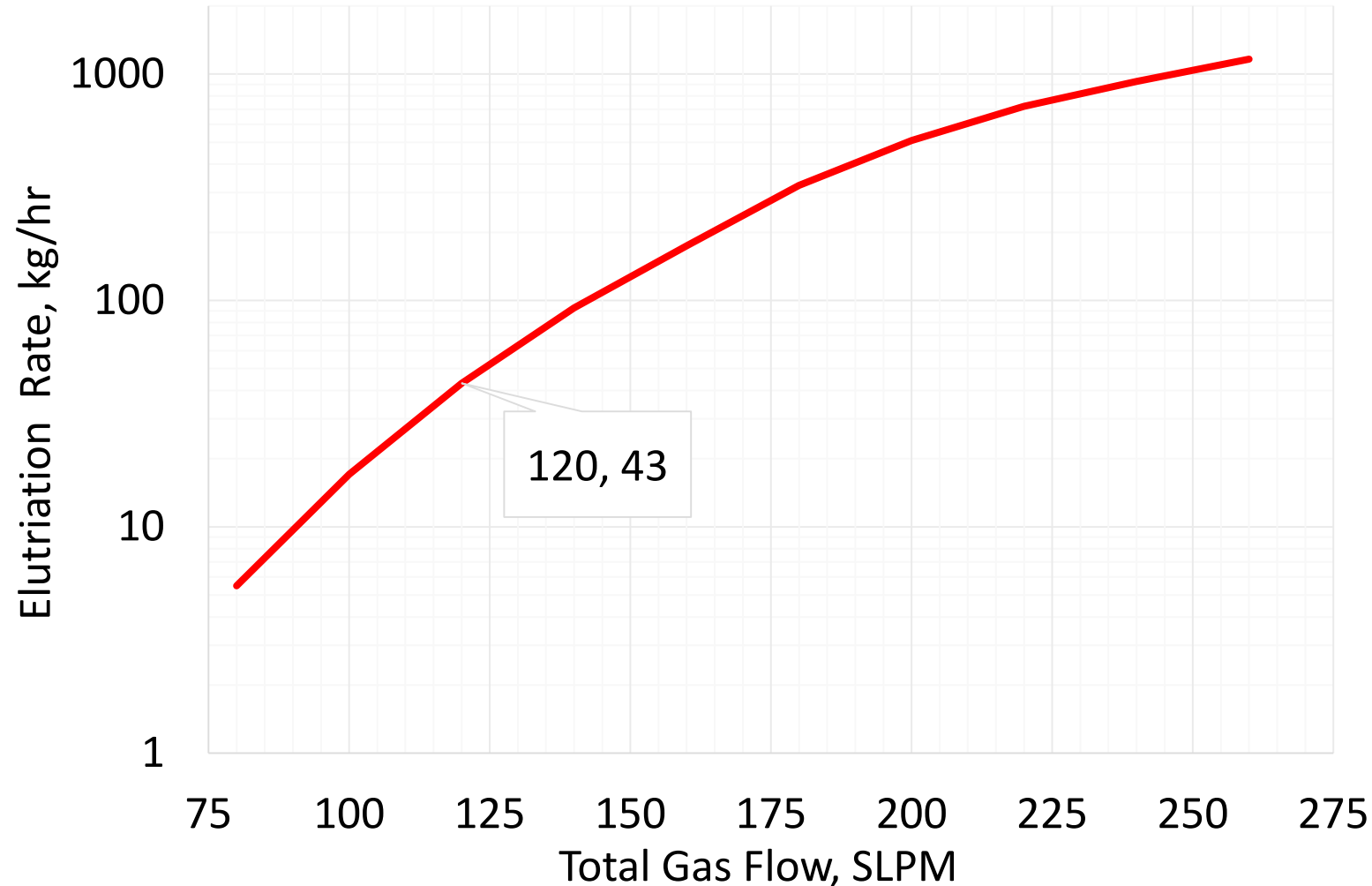
Upsized diameter of entire line

# Insufficient Regen Air

- Initial Design: Coke loading estimated from *preliminary* results on Bench-scale FBR
- No O<sub>2</sub> measured on regen exit
- Limited by reactor geometry:
  - Exit line too small diameter
  - Too much carryover (elutriation) at higher air flow



# Elutriation of Catalyst at Increased Air Flow

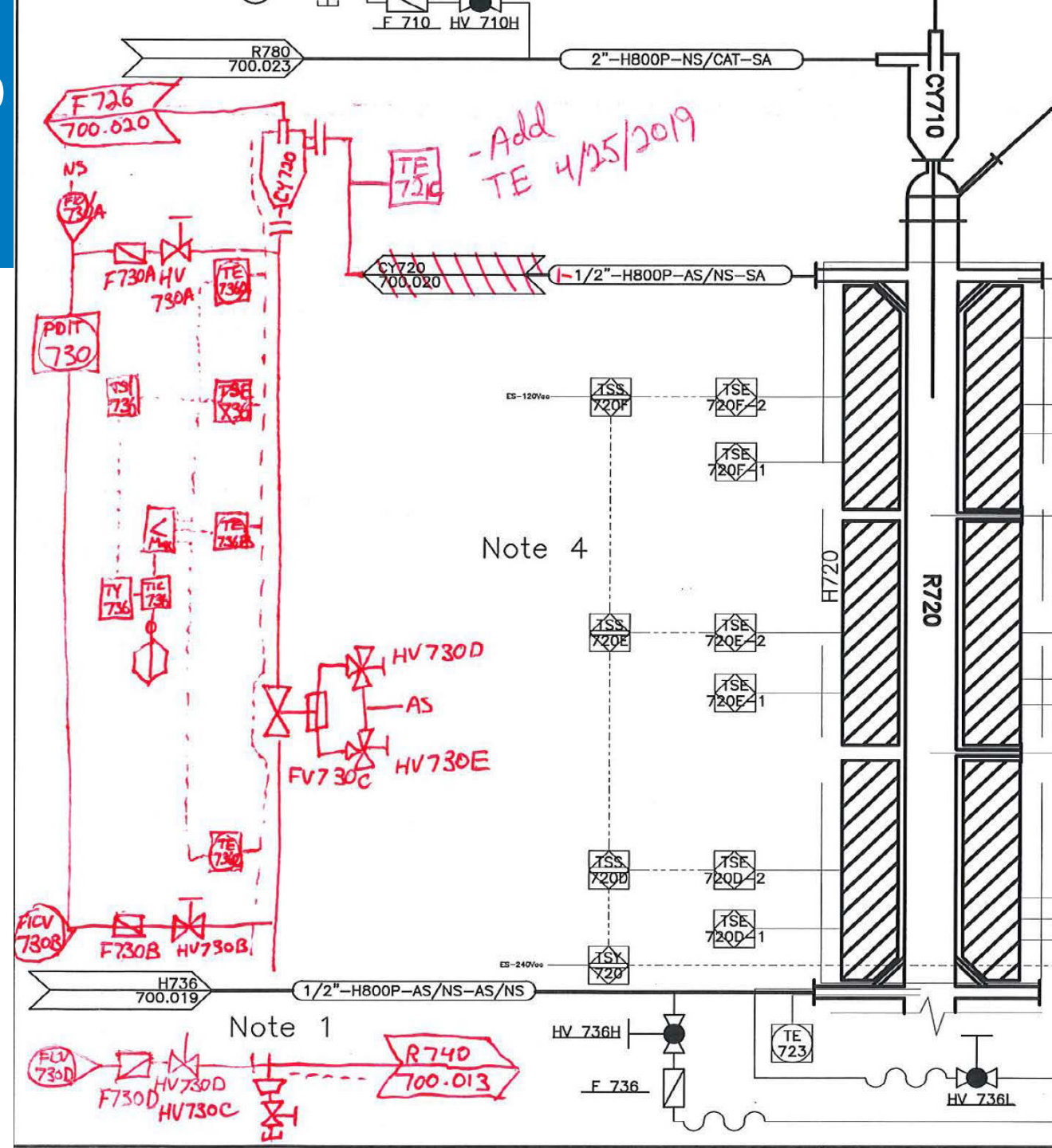


Credit:  
Bruce Adkins (ORNL)  
(using PSRI models)



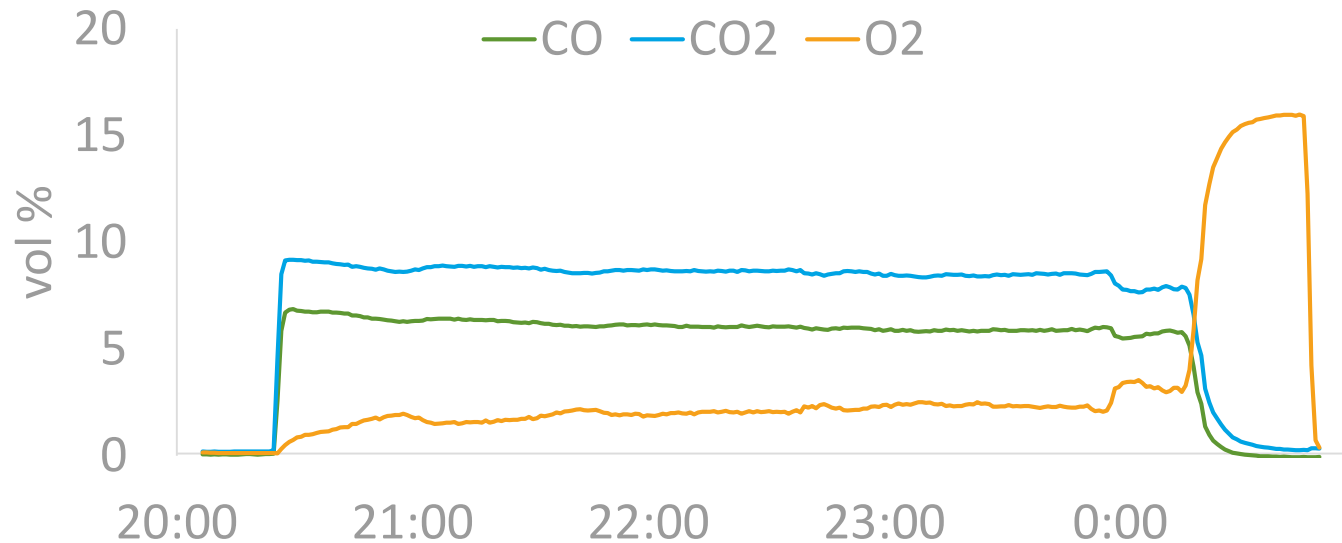
# Add Cyclone Return into Regenerator

- Increased air flow carries over MUCH more catalyst
- Effectively increased Regen. diameter
- Installed cyclone to return catalyst into reactor
- Tricky design: must keep horizontal section of return pipe fluidized



# Air Regeneration Results

Coke on Catalyst (%C by wt)	Insufficient Regen	Complete Regen
Post-stripper, after ~2 hours	0.94%	0.58%
Post-regen, after ~2 hours	0.66%	0.01%



Insufficient  
Regen



Complete  
Regen



## Lessons Learned

- Catalyst mass flow rate, which is critical for VPU kinetics,
- can be empirically determined by change in level in fluidized bed reactors,
  - then correlated to differential pressure across Riser,
  - *as long as gas flow rate stays constant*





## Lessons Learned

- Pressure in top of fluidized beds varies linearly with level of catalyst in bed
- D Pressure across Regen. cyclone must be positive
  - *Flowrate out sidearm must be greater than flow in, or pressure flips and catalyst empties*

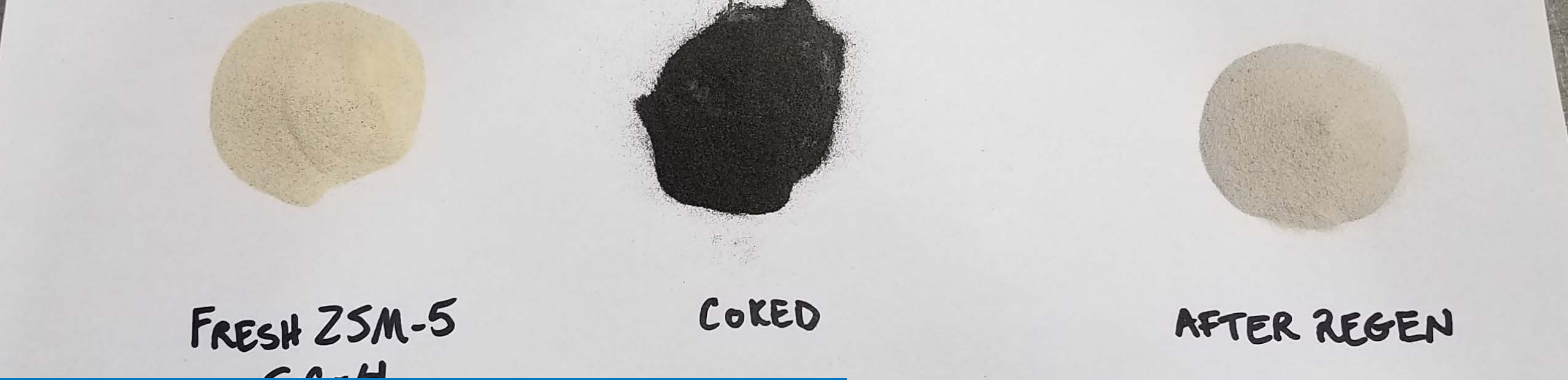




## Lessons Learned

### DON'T PLUG SIDEARMS

- *Don't overflow fluidized beds*
- *Filter catalyst particles out while hot  
(mud plugs lines & is difficult to clean out)*



FRESH ZSM-5

COKED

AFTER REGEN

## Lessons Learned

### NEED PLENTY OF O<sub>2</sub> FOR REGENERATION

- High-risk to scale up using bench-scale data from dissimilar reactor system
- We mitigated catalyst elutriation out of Regen. by adding a cyclone
- Ideally, disengagement zone (freeboard) keeps catalyst in reactor
- Pure oxygen is dangerous & expensive, but plausible



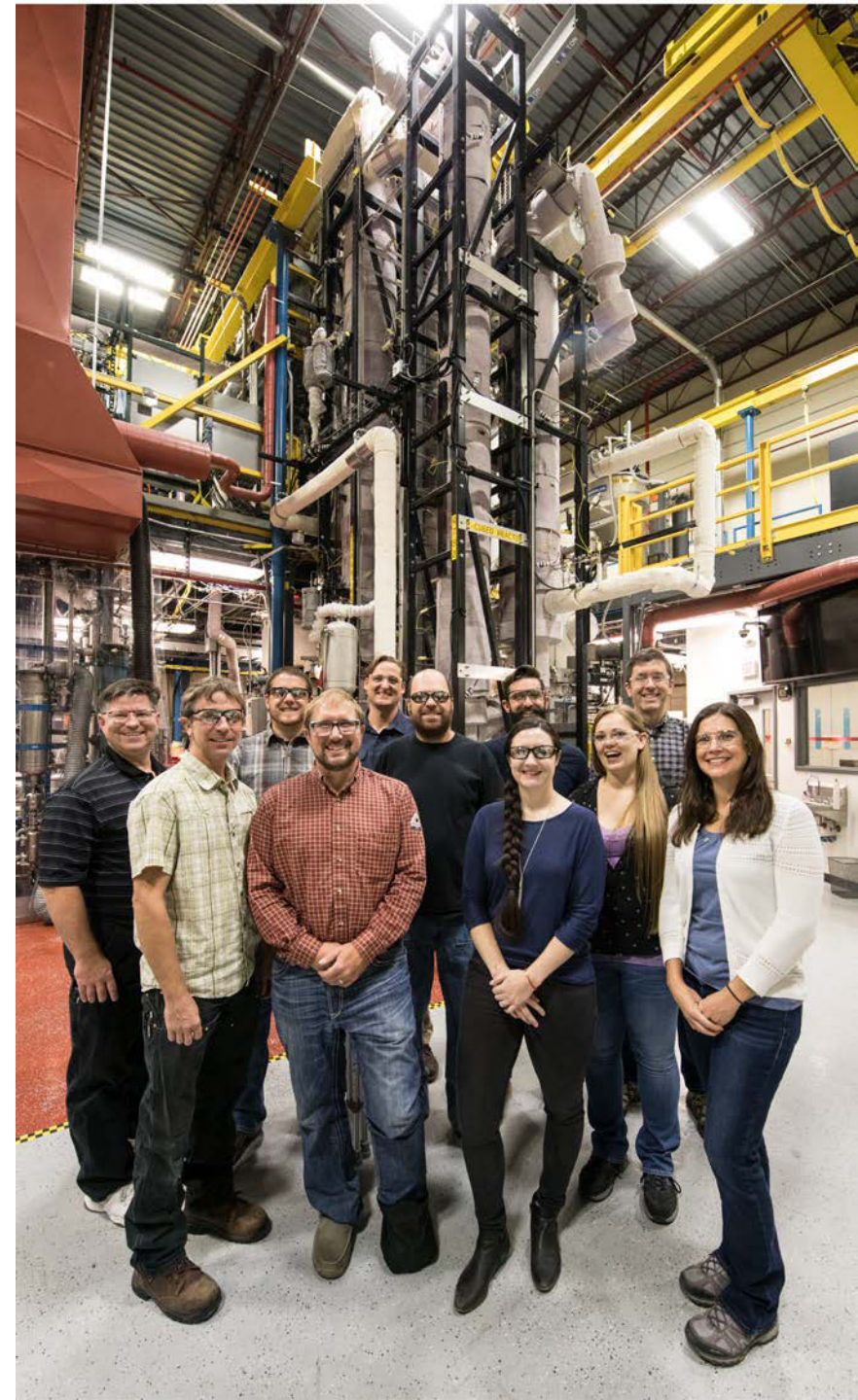
# Acknowledgements

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Xi Gao, Bill Rogers (NETL)

DOE Advanced Development & Optimization  
program (ADO)

10/9 @ 4pm Jessica Olstad (NREL)  
*Co-Processing Catalytic Fast Pyrolysis Oils with Vacuum Gas Oil in a  
Davison Circulating Riser – Upgrading Track*



# Questions?

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**[www.nrel.gov](http://www.nrel.gov)**

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