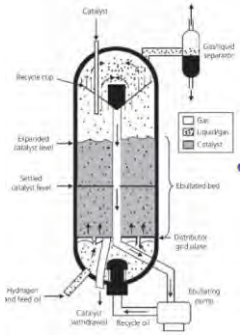
Heavy-Feed Catalyst Testing Solutions

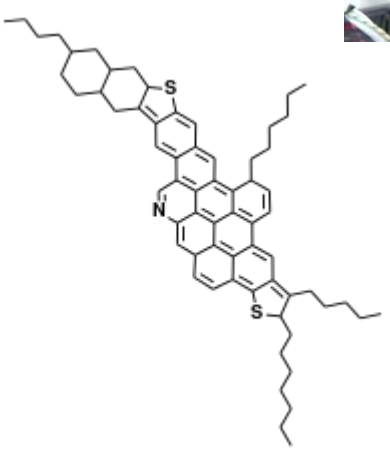
Heavy Feed Challenges

BOB Conversions

Industry-Specific Challenges

- BOB conversion can significantly improve refinery margins
- Difficulties in handling complicated feedstocks (sour and heavier feeds - large number of contaminants S, N, metals, asphaltene, etc.)
- Salt formation & corrosion issues (NACE)
- Asphaltene & Maltene precipitation resulting from mixing incompatible feedstocks can result in catalyst deactivation and fouling
- Scalable lab-scale testing for ebulating-bed reactors difficult to find
- Heavy feed-to-chemicals represents an attractive valorization route





Case Study 1

Catalyst Performance Data for Low-Volume Product

Problem: Client requires catalyst performance data for very heavy feedstocks.

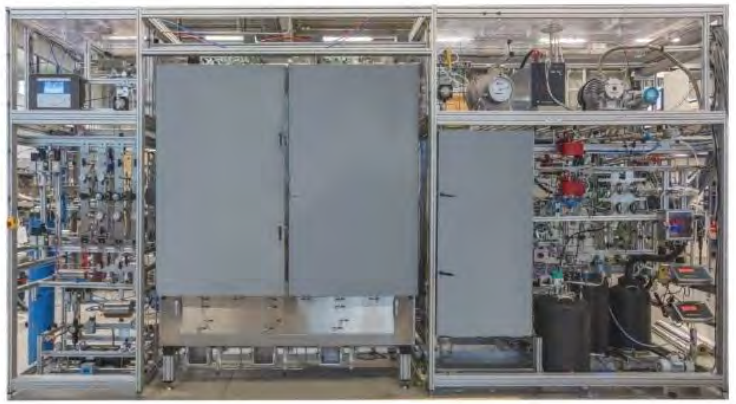
A small European refiner specializes in Base Oil synthesis. This is a low-volume/high-value product for which catalyst providers do not have relevant data for these low-volume feedstocks.

The client needs a flexible tool for doing internal catalyst evaluations with extremely heavy feedstocks (Bitumen and heavy resid).

ILS Solution:

- Rapid Screening Tool: A 4-parallel, mid-scale unit for rapid catalyst pre-screening
- Scale-up Testing Tool: 3-reactor-in-series unit simulating full-scale process

4-Parallel Rapid Screening Unit



3-Reactor-in-Series Micro-Pilot with Downstream workup

Case Study 1: Step 1

Rapid catalyst evaluation tool for heavy feeds!

Gas Compression & Pump Module

- Haskel Compressor for H_2/N_2 to 300 Bar
- Mass-Flow controllers for flow control
- High-Temp Lewa Dosing pumps
- Main emergency gas-detection computer

4-Parallel Reactors

- Enclosed in isothermal OVEN
- Parallel operation

Downstream

- High-Temp G/L Separators
- Low-Temp G/L separators
- Volumetric Gas Flow Measurement
- Online GC for Gas-phase product analysis

Case Study 1: Step 2

Micro-Pilot Reactor Simulate the entire process!

Gas Compression & Pump Module

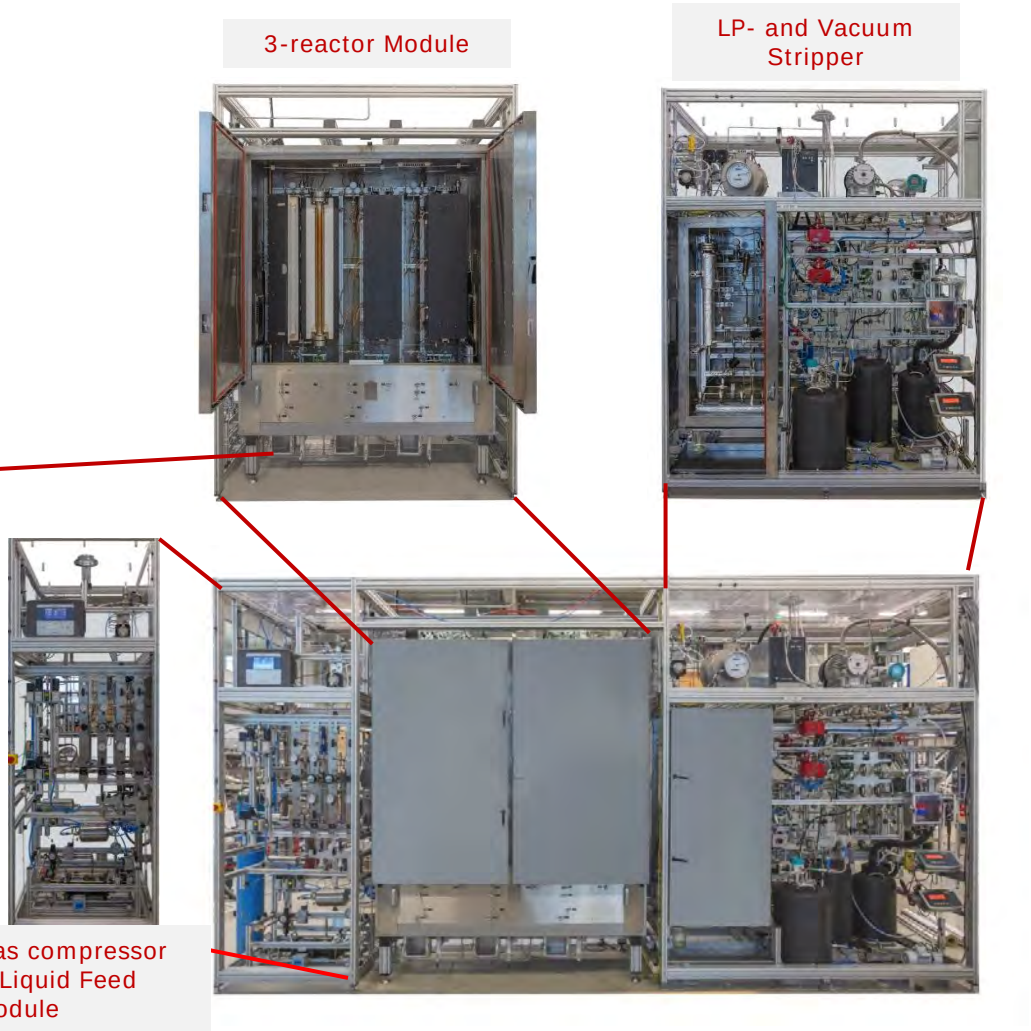
- Haskel Compressor for H_2/N_2 to 300 Bar
- Mass-Flow controllers for flow control
- High-Temp Lewa Dosing pumps
- Main emergency gas-detection computer

3-Reactor in Series Module

- Enclosed in isothermal OVEN
- Upflow / Downflow
- Parallel operation
- In-series operation
- Inter-reactor sampling possible
- Parallel + series
- Online GC as-Phase product analysis

LP- and Vacuum-Stripper

- Enclosed in isothermal OVEN
- Low-pressure Stripper (0-10Barg)
- Vacuum-Stripper
- Vacuum sampling possible
- Ritter Off-Gas flow measurement



Case Study 2

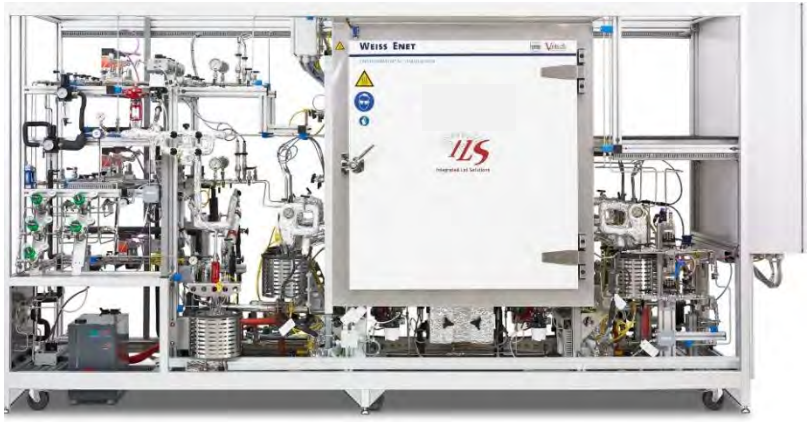
Rapid Catalyst & T/P Testing of Tar Sand Cracking Catalysts

Problem: Client requires a way to do rapid evaluation of additive-type liquid-phase cracking catalysts (Molybdenum- and Iron-Sulfide based) for tar-sand bitumen upgrading.

Phase stability based on Asphaltene and Maltene precipitation critical

ILS Solution:

- A 4-parallel, Miniature-scale parallel Slurry testing unit for catalyst pre-screening
- Extra pre-sulfiding reactor



Reactor design:

- 4 x 100ml reactors
- 1 extra pre-sulfiding reactor
- Tmax=550° C
- Pmax=250 Barg
- 316SS / optional HC276
- Hot G/L separator components mounted in isothermal oven
- Cold G/L separator for volatiles collection

Feed gasses:

- HP Hydrogen P 200 barg
- HP/LP Nitrogen

Analytics:

- Integrated Agilent GC for vapor analysis

Case Study 3

Rapid BOB Feedstock Evaluation

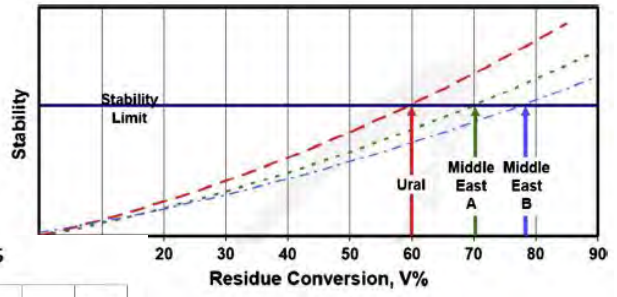
Problem: Client requires a way to rapidly determine which heavy BOB feedstocks are phase-stable for their ebulating bed process. Asphaltene and Maltene precipitation result in catalyst de-activation and serious fouling issues

ILS Solution:

- A 4-parallel, Miniature-scale parallel Slurry testing unit for catalyst pre-screening

Reactor design:

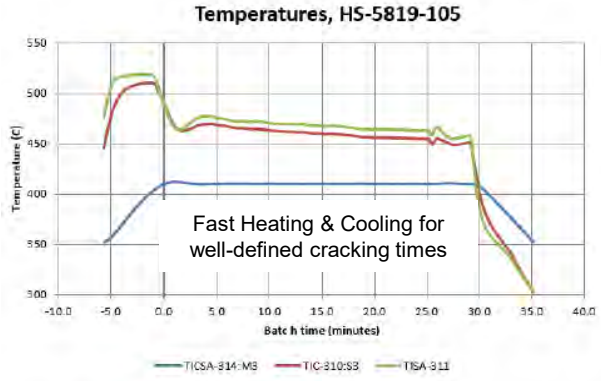
- 4 x 100ml reactors
- Tmax=550° C (rapid heating/cooling for short RT)
- Pmax=200 Barg
- 316SS / optional HC276
- Heated/cooled G/L separator for cracked product collection



Gragnani A. Understanding and optimization of residue conversion in H-Oil. <http://www.pdfio.com/k-249933.html>.

Feed gasses:

- HP Hydrogen P 200 barg
- HP/LP Nitrogen



Analytics:

- Integrated Agilent GC for vapor analysis

Case Study 4

Heavy Oil to Chemicals

Problem: Client wishes to explore opportunities to upgrade heavy feedstocks directly to chemicals.

ILS Solution:

- A 2-parallel, Miniature-scale parallel Slurry testing unit for pre-screening of catalyst and reaction conditions

Reactor design:

- 2 x 500ml reactors
- Tmax=650° C
- Pmax=250 Barg
- 316SS / optional HC276
- Downstream components mounted in isothermal oven

Feed gasses:

- HP Hydrogen P 200 barg
- HP/LP Nitrogen

Analytics:

- Online liquid sampling
- Online GC Analysis up to C10
- Integrated Agilent GC for vapor or offline liquid analysis via autosampler

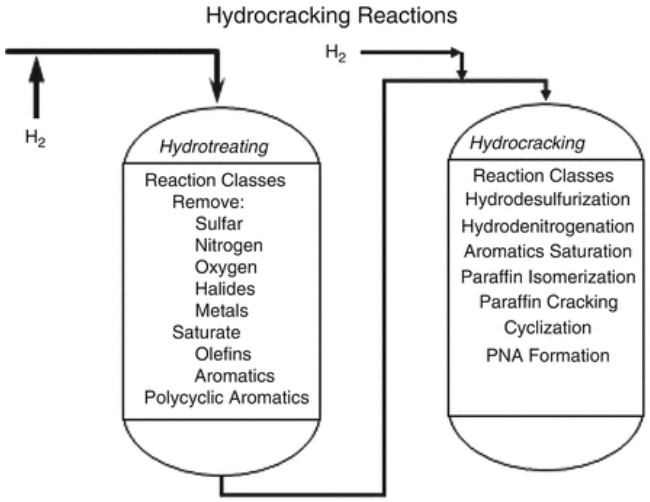
Case Study 5

Parallel Operation of Hydroprocessing/HCK Reactors in Series

Problem: Multiple clients wish to study both hydroprocessing (HDS/HDN) and hydrocracking (HDK) catalysts in series.

ILS Solution:

- Modular skids each containing 2-reactors which can be operated in parallel or series
- Multiple modules enable rapid catalyst testing



ILS Solutions

The flexible High-Throughput bench-scale reactor

Customized and proven turn-key reactor systems for hydroprocessing applications developed for major oil companies and leading catalyst manufacturers

ILS can provide in-house:

- Scale-specific, specialized engineering expertise
- Chemical-, Safety-, mechanical and software engineering
- Construction and testing
- Analytics integration
- FAT, SAT, Chemical validation
- Commissioning & on-site training

CLARIANT

Bharat Petroleum

REPSOL

sasol
reaching new frontiers

Typical HP+HCK Unit

Typical process conditions

Reactor design:

- Upflow (for catalyst flooding & wetting) and/or downflow
- Reactor operation T: from 25° to max 450°
- Reactor operation Pressure: from 10 barg to max 200 barg
- Upstream operation T: min. 25° to max 150° C
- Downstream operation T: from 25° to max 150 ° C
- Catalyst bed: 20gr-100gr (typically)

Feed gasses:

- HP Hydrogen P 200 barg
- HP/LP Nitrogen
- Mix gases optionally, up to 200 bar

Feed liquids:

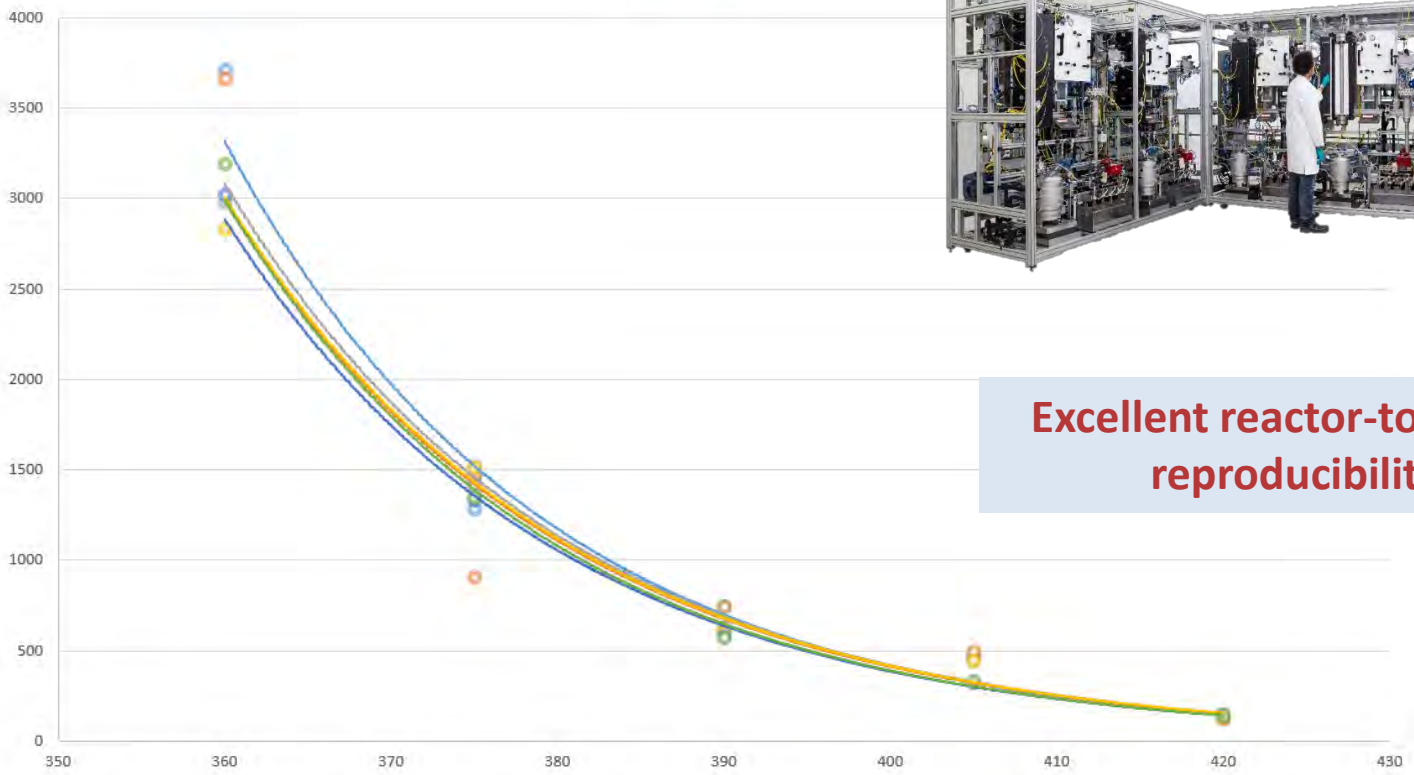
- Oil (Naphtha – HVGO & Residues)
- Spiked Oil (DMDS for pre-sulfidation)

LHSV:

- Min. 0.25 / hr Max. 4 / hr
- G/O ratio typically 50 - 2000

Results

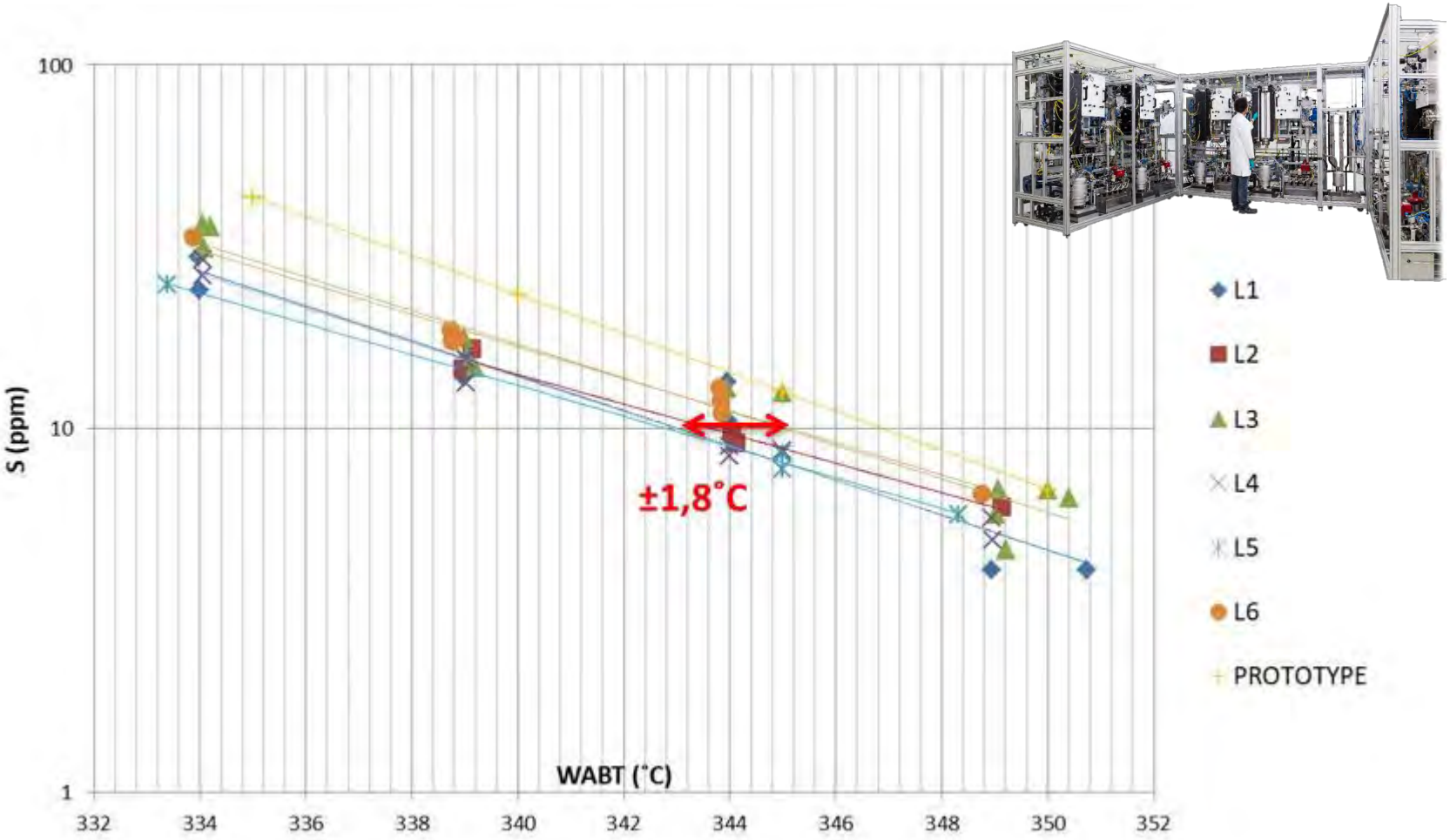
Typical Results of 6-Parallel Testing Unit



Excellent reactor-to-reactor reproducibility!

Results

Typical Results of 6-Parallel Testing Unit

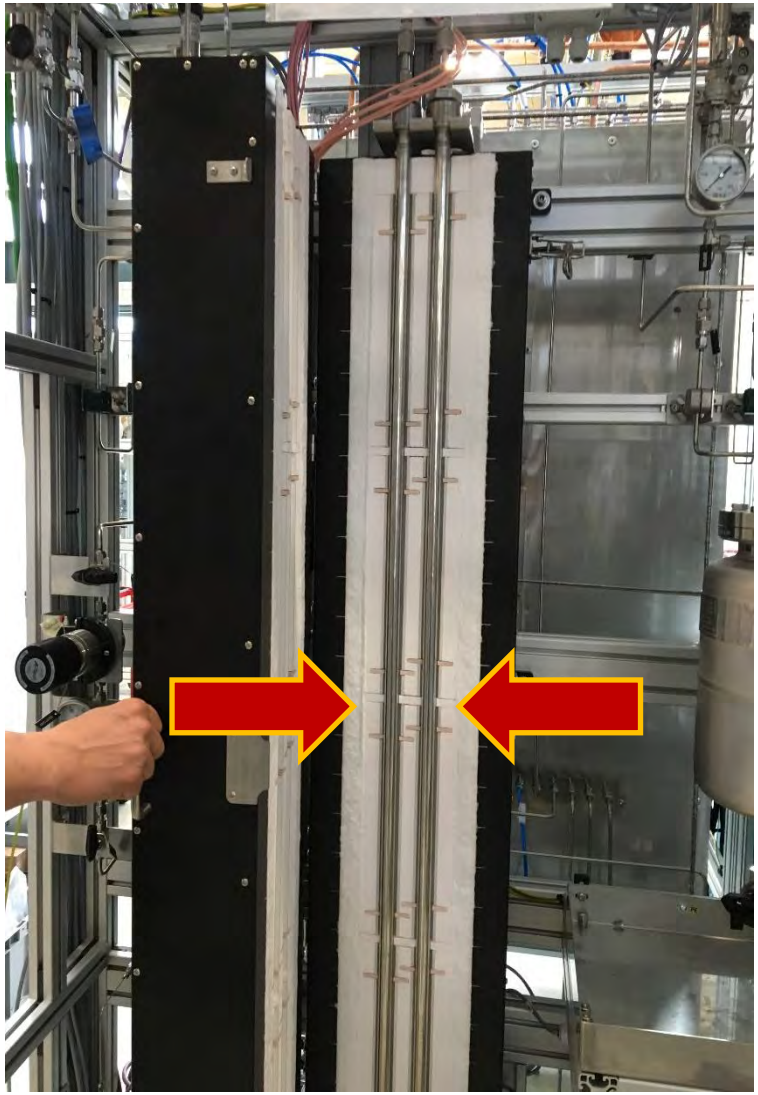


Optional Dual-Reactor/Oven Design

Identical external temperature profiles

Dual Reactor Design:

- Reactor Length 1-2 meters
- Hydrodynamically optimized for full-particle testing (with SiC dilution)
- Up- and downflow
- Multipoint internal temperature measurement
- Simple but robust low-torque metal-metal reactor sealing system
- Low-weight reactor design without flanges (improved isothermicity too)
- 5-zone Ovens for maximum isothermicity
- Diameter ½ to 1”
- 2 Reactors in each oven ensure best comparison of catalysts back-to-back
- Ultra compact cven design
- Goal is WABT variation under 1° C!

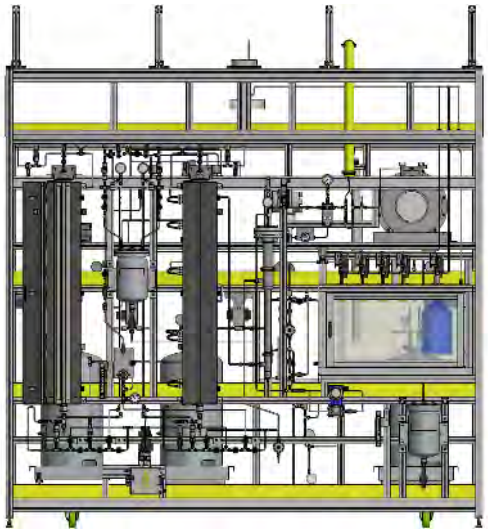


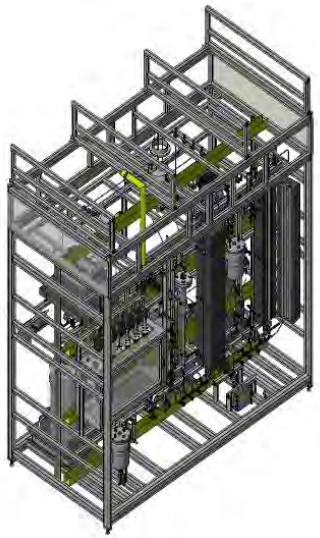
Standard proposal

Modular process skid design

Benefits for a modular process method mounted in an enclosed aluminium profile:

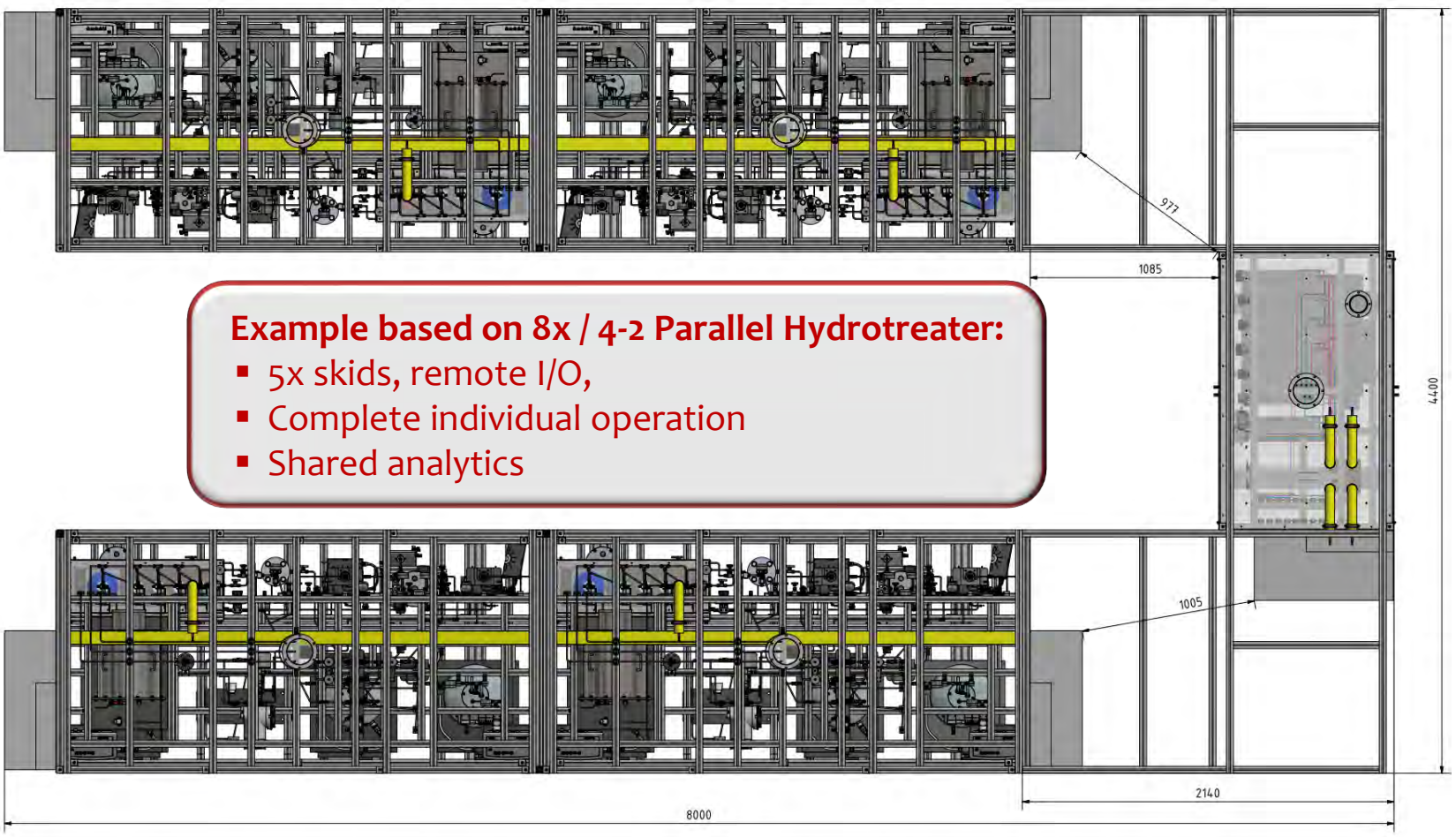
- Project timeline maximizes efficiency
- Higher quality construction
- Increase degree of safety as it allows for the system to be ventilated and make use of the gas detection system (HC/H2S/H2) present in the room
- The unit is outfitted with wheels for easy transport
- Ventilation calculations are performed





Typical Dimensions

Modular process skid design



Example based on 8x / 4-2 Parallel Hydrotreater:

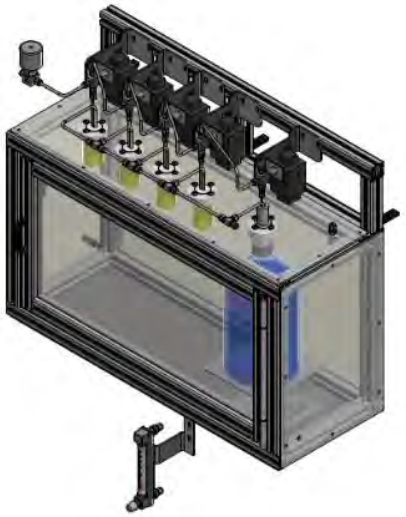
- 5x skids, remote I/O,
- Complete individual operation
- Shared analytics

Product Sampling

Sampling

Inter-Reactor & Post-Reactor Liquid Sampling:

- Fully automated!
- No disruption of reactor pressure during sampling
- No dead-end sulfur hold up
- Heated transfer lines
- Sampling vials enclosed separately to prevent H2S emmissions
- Intermediate reactor sampling possible when performing reaction-in-series (HDS/Hydrocracking)



Standard proposal

Safety standards

For all units ILS will carry out a HAZOP, SIL and risk analysis according to DIN ISO 13489.

A ventilation calculation of the unit is performed to determine required suction capacity based on expected leak rates and worst case scenarios

Safety measures taken into account:

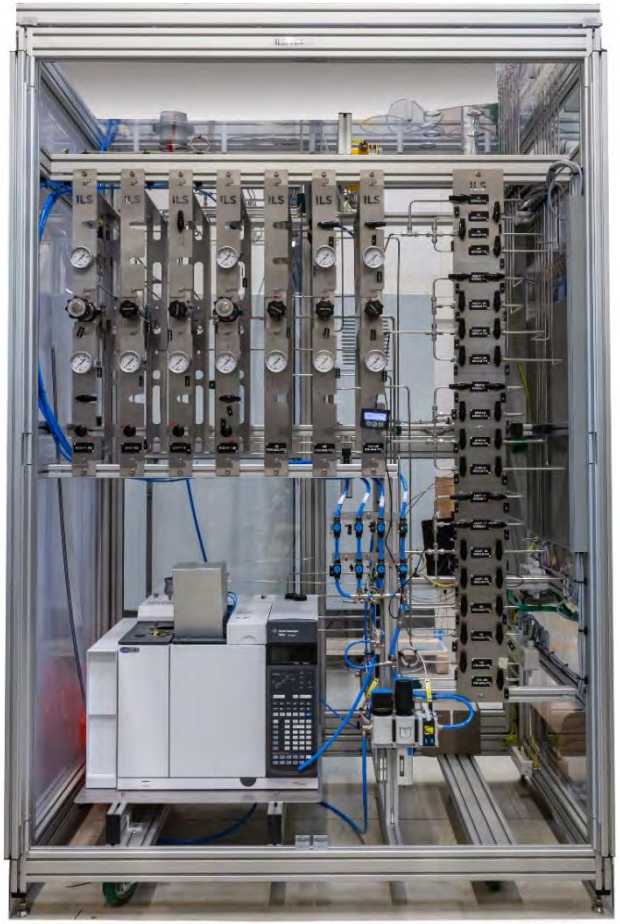
- Separate overtemperature safety switches
- Separate thermocouples are user for control and alarm purpose
- Overpressure relief valves
- Ventilation sensor with LL alarm and local display
- Gas detectors for H2S and flammable gases

Liquid & Gas-Phase Analytics

On and Offline Analytics

Seamless integration of analytical data in consolidated reports can be realized:

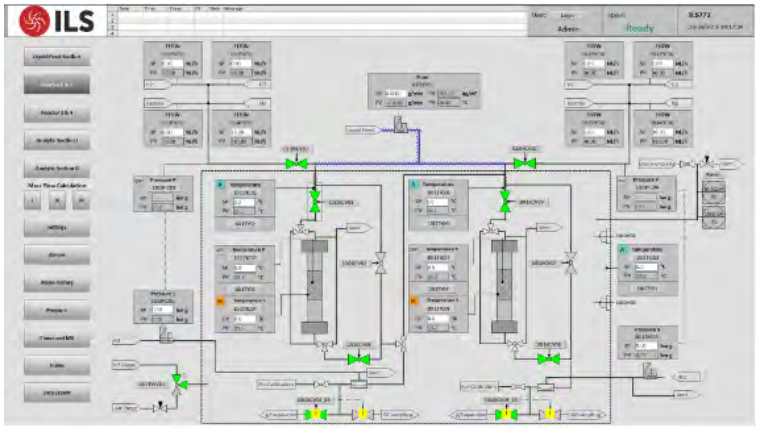
- Online GC
- Offline analytics (S, N, SimDis)
- Sampling robotics (online / offline)



Full Automation

Process Control Solutions/PLC/HMI

- Various PLC‘s available :
  - Siemens S7 & PCS7
  - Rockwell
  - Delta-V
  - Honeywell
- High stability for 24/7 operation
- Highest safety standards

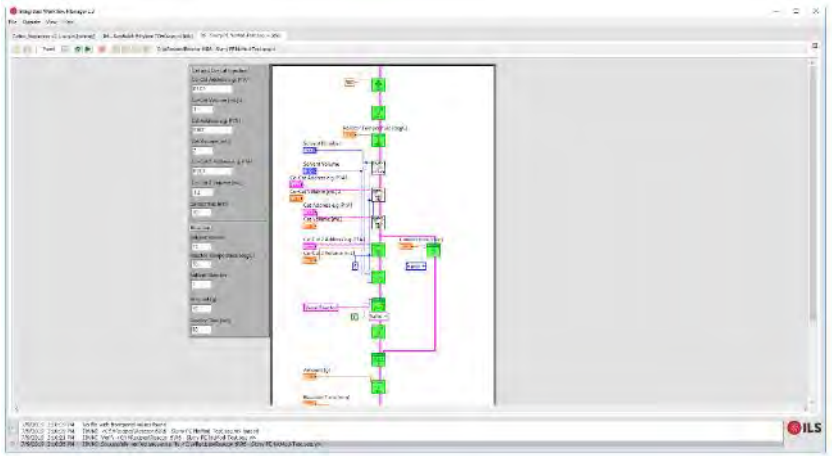


Standard proposal

Scientific Software/WM – Workflow Manager

ILS Workflow Manager:

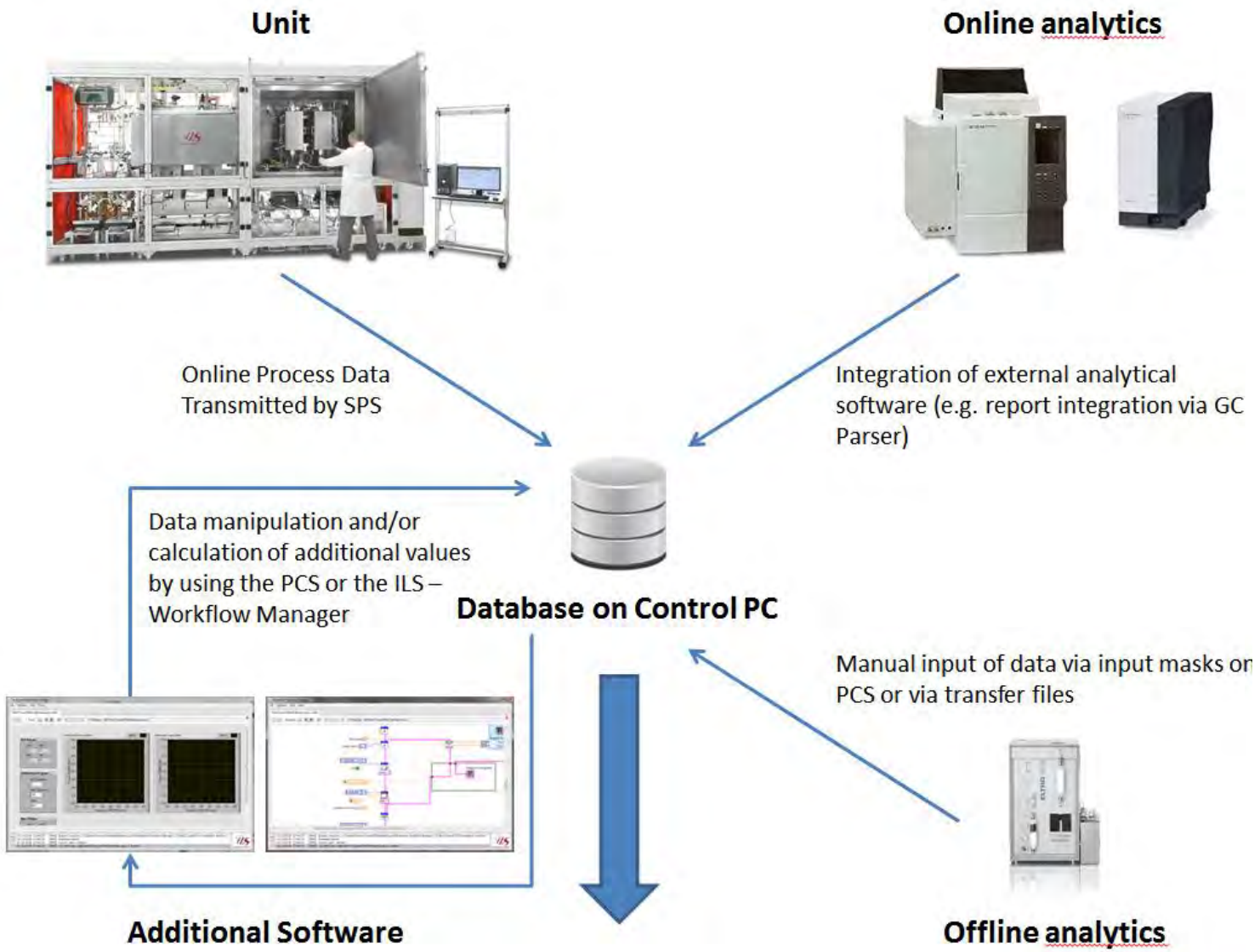
- Powerful recipe engine
- Drag & drop system
- Liveview of critical catalyst parameters
- Data export and integration
- Calibration functionalities
- Report generation



User friendly and suitable for complex applications

ILS Workflow Manager

Integrating online and offline analytics



ILS Solutions

Chemical Validation in ILS laboratories

Chemical validation in ILS validation laboratories ensure unit performance BEFORE delivery:

- Risk reduction, faster startup on site
- System optimizations done before delivery
- Integration with online analytics
- Testing on actual catalysts from the clients before delivery
- Improved data quality
- Training at ILS prior to shipment

ILS-Company Overview

Key facts

- Driven by visionary scientists and engineers
- Technology leader in chemical R&D systems
- Integrated in Germany's largest science and technology park
- Over 15 years of experience in the industry
- 50 employees
- Over 200 reactors delivered worldwide

Headquarter	Berlin
Type of company	Private
Foundation	2003
Phd, Ms Chemical Engineers and Chemists	50% staff

Systems & Solutions

Core competencies

ENERGY AND REFINERY :

Syngas processes :

- Fischer Tropsch synthesis (FTS)
- Gas to Liquid (GTL)
- Gas to Olefins (GTO)

Hydroprocessing :

- Hydrodesulfurization (HDS)
- Hydrodenitrogenation (HDN)
- Hydrodeoxygenation (HDO)
- Hydrogenation (HYD)
- Hydrocracking (HCK)

Steam/dry, catalytic reforming
Alkylations, Isomerization
Biofuels / BGTL

POLYMERS :

- Polyolefins
- Polyamide
- Polycarbonate
- Polystyrene

(PETRO)CHEMICAL :

Selective oxidations
Hydrogenations
Carbonylations
Dehydrogenations
Alkane activation

AUTOMOTIVE :

Diesel Oxidation Catalyst (DOC)
Three Way Catalyst (TWC)
DeNOx and Selective Catalytic Reduction (SCR)
Diesel particulate filter (DPF) and soot combustion units
Methane Oxidation Reaction (MOC)
Catalyst aging units
Transient testing / driving cycle simulation

ENVIRONMENTAL AND RENEWABLES :

Bio chemicals :

- MC- catalysis, Coke oven gas (COG) BTX removal
- Flue gas cleaning

CO_2 utilisation:

- Syngas to Ammonia, Syngas to Methanol
- Electrochemical reduction of CO_2

SPECIAL APPLICATIONS :

Fluorination
Synthesis robots
Styrene monomere & propylene oxide
SMPO catalyst preparation
$SiCl_4$ Fluidized Bed Reactor

Special ILS Reactor design :

- Temkin Reactors
- Berty Reactors

ILS Integrated Lab Solutions GmbH
Barbara-McClintock-Str. 11
12489 Berlin – Germany
info@integratedlabsolutions.com

www.integratedlabsolutions.com