INTEGRATED LAB SOLUTIONS

Designing the System for Your Chemistry

Company Presentation

Dr. Anton Nagy

Dr. Andre Unger

www.integratedlabsolutions.com

Business Areas

Materials testing systems for
Chemical, Pharma and
Chemical Engineering R&D and QC

Core Competences

- Design of automated, turn-key systems
- Fixed bed reactors from lab to pilot scale
- gas, liquid, and multiphase flow systems
- Autoclave test reactors
  - Ranging from 2ml-100L
  - Continuous reactors (CSTR)
- High throughput and parallel units

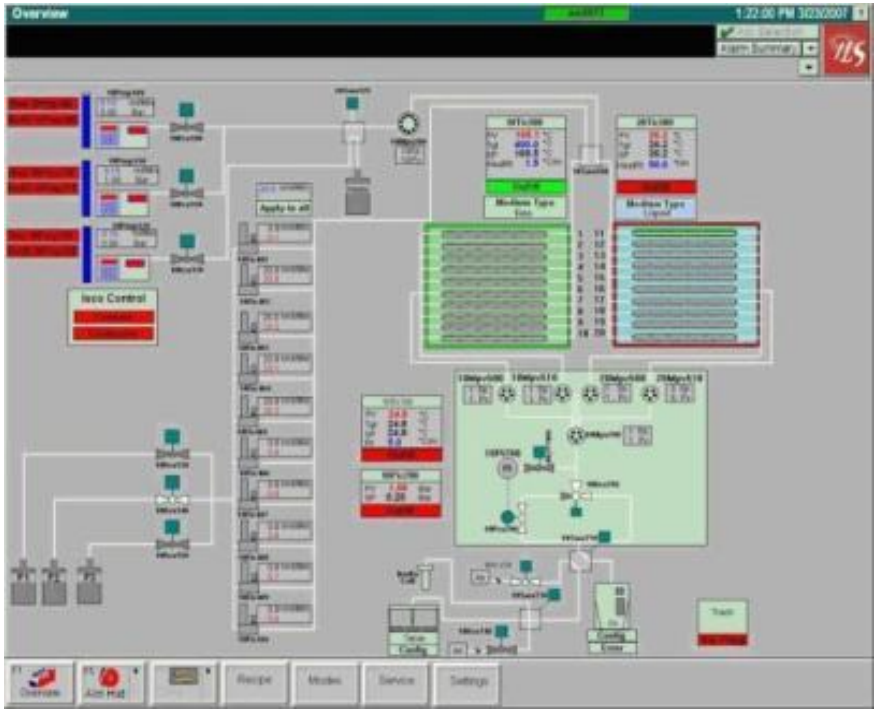
Our Philosphy

- Design the system to fit the process, not the other way around
- EVERY unit is custom made
- Close cooperation with customer

Process Control Philosphy

Not Re-inventing the wheel

- 100% Industrial Process Control System
- PLC & I/O Based
  - Systems used include
    - Eurotherm, iFix, Honeywell, Siemens, Delta V
    - *Can be adapted to existing in-house standards*
- Integration of analytical equipment & results
- Recipe Control



Screenshot from VUB HT Adsorption Unit

INTEGRATED LAB SOLUTIONS

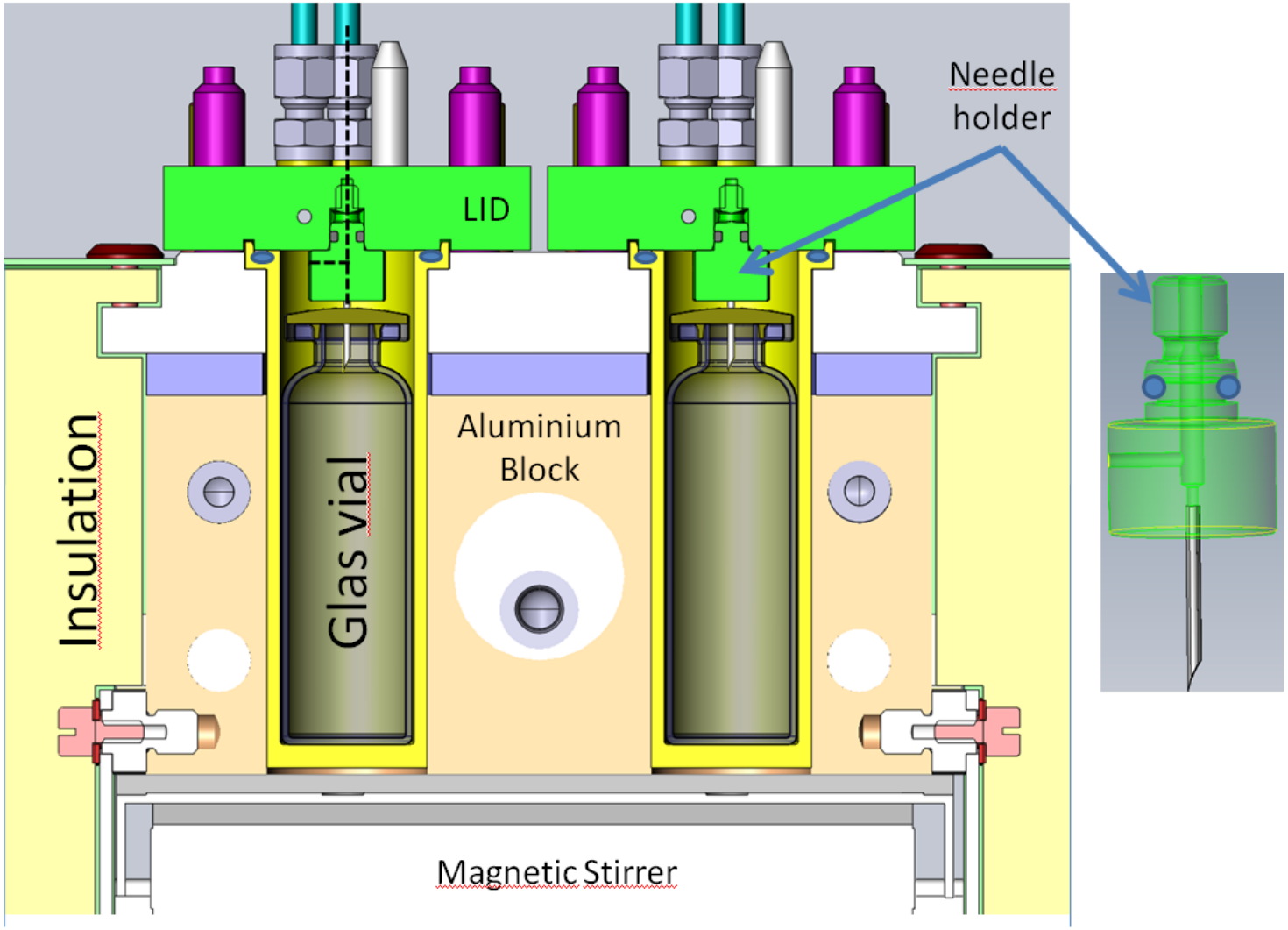
Batch Systems

20-Parallel PE Catalyst Testing

High Pressure Asymmetric Hydrogenation Unit

- 20 Reactors
- Manual control, multiple temperature and pressure zones
- Optional automation
- 100 bar, 180°C
- Extremely robust, easy to learn, low maintenance

Section drawing Aluminium Block



Metallocene and Ziegler-Natta Catalyzed Results

Left

metallocene catalyst

Remark:
Pink color is characteristic of the active catalyst meaning that the catalyst is still « alive »

Right

Conventional Ziegler Natta catalyst

Remark:
Excellent particle morphology of polymer (see the difference of PE crystallization)

40 Parallel Hydroformylation Catalyst Testing

High Pressure Asymmetric Hydrogenation Unit

- ´40 Reactors
- Manual control, multiple temperature and pressure zones
- 100 bar, 200°C
- Extremely robust
- Easy to learn
- low maintenance

Fully-Automated 96-Parallel Batch Testing Unit (Designed for Asymmetric hydrogenations)

96 parallel batch system

- 100 bar, 200°C
- Uses GC vials as reaction vessels
- Air/water sensitive chemistry without a glovebox
- Used for hydrogenations, oxidations, polyol synthesis, aldol condensation etc…

48 Parallel Manual Asymmetric Hydrogenation Catalyst Screening

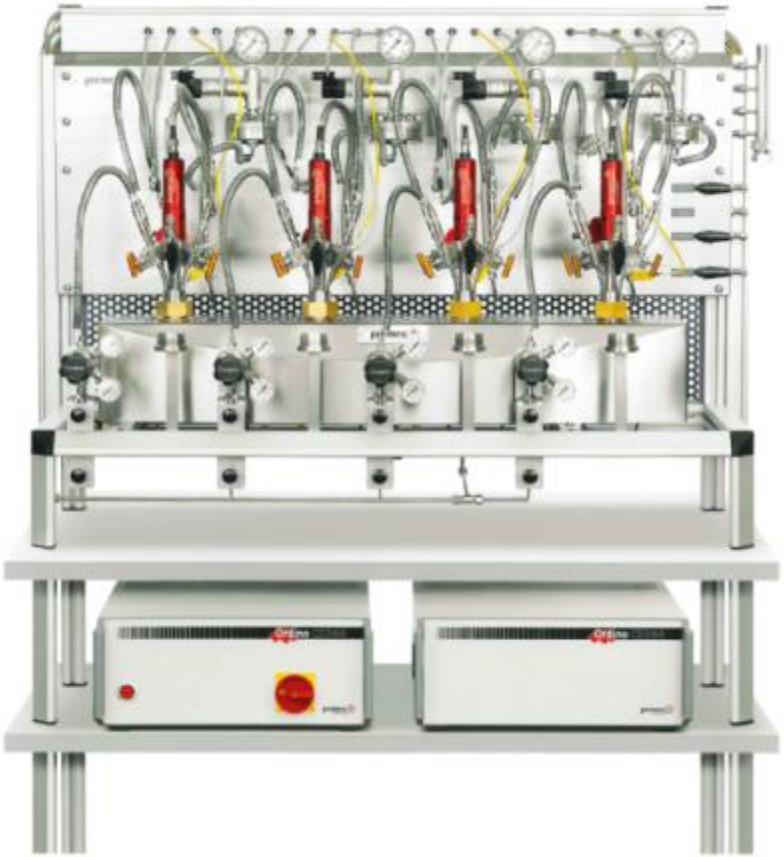
Asymmetric Hydrogenation Unit

- Manual control, multiple temperature and pressure zones
- 48 reactors
- Optional automation
- 30 bar, 200°C
- Extremely robust, easy to learn, low maintenance
- Air/moisture sensitive catalyst testing

Process-Optimization Catalyst Testing (Olefin oligomerization)

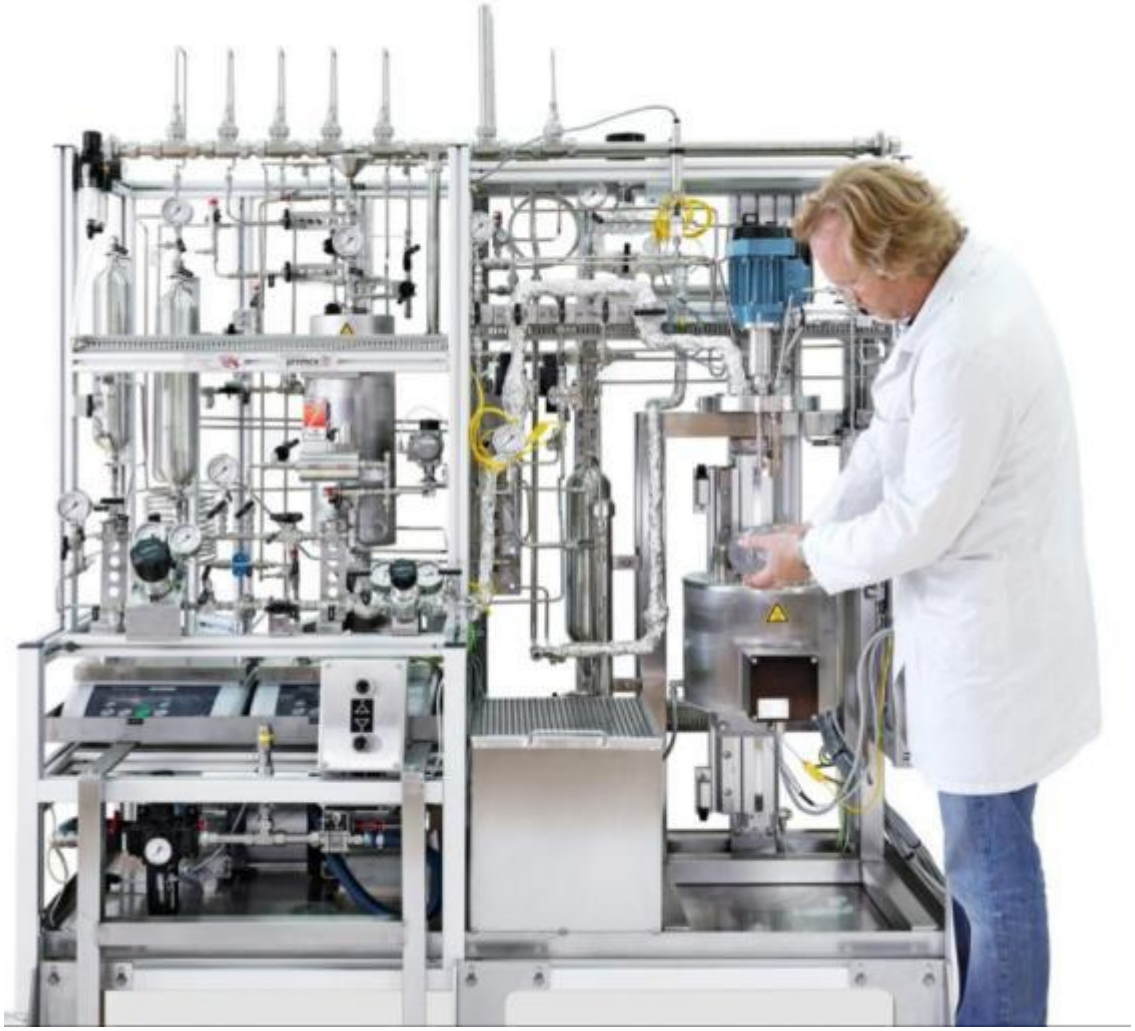
4 Independent Autoclaves;

- 300°C, 100-200 bar
- Independent heating and cooling for exotherm control
- Fully automated with recipe control
- Online gas uptake measurement, multiple flow controllers to extend range
- Optional liquid sampling during reaction



Fully-Automated Biodiesel Batch Mini-Pilot Unit

- 1 Liter
- 350 Bar
- 350°C
- Fully Automated
- Hydrogen Compressor
- 2 Liquids Dosed
- Supercritical Ammonia Reaction
- Automated on-line liquid sampling



Hydrothermal Synthesis Micro-Pilot Unit

- 1 Liter
- 200 Bar
- 550°C
- Fully Automated
- Cascaded Reactor Design
- Liquid dosing via HPLC
- Gas dosing via MFC
- High-Temperature/High-Pressure pH Measurement (T>150°C)

Supercritical CO_2 Oxidation Workstation

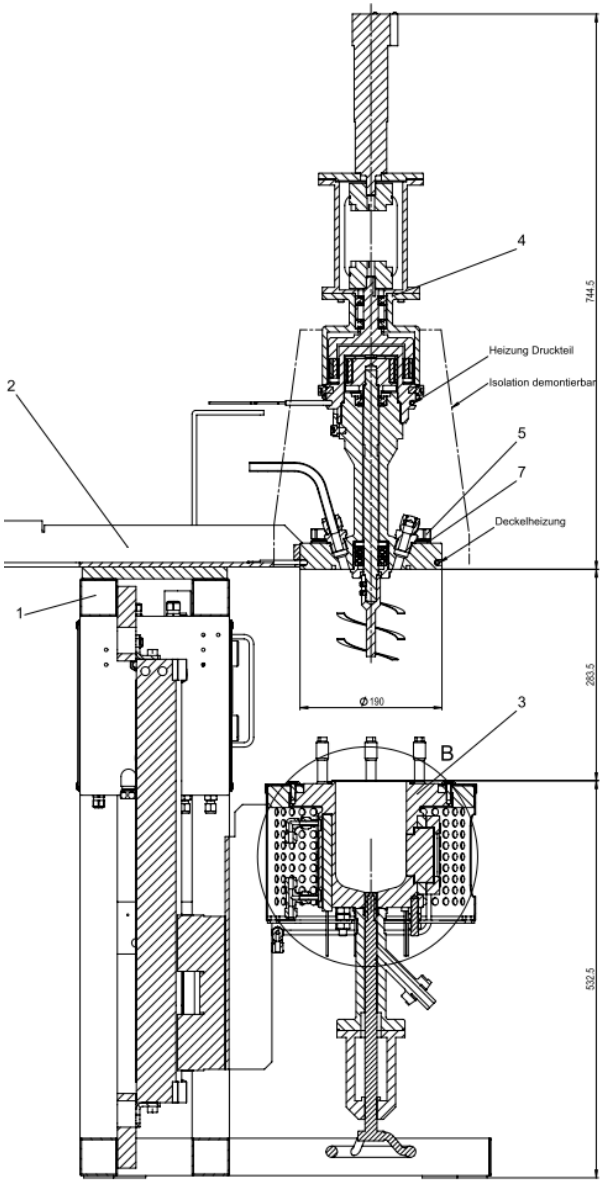
KU Leuven

Turnkey unit, combining;

- 24-Parallel Slurry-phase screening reactor
- Visualisation Reactor
- 10-parallel Hydrothermal zeolite synthesis reactor
- Fixed-Bed lifetime test unit

Polyamide (PA66, PA11) Synthesis Reactor

- 1 Liter
- 60 bar
- 360°C (also inside Mag drive)
- Sapphire Glas window
- Very precise Temperature (+/- 0.5°C) and Pressure control (+/- 0,05bar) over the whole range
- Liquid dosing with HPLC pump
- Torque sensor
- Special Bottom drain valve
- Water collection system with condenser and balance


4
Heizung Druckteil
Isolation demontierbar
2
5
7
Deckelheizung
1
Ø 190
3
B
744.5
283.5
532.5

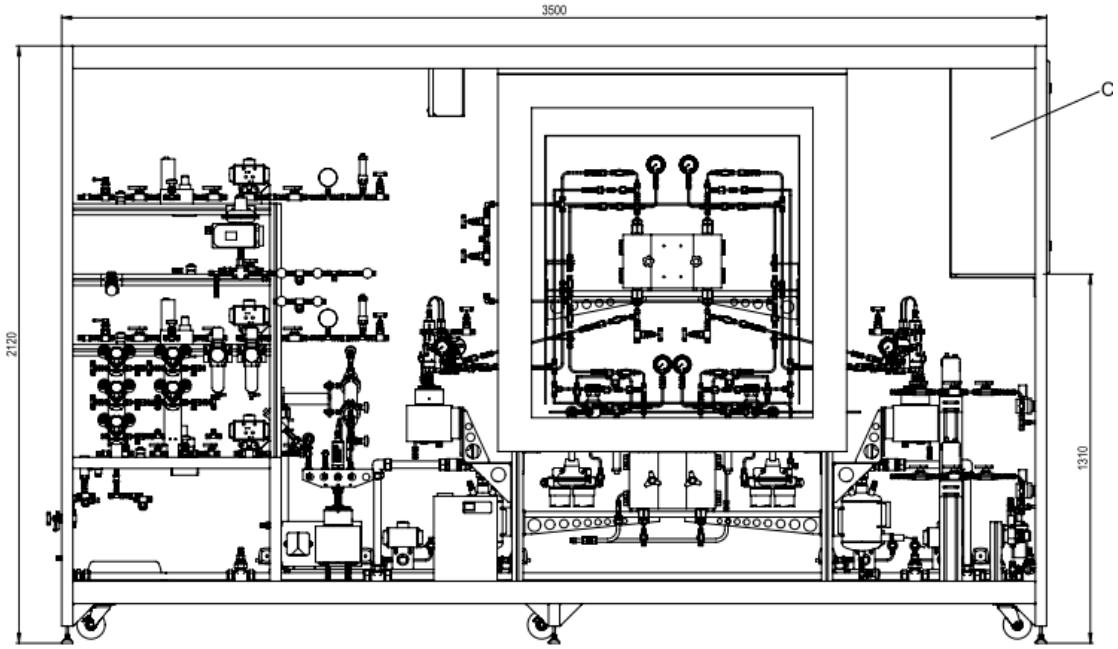
Spherical Reactor for Slurry-Phase PP Synthesis

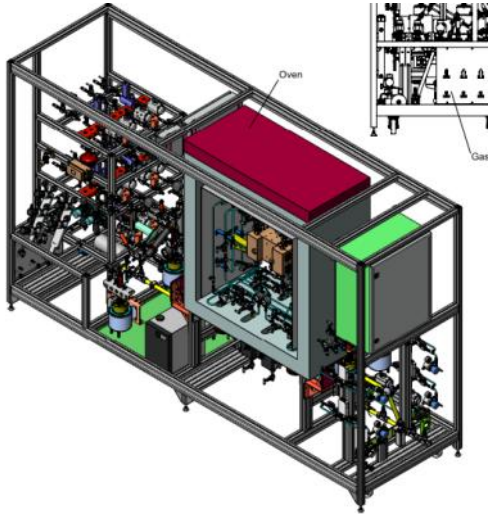
- 8 Liter
- Steam/water heating-cooling
- Kinetic-Quality Data
- Custom Stirrer Design
- ATEX-Explosion Proof
- TüV approved

Pilot Plants

- 100 liter Autoclave
- 100 Bar
- 200°C
- *Atex Zone II* Certified
- Hastelloy® C
- Overpressure nitrogen purge system from Pepperl & Fuchs

Medium-Throughput Tar Sand Hydrotreating





1 Pre-Sulpphiding Reactor
4 Parallel Hydrotreating reactors
Max T=550oC
Max P=250 Bar
Gas uptake measurement
Hot and cold G/L separators

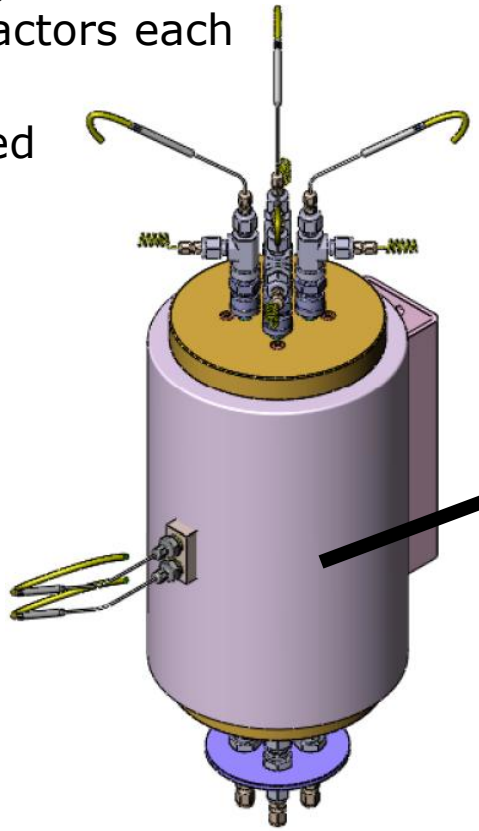
INTEGRATED LAB SOLUTIONS

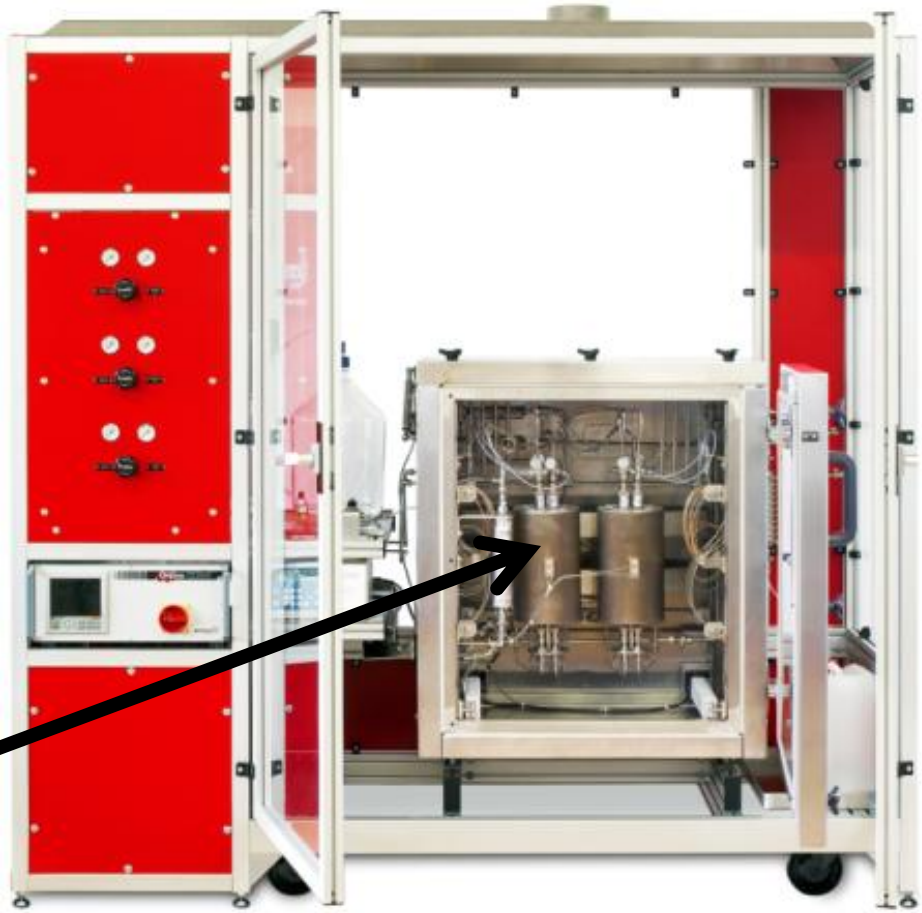
Fixed-Bed Systems

Parallel 8x Fixed-Bed Reactor

Catalyst Pretreatment & Testing

- 550°C
- Atmospheric Pressure
- 2 Heating Blocks for 4 Quarz reactors each
- Partially Automated





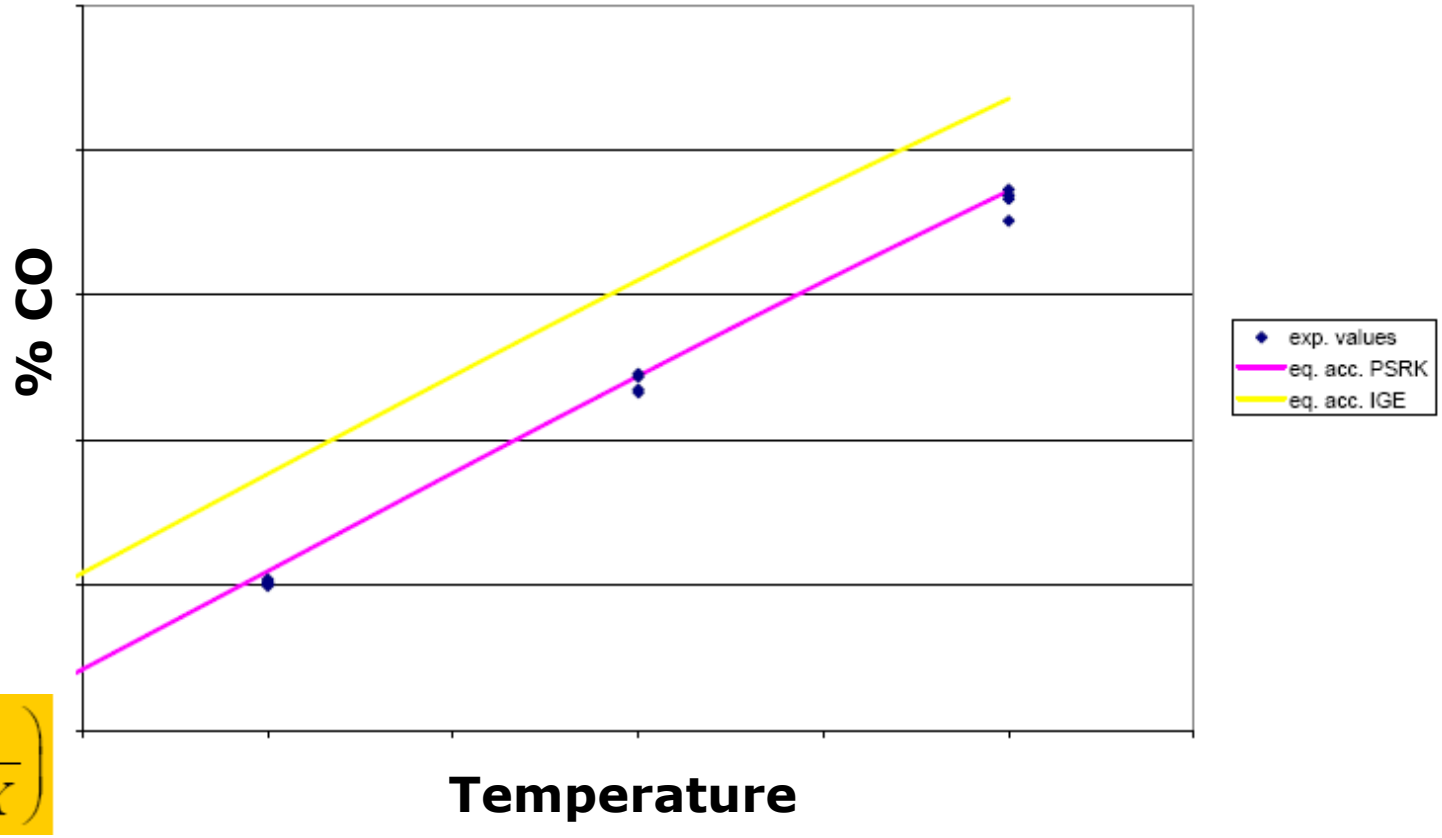
Parallel Fixed-Bed Technology

- Specifications
  - 550°C, 100 bar
  - 10+1 Reactors
  - Reactor i.d. 2 or 4mm
  - Temperature accuracy +/- 0.5°C
  - Independent temperature control
  - Same pressure
  - On-line micro GC
  - Recipe Control

+/-0.5 °C Isothermicity (Experimentally Verified by Equilibrium Conversion)

$$Pe > 8n \ln\left(\frac{1}{1-X}\right)$$

Sie, AIChE-J. 1996

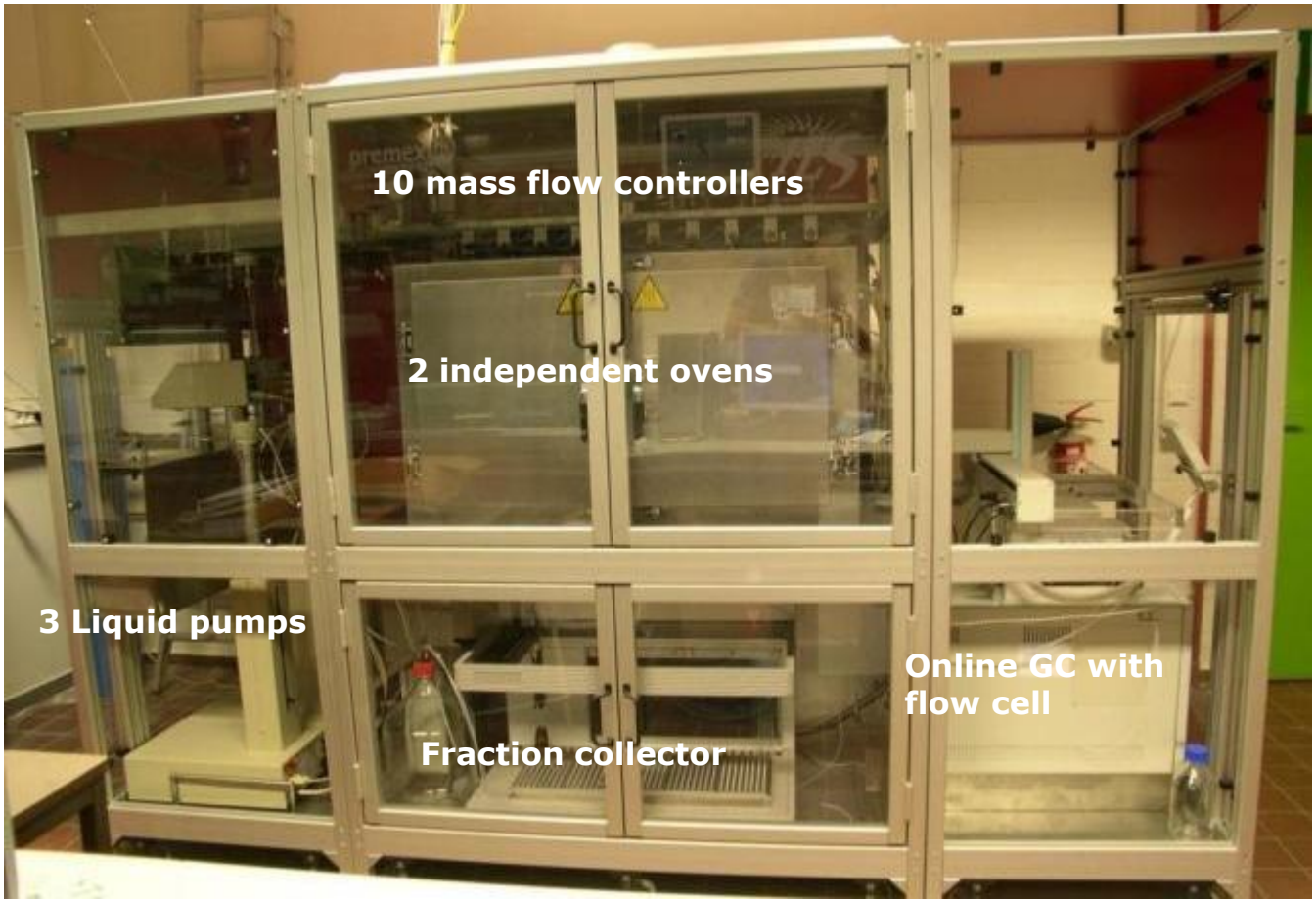


Parallel Olefin monomer adsorption testing

Designed for VU Brussels high throughput adsorption studies

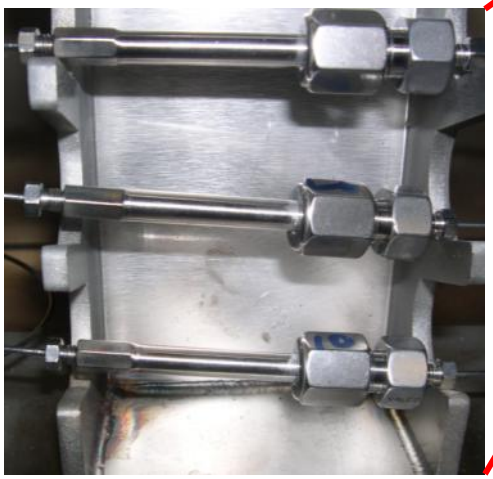
- 20 reactors
- 250 bar, 450°C
- Fully automated, work in fast sequential mode
- Automated sample collection and fraction collection
- Integrated fast GC
- Intrinsic diffusion kinetics (0,1 micron zeolites!)

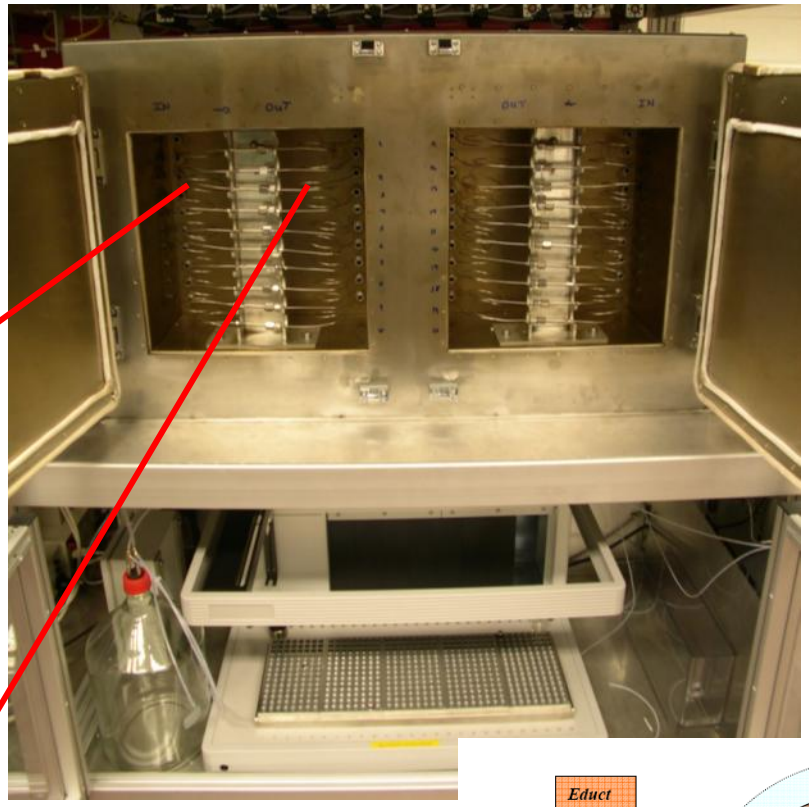
Thanks to:
dr. ir. Joeri Denayer
dr. ir. Eileen Dejaegere
Prof. Gino Baron
Pierre Martin



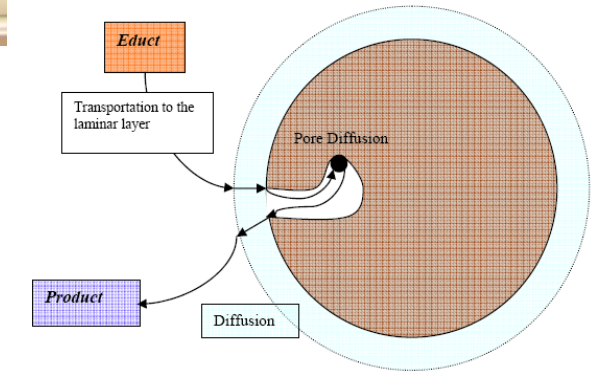
Packed Microreactors (disposable)

- 2mm i.d.
- 5mc length
- 0,1-1micron particle size

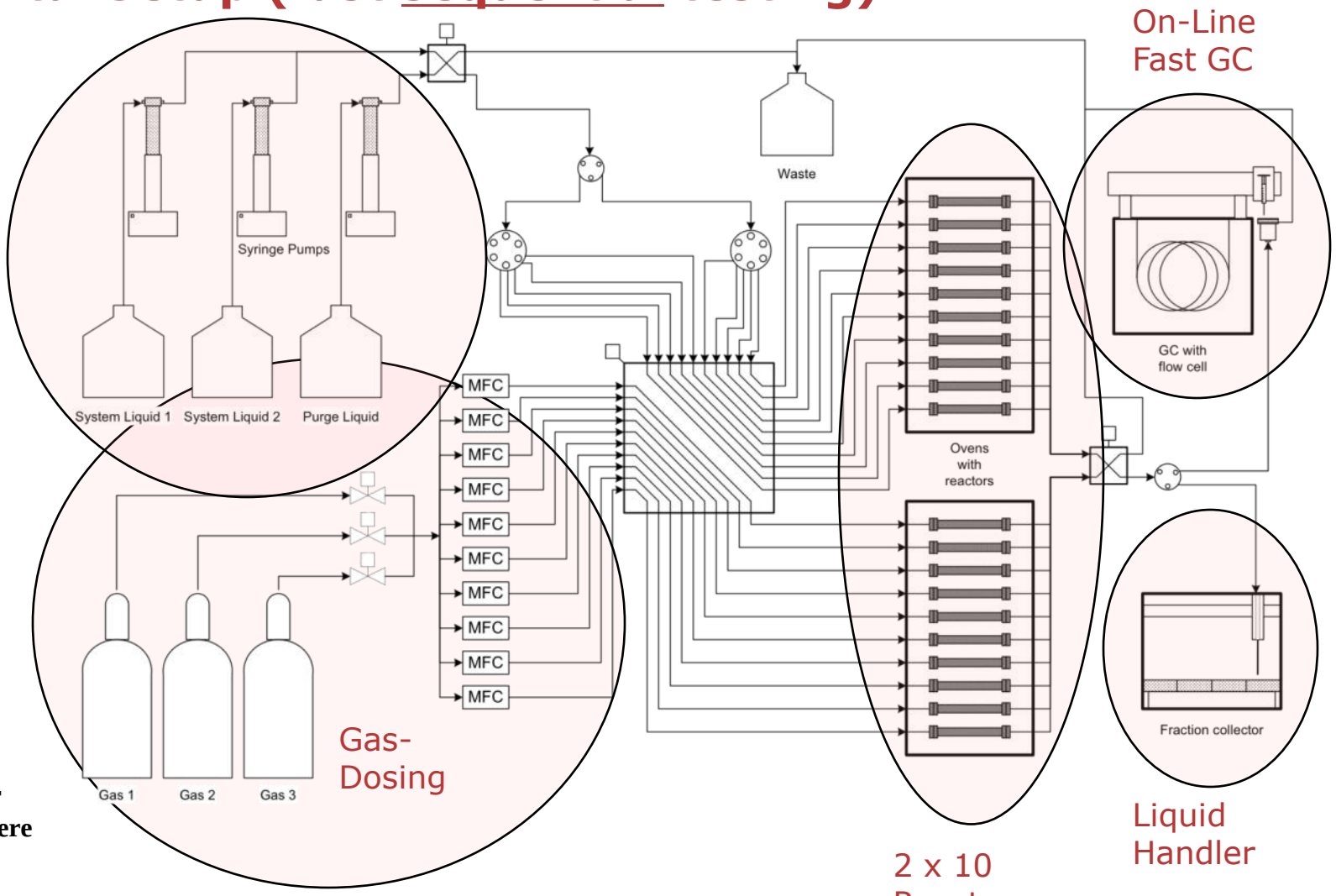




Small particle size (0.1micron) allows study of intrinsic diffusion kinetics



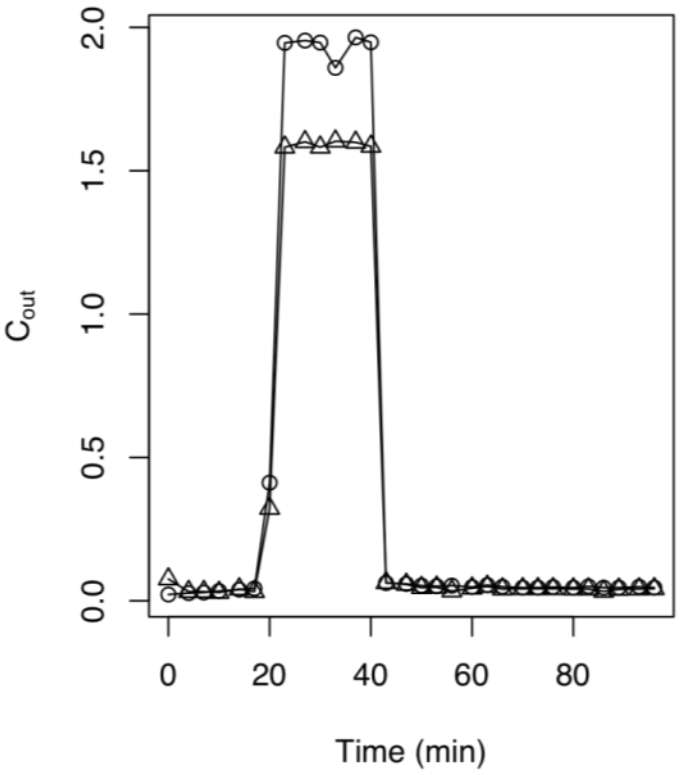
Experimental Setup (fast sequential testing)



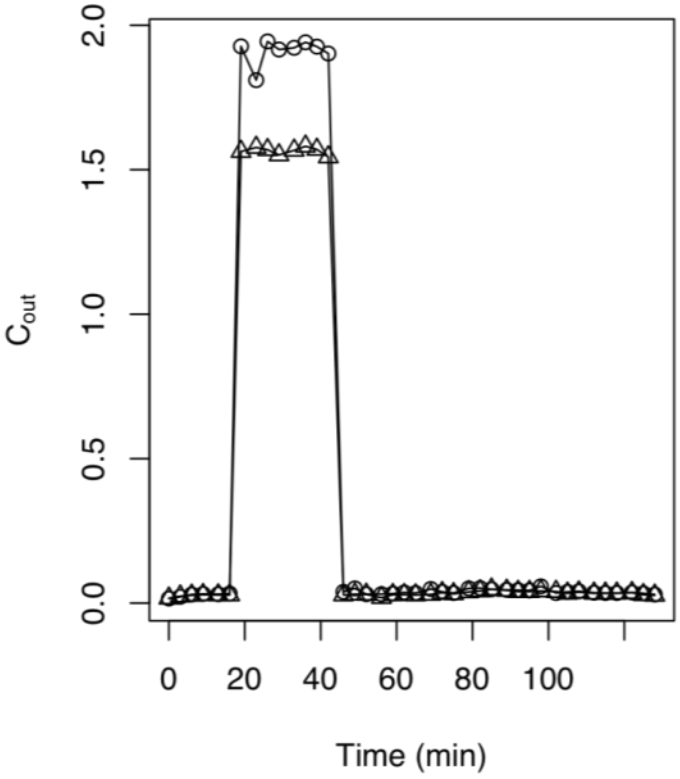
Thanks to:
dr. ir. Joeri Denayer
dr. ir. Eileen Dejaegere
Prof. Gino Baron
Pierre Martin

Liquid phase adsorption: Blank experiment (Low dead-volume=narrow RTD)

feed: 0.5 ml/min

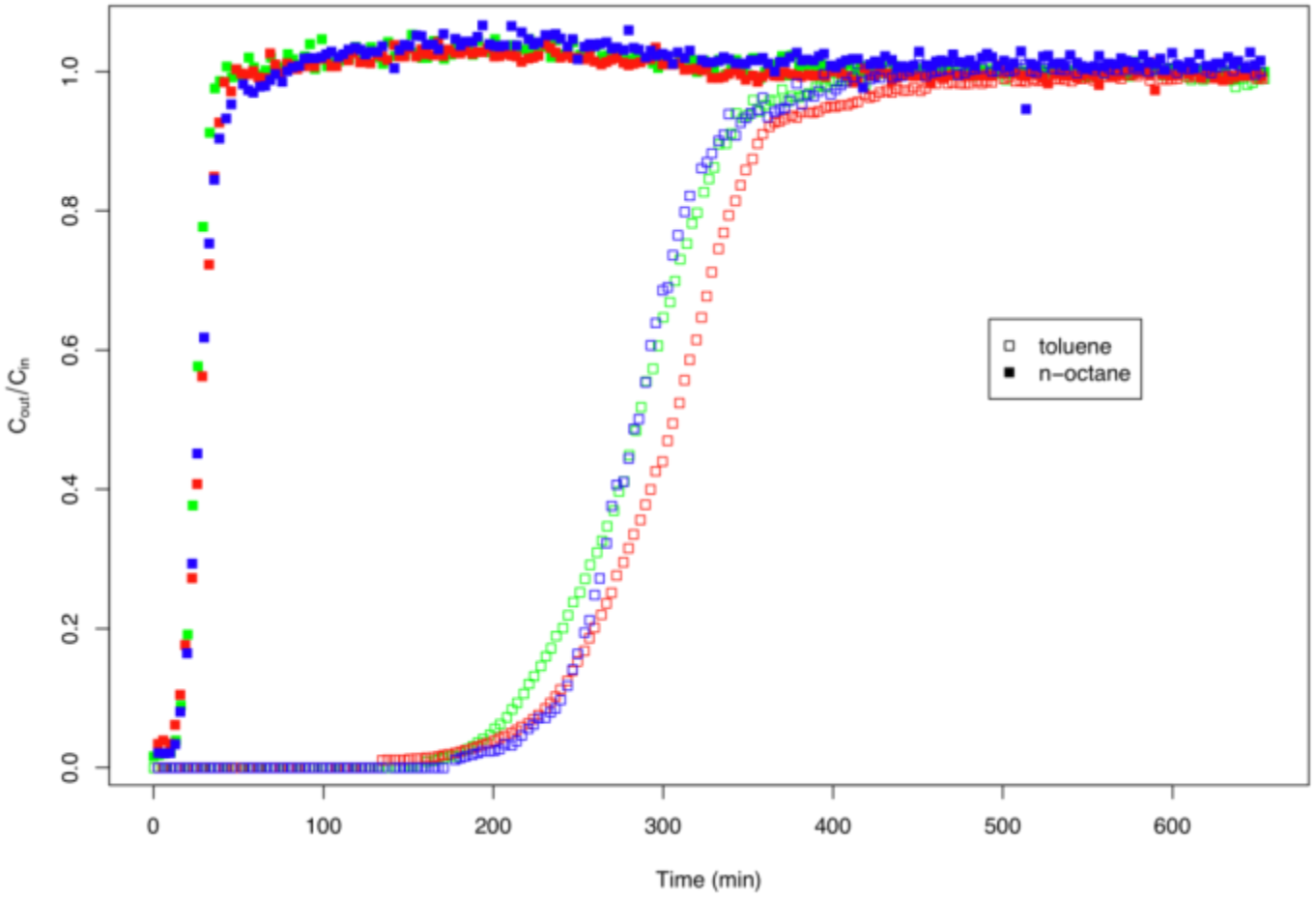


feed: 1.0 ml/min



Thanks to:
dr. ir. Joeri Denayer
dr. ir. Eileen Dejaegere
Prof. Gino Baron
Pierre Martin

Liquid phase adsorption: 2 mol% nC8/tol in isoC8 on NaX



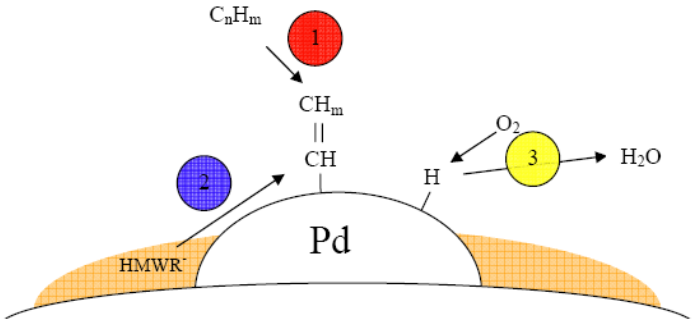
Intelligent sampling with feedback from analytical device

Thanks to:
dr. ir. Joeri Denayer
dr. ir. Eileen Dejaegere
Prof. Gino Baron
Pierre Martin

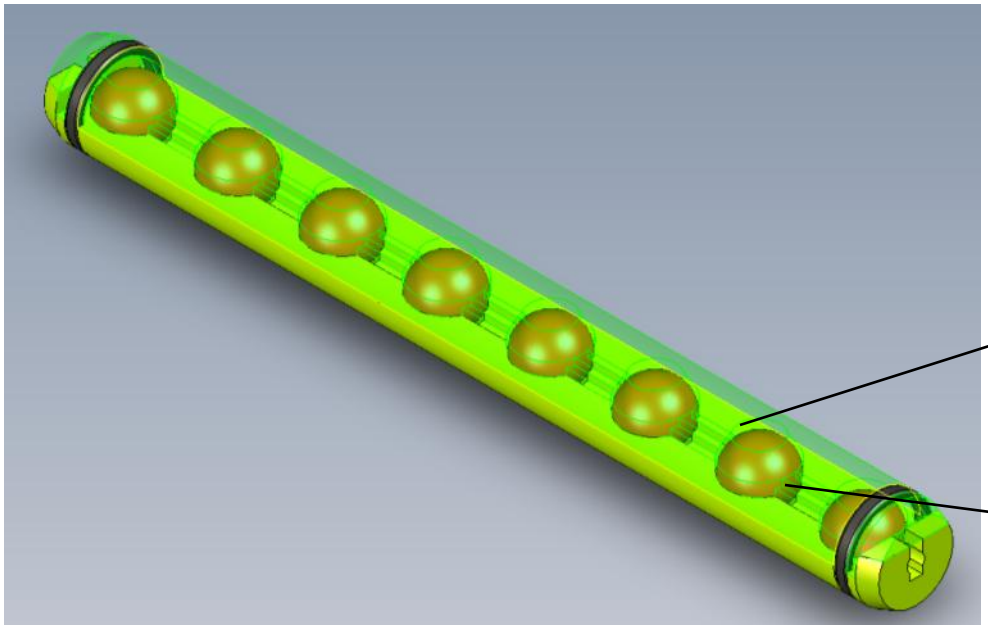
Temkin Reactor -Alternative approach to separating hydrodynamics & kinetics

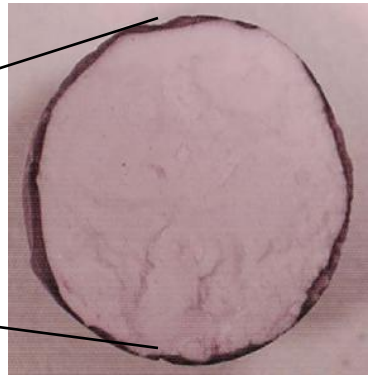
$$HMWR + \tfrac{1}{2}O_2 + C_nH_m \xrightarrow{PdAu} Product + H_2O$$

$$C_nH_m + O_2 \xrightarrow{PdAu} CO_2 + H_2O$$



Ideal for egghell-type catalysts (no crushing required)





Vinyl Acetate Monomer Synthesis Testing on Real Impregnated, Shaped Catalysts

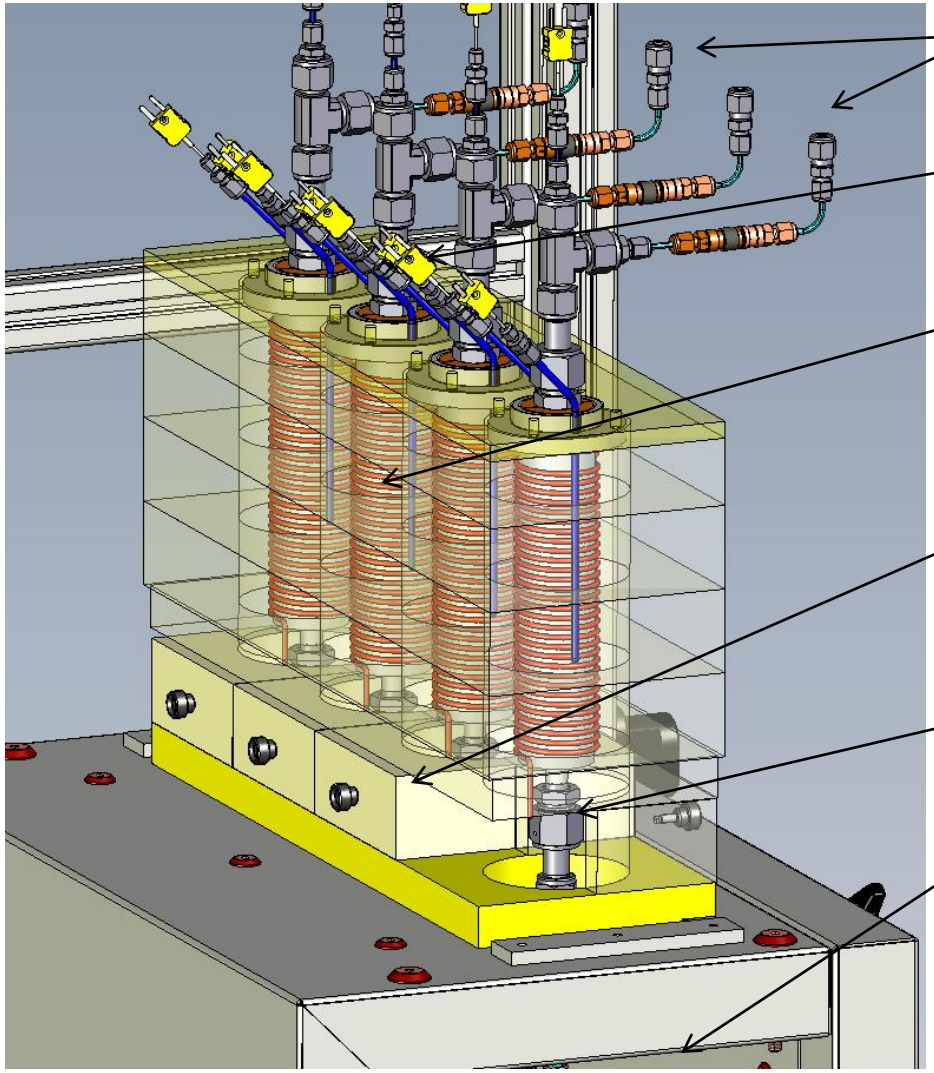
Propane & C_3 Oxidation in a Simulated Fixed-bed Temkin Reactor

- Corrosion resistant materials, PEEK®, Hastelloy®C276
- 20 bar, 500°C
- Online GC and IR
- 7 fold
- Extrudate (egg-shell) catalyst testing

4-Parallel Process-Optimization Fixed-Bed Reactor for Cobalt/Alumina Fischer-Tropsch

Powder Testing

- Fixed bed
- 2 to 7mm i.d. reactors
- 550°C max T
- In-Situ Reduction
- CO/H_2 gas alarms
- On-line GC
- COMPLETELY independent reactors (T,P, flows)



Educt inlets

Thermo-couples

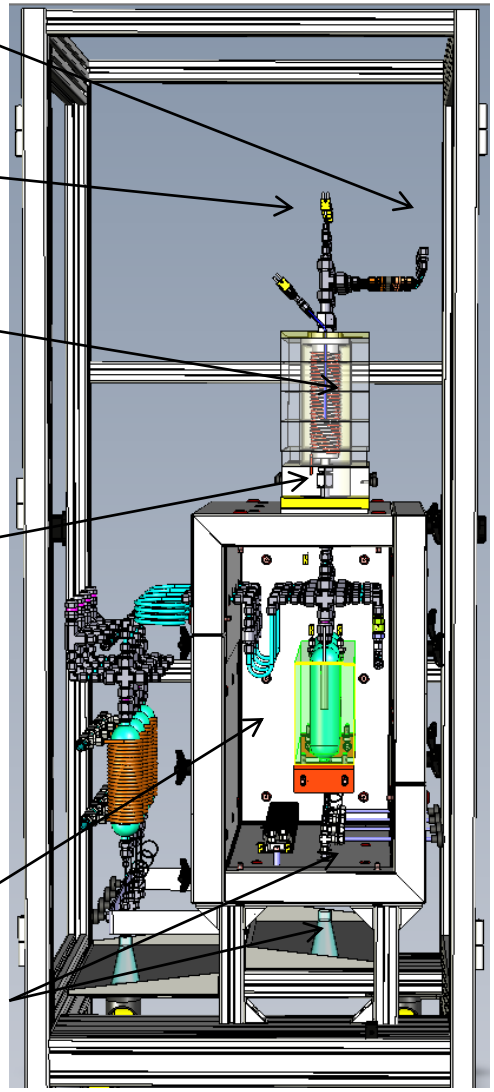
Tubular reactors

Insulation blocks

VCR connectors

Product oven

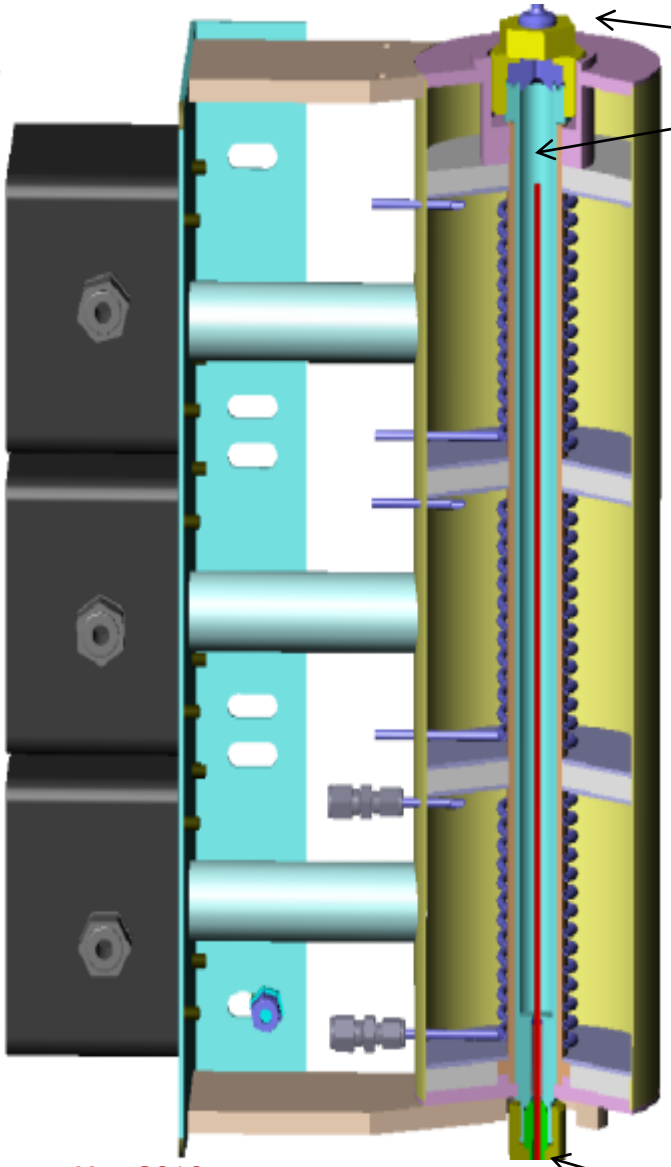
Liquid Outlets



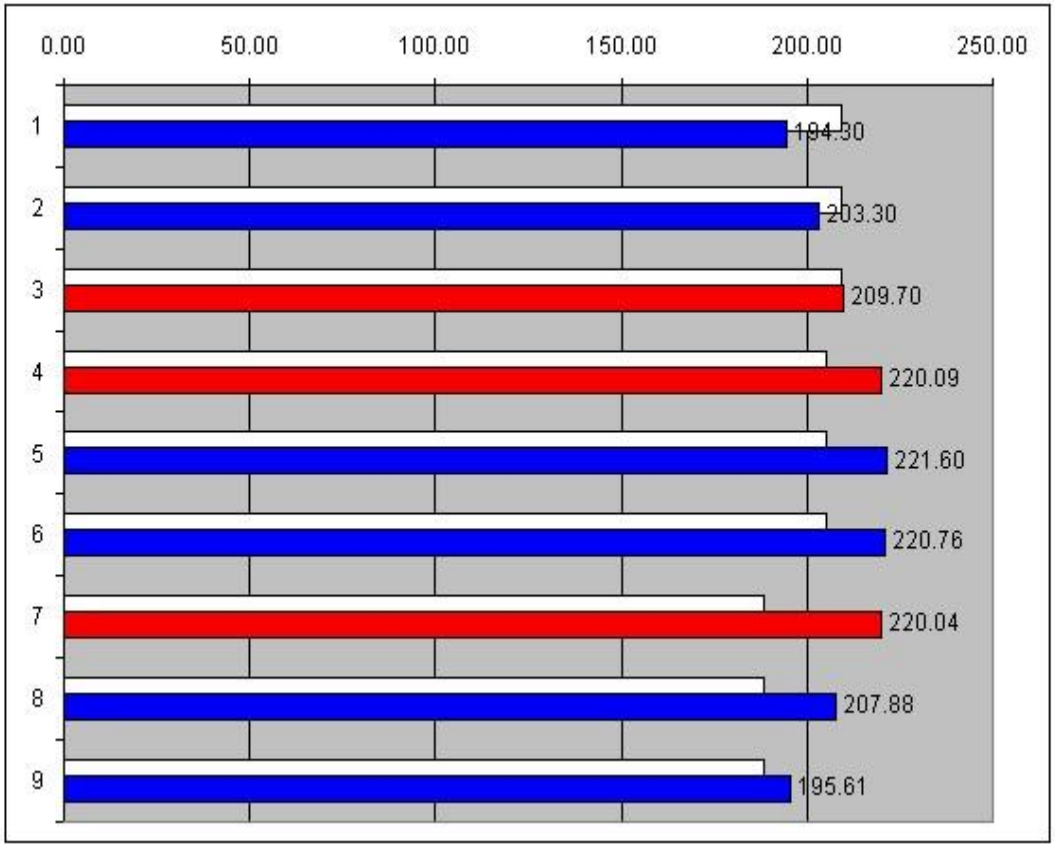
Fischer Tropsch Micropilot Unit

Extrudate Testing

- Fixed bed
- 5mm or 20mm reactor i.d.
- 3-Zone Heating and cooling of
- block, 3°C exotherm at 70%
- Conversion
- Automated 2-stage product
- Collection
- Integrated online analytics



20mm O.D. Reactor with 3 heating/cooling zones



Excellent T-Ctrl at FT-X>60%
T_{R_SP}=220°C

Difficulty Testing Fischer-Tropsch Catalysts in Fixed-Bed Reactors

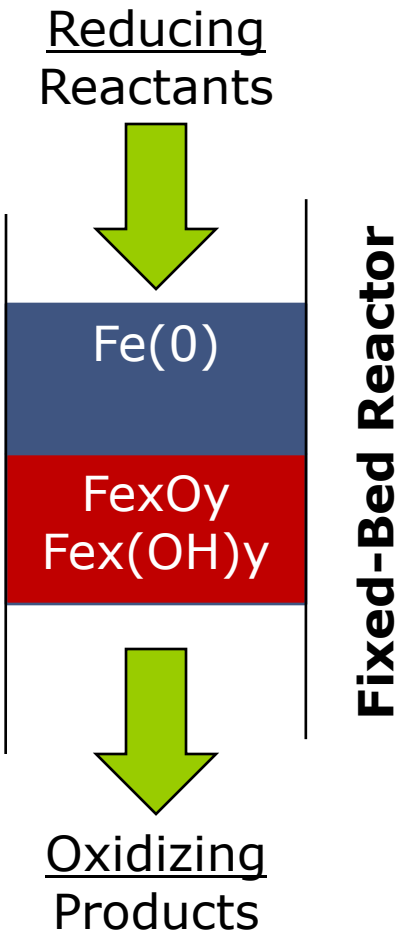
$$(2n+1)H_2 + nCO \rightarrow C_nH_{(2n+2)} + nH_2O$$

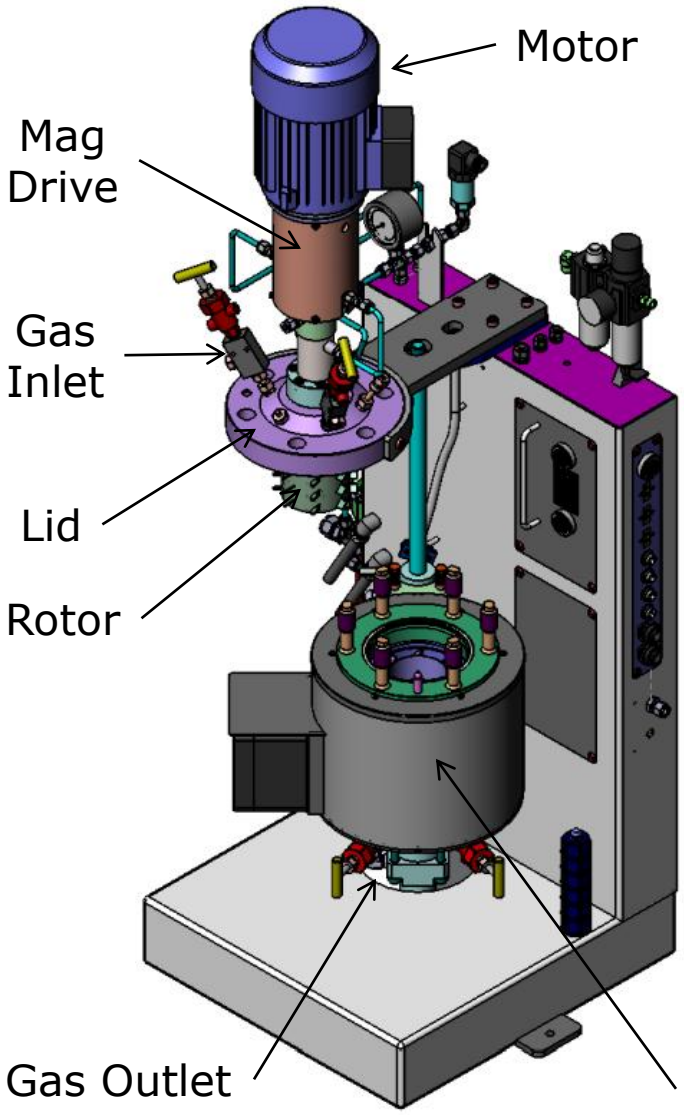
Catalyst oxidation state is a function of redox enviroment and varies over the length of the catalyst bed!

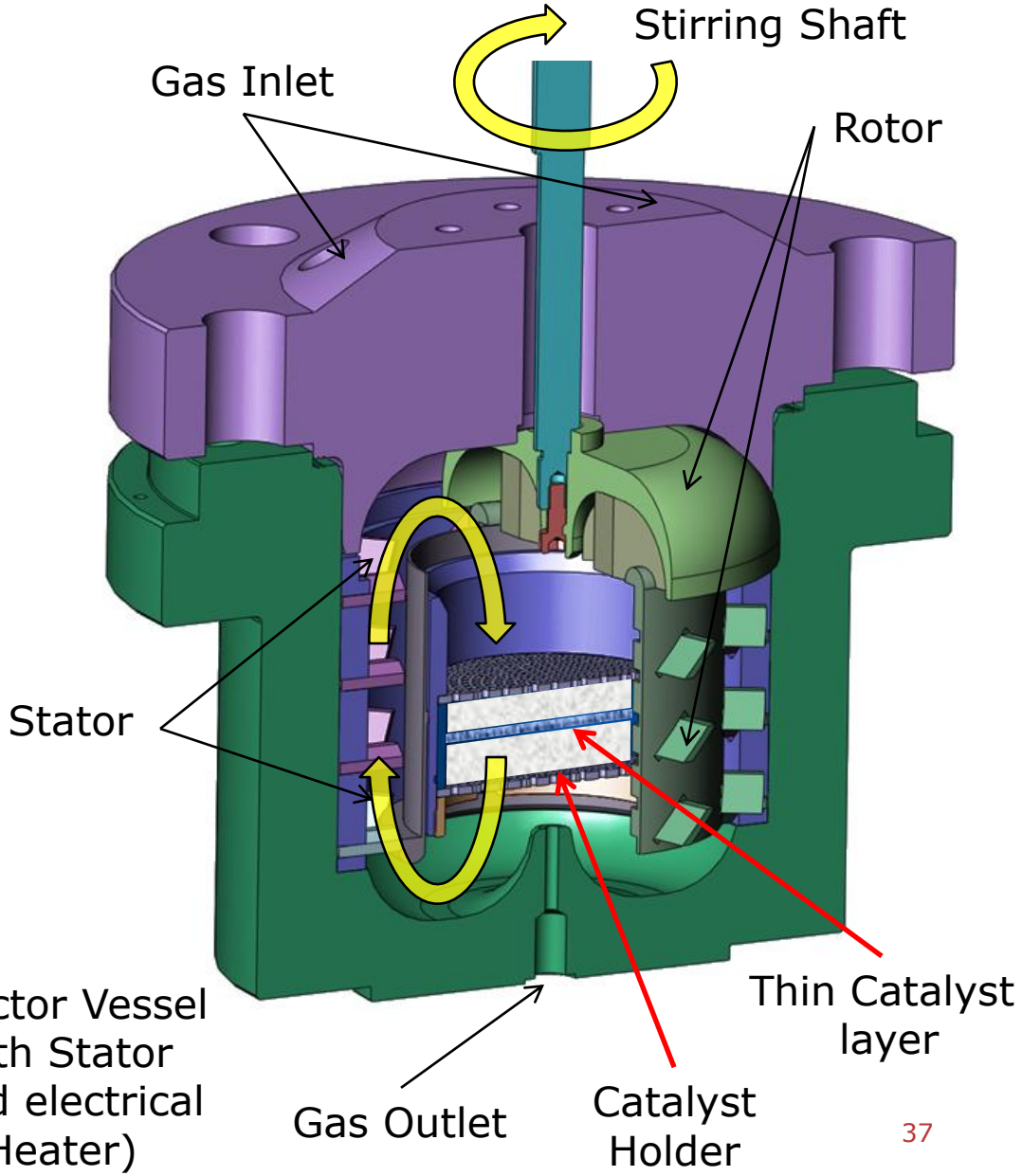
Solutions

-Differential Reactor Operaiton

-Gradientless Internal Recycle Reactor




Motor
Mag Drive
Gas Inlet
Lid
Rotor
Gas Outlet
Reactor Vessel with Stator (and electrical Heater)


Stirring Shaft
Gas Inlet
Rotor
Stator
Thin Catalyst layer
Gas Outlet
Catalyst Holder

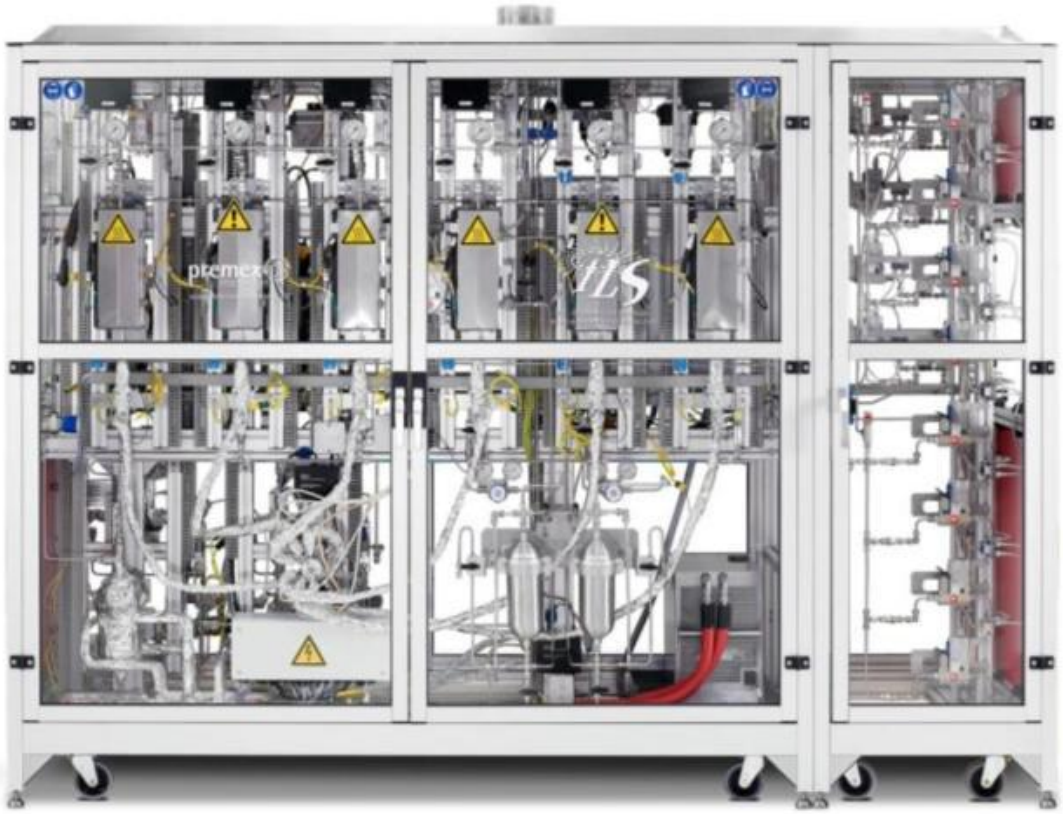
Fixed-Bed Internal-Recycle CSTR for Kinetic Studies of Heterogeneous Catalysts

Continuous reactors for kinetic studies

- 400°C, 300bar
- 700ml
- Filter design ensures blockage free operation, even with fine particles in use
- Gradientless internal recycle reactors, with stirrer speeds to 4500 rpm
- Online gas & liquid phase analysis
- Hybrid ceramic/metal bearings allow long-term operation (6 months!)

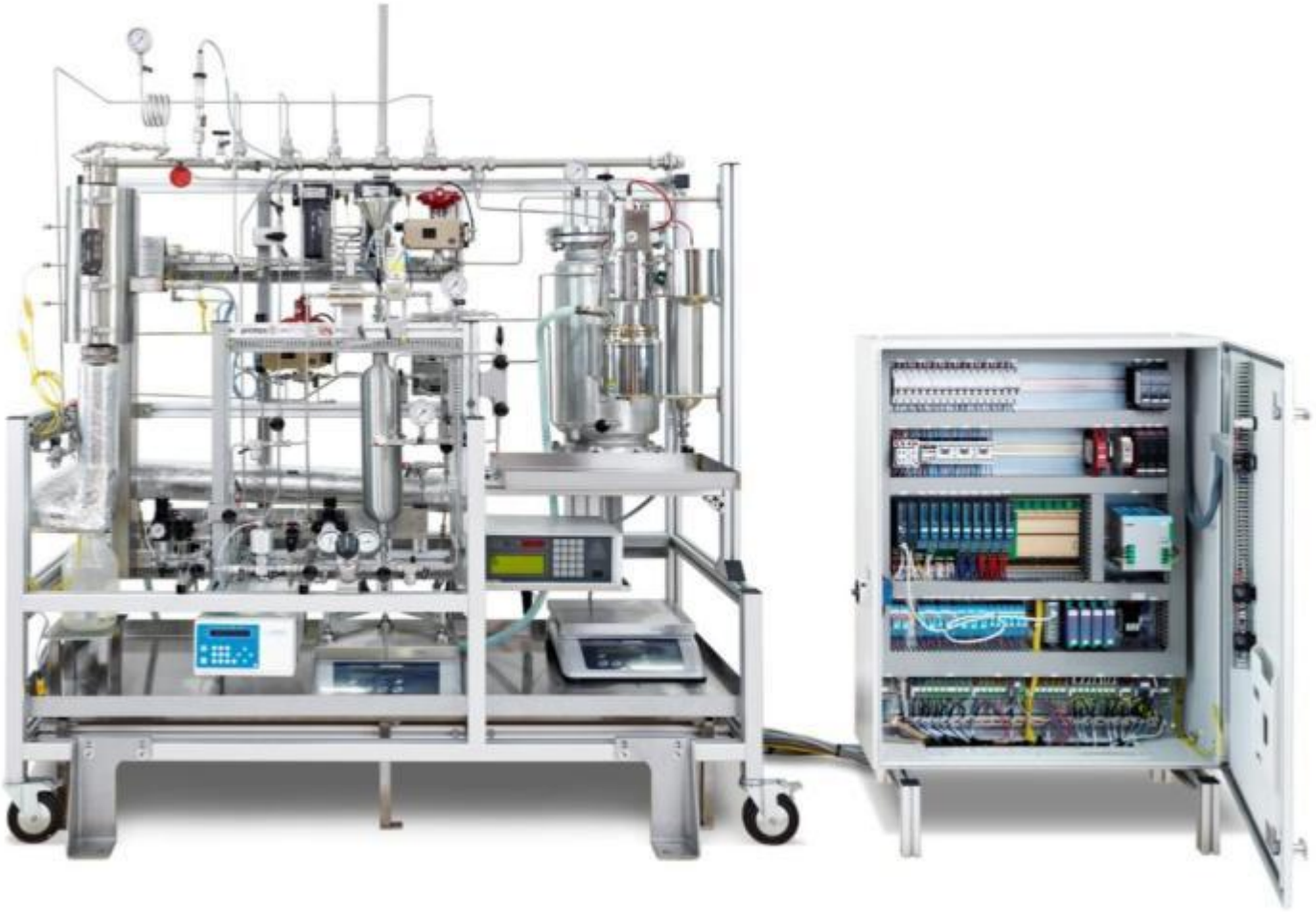
High-Temperatue (1000°C) 6+2 Parallel Screening Reactor

- 6 Screening Reactors (independent tempera-ture, same gas flows, 100-200mg)
- 2 Kinetic Testing Reactors (completely independent 1-2g)
- Two on-line GC's
- Fully Auotmated
- Automated liquid sampling
- 1000°C
- 10 bar

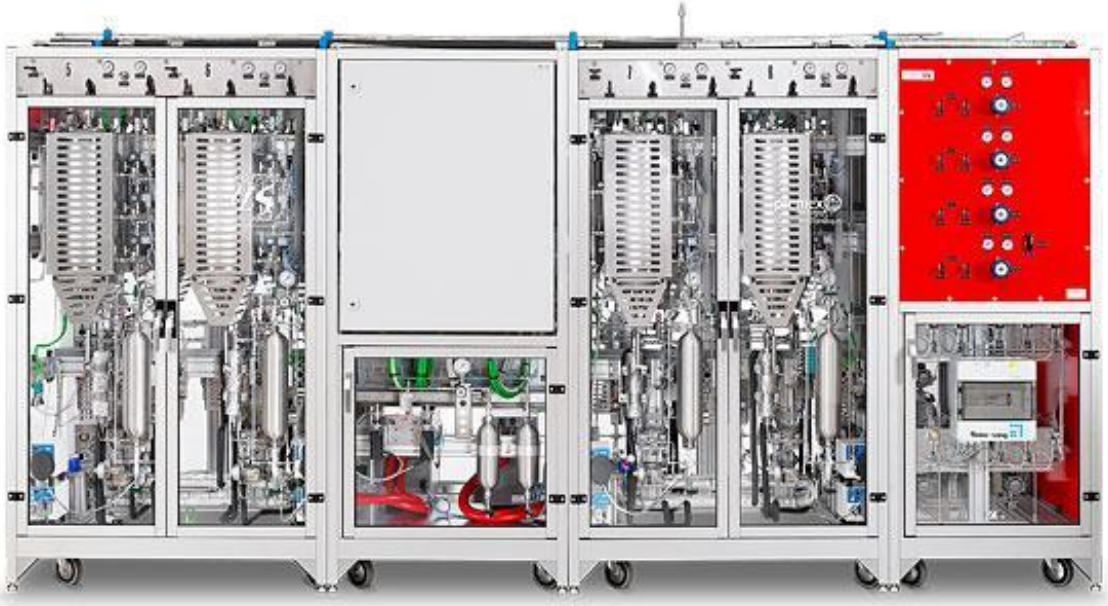


Fully-Automated Fixed-Bed Glycerine Upgrading Unit

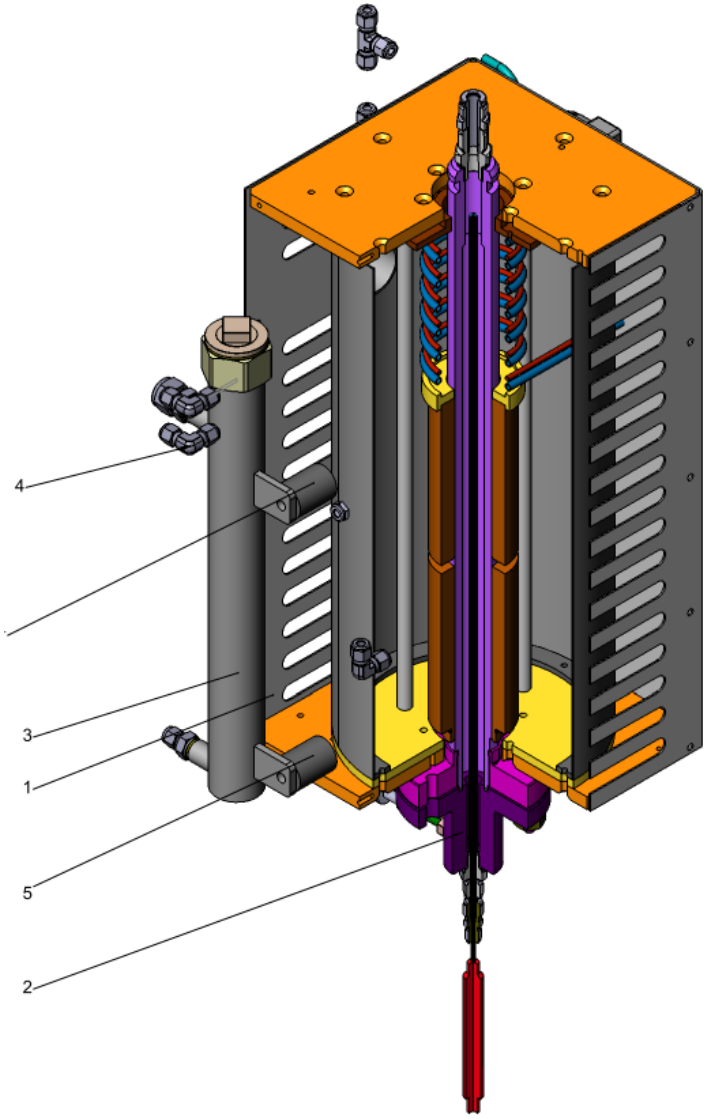
- 1-5g of Heterogeneous Catalyst
- 100 Bar
- 500°C
- Fully Automated
- 2 Liquids Dosed
- Automated mass-balance calculations



Fully-Automated, Modular 8-Parallel Trickle Flow Unit

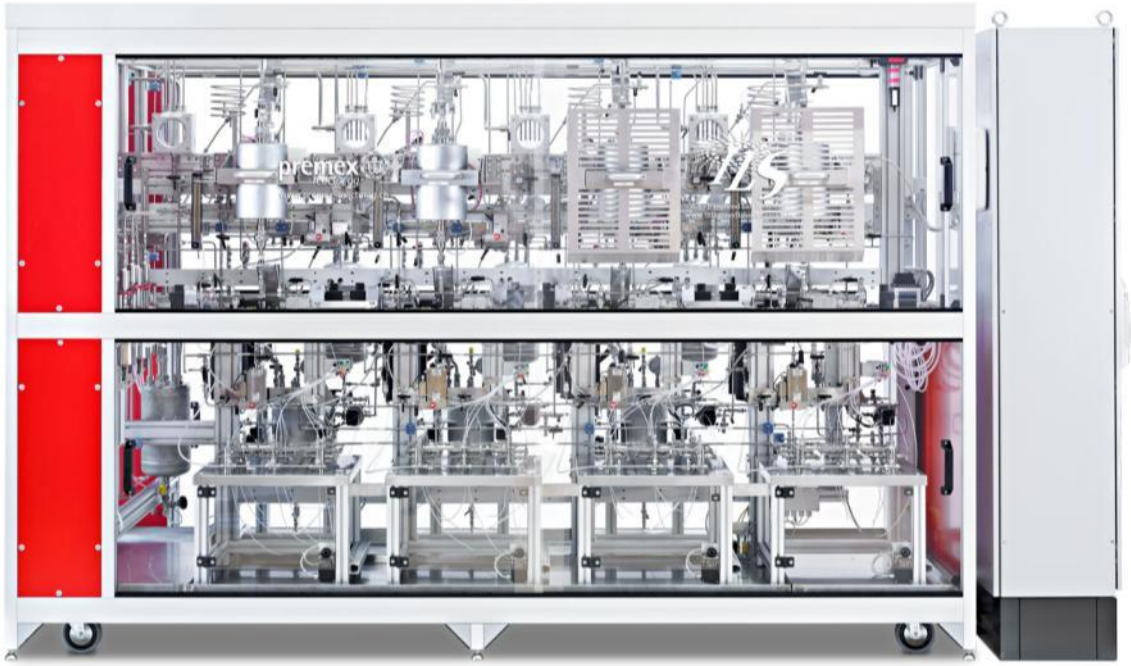


- 8 Fully Independent Reactors
- 3-Zone Heating AND water-cooling (for highly exothermic reactions) in each reactor
- Multipoint thermocouples for axial T-profiles
- Two liquids (one heated) dosed per reactor
- Co-feeding of inert or reactive gases
- 350 bar maximum pressure
- 550°C maximum temperature
- On-Line GC
- Fully-automated liquid sampling with liquid handler
- TüV Certified


4
3
1
5
2


1
8

4-Parallel Liquid/Liquid Reactor with In-Situ Catalyst Synthesis



- Works with air/moisture sensitive catalyst precursors
- 20 Bars
- 300°C
- 4 completely independent reactors
- Fully-automated liquid sampling
- Automated filter switching
- Fully-automated

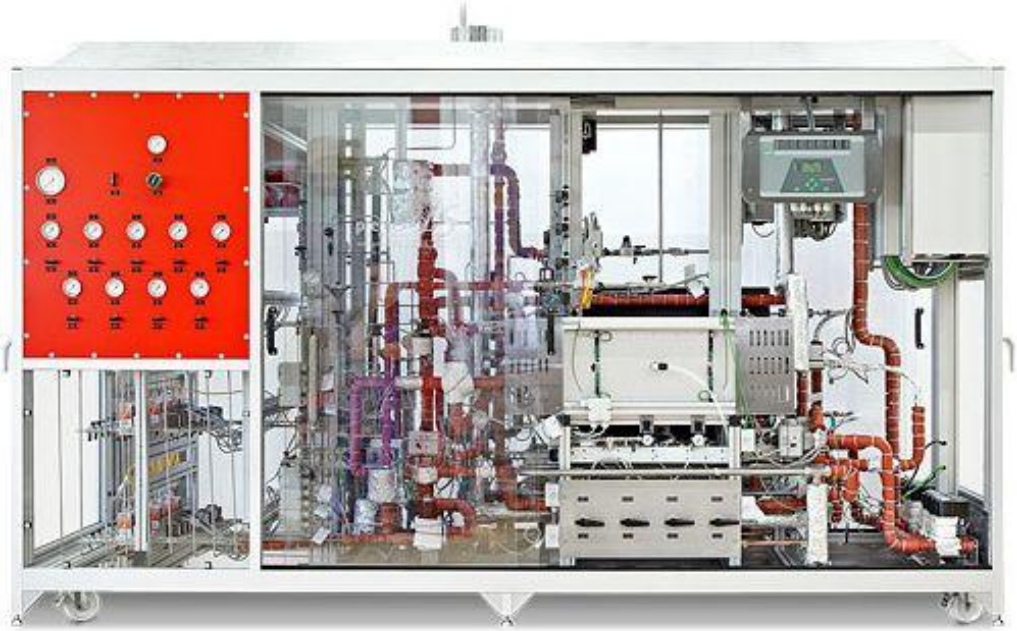
VGO Upgrading Pilot Plant

- Hydroprocessing
- 2 Trickle-Flow Reactors
- 2000mm Long
- 7-Zone heating
- Parallel or recycle operation
- 600°C
- 300 Bar
- Complete Distillate Workup
- Vacuum Distillation
- Fully Automated
- Package Unit

Diesel Catalyst Test Unit

Testing of Diesel particle Filters

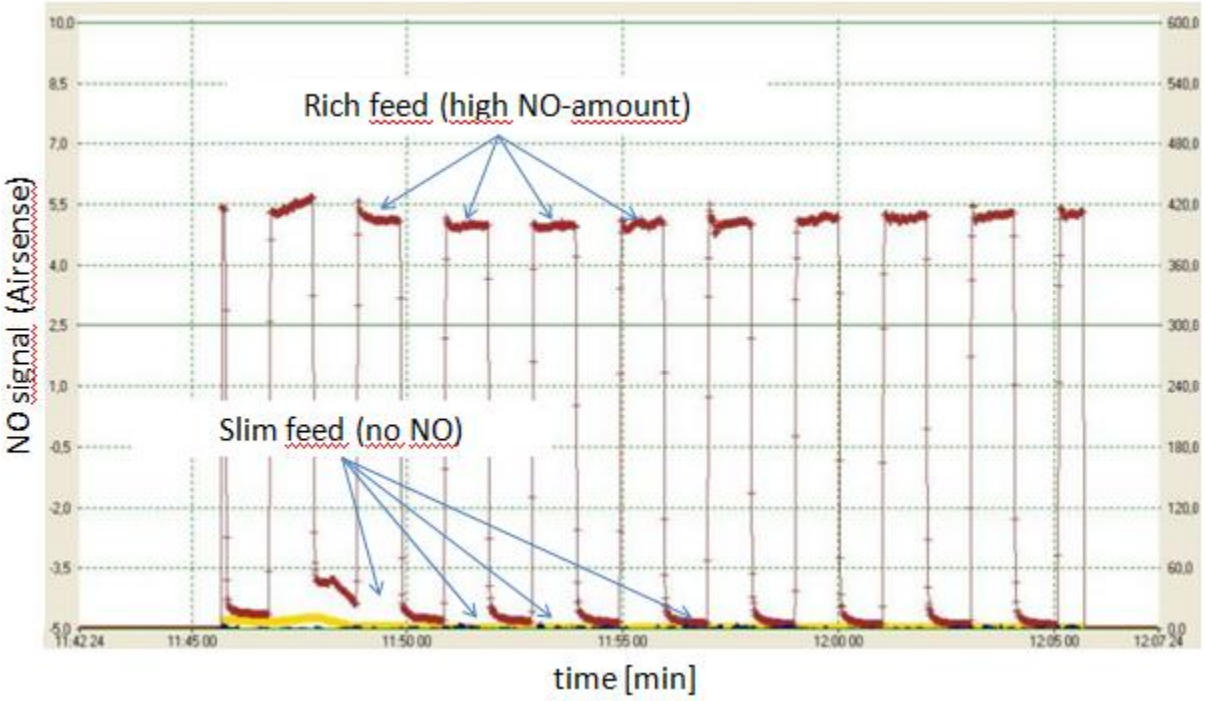
- 2 Steady state quartz reactors (T_{max}=1000°C) for Diesel Particle Filters
- 1 High dynamic oven (T_{max}=850°C; 50°C/min) for DeNOx storage cats.
- 36 MFCs, 4 evaporators
- Integrated FTIR, FID, MS, Lambda sensors
- Applicable for monoliths, other structures



High dynamic oven

Requirements for testing DeNOx catalysts:

- Pulsation free
- Plug flow
- Stable gas flows
- Rich/Lean switching with 1-3Hz



Diesel Particle Filter-deNOx Test Unit

Testing of complete particle filters

- 3 quartz reactors
- 850°C
- Ultra-Fast IR heating zone
- 300l/min gas flow
- Integrated FTIR
- Applicable for monoliths, other structures

IR Oven