EURECAT

Katalizátor management:

regenerálás, reaktiválás, ex-situ szulfidálási technológiák

Gabriella Fogassy

20 March 2013, Sisak

WHERE WE ARE LOCATED?

EURECAT IN THE WORLD

IFP Energies nouvelles — Axens (IFP Group Technologies)

ALBEMARLE®

EURECAT

Americas

Eurecat US
Pasadena/TX

Petroval US
Houston/TX

Tricat US
McAlester/OK

Europe

Eurecat France
La Voulte-sur-Rhône
Petroval SA
St Romain de Colbosc

Eco-Rigen
Gela, Italy

Tricat
Bitterfeld, Germany

Middle East

Al-Bilad Catalyst
Al-Jubail, Saudi Arabia

Asia

Eurecat India Catalyst Services
Jhagadia, Gujarat, India

Petroval AP
Eurecat Rep. Office
Singapore

STANDARDS & VALUES

Responsible Care Program

Quality ISO 9001 certification

Environmental ISO 14001 certification

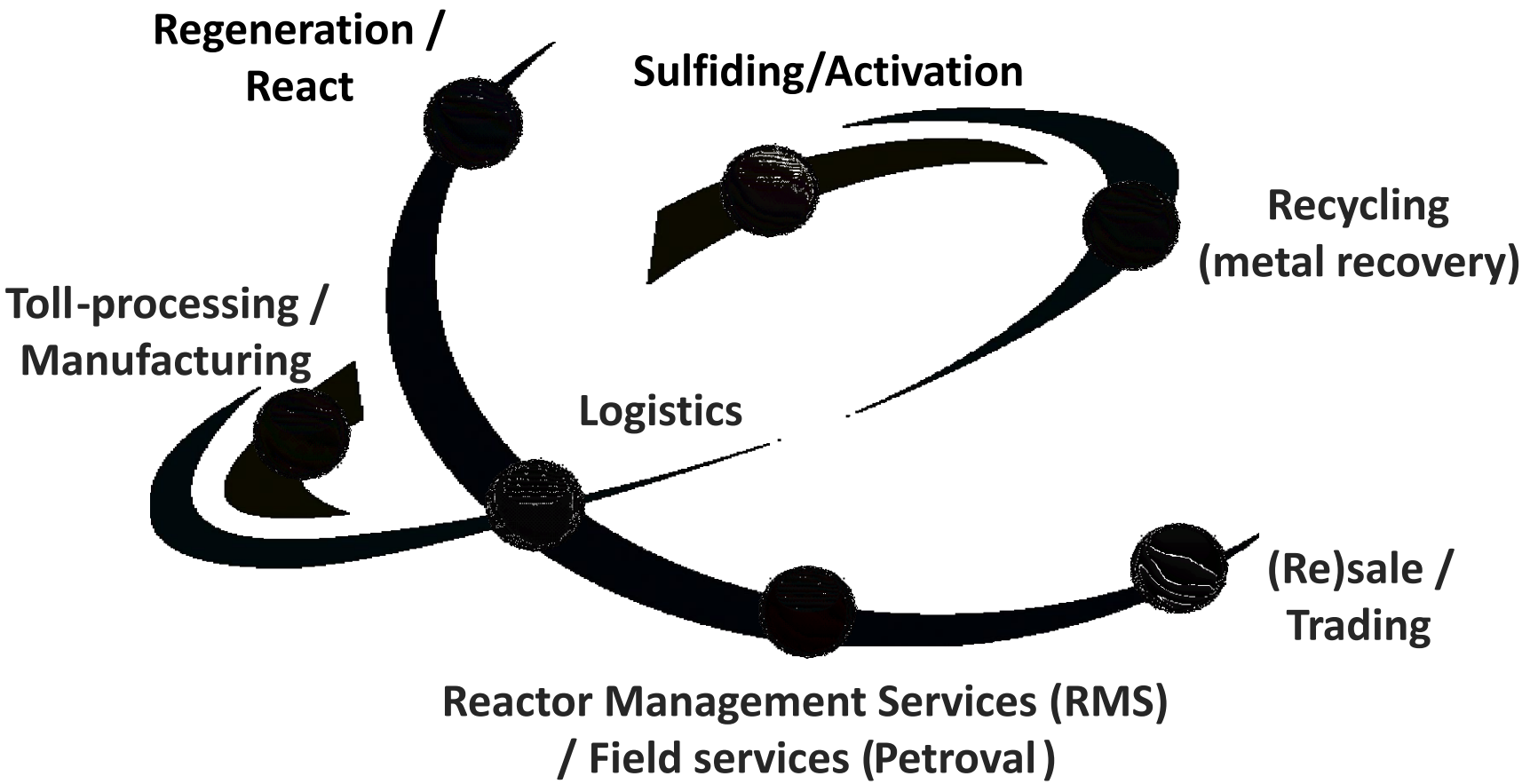
Safety OHSAS* 18001 certification or equivalent

*(*Occupational Health and Safety Assessment Series)*

Our values:

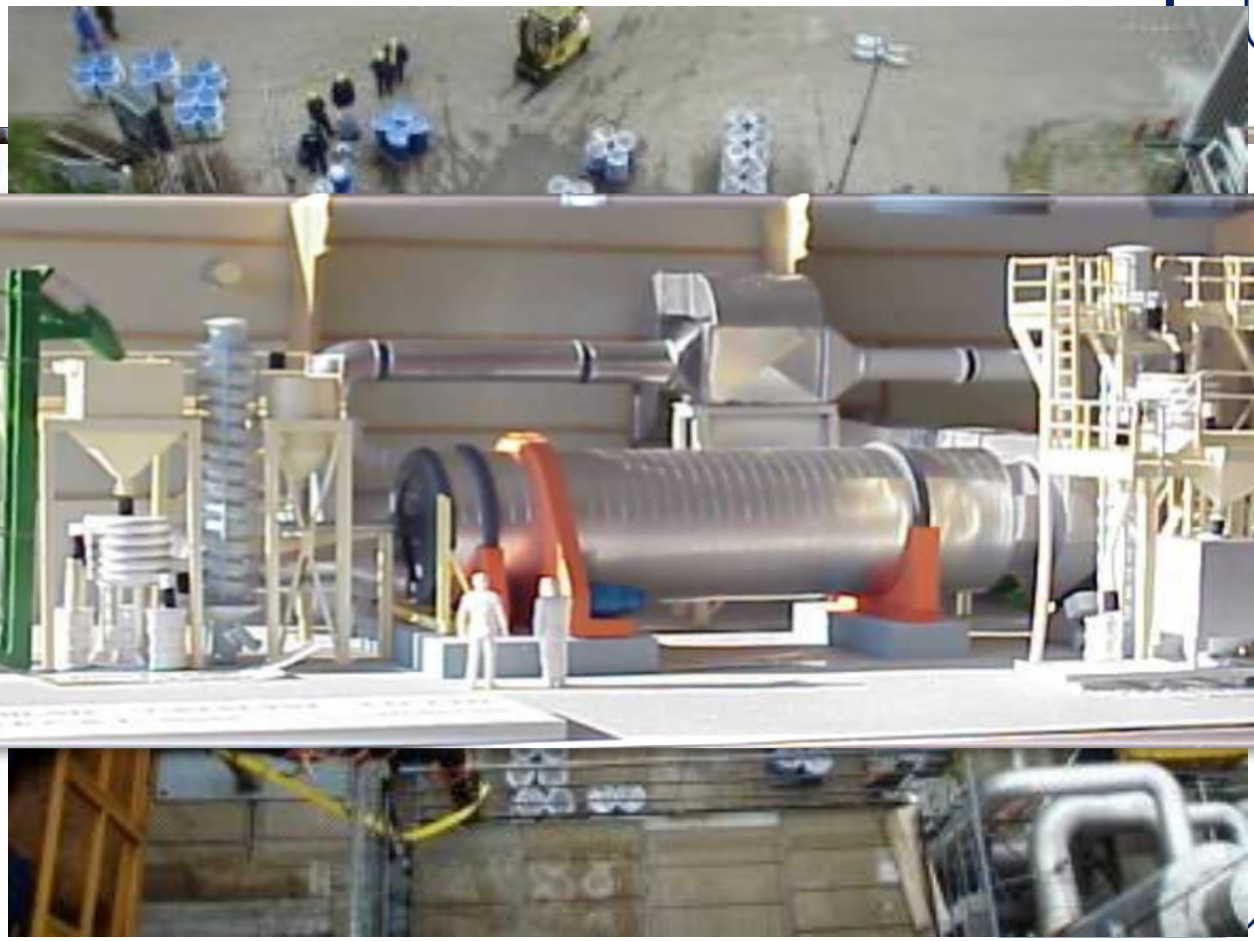
- Innovation
- Customer satisfaction
- Confidentiality
- Ethics

SERVICES, PRODUCTS & TREATMENT



ÜZEMI MŰKÖDÉS/KATALIZÁTOR ÉLETCIKLUS

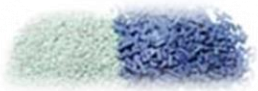
Metal reclaim

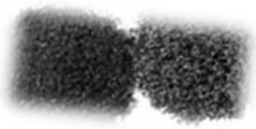


Fresh

ration
ation

ÜZEMI MŰKÖDÉS/KATALIZÁTOR ÉLETCIKLUS





Fém visz-szanyerés

Új → Betöltés → Üzemindítás → Termelés → Leürítés → Regenerálás Reaktiválás

In-situ szulfidálás

Aktivitás Δp

SMARTDUMP CARBODUMP

ISMÉTELT FELHASZNÁLÁS
KASZKÁDOLÁSI KÉSZLET ELADÁS

Ex-situ szulfidálás: TOTSUCAT

Kezdeti aktivitás Δp előrejelzés

REGENERÁLÁSI TECHNOLÓGIÁK

HAGYOMÁNYOS REGENERÁLÁS

- Zeolites
- Ni Catalysts
- Pd Catalysts
- CoMo, NiMo, NiW Catalysts

SZÁRAZ REGENERÁLÁS

- Zeolites (Confidential)

REJUVENÁLÁS

- CoMo, NiMo catalysts

REGENERATION TECHNOLOGIES

CONVENTIONAL REGENERATION

- Zeolites
- Ni Catalysts
- Pd Catalysts
- CoMo, NiMo, NiW Catalysts

DRY REGENERATION

- Zeolites (Confidential)

REJUVENATION

- CoMo, NiMo catalysts

HPC DEAKTIVÁLÓDÁSI MECHANIZMUSOK

2. **Szerkezeti módosulások:**
   - Fém részecskék szintereződése
   - Migráció és/vagy aktív specieszek megszűnése
   - Szerkezeti változások

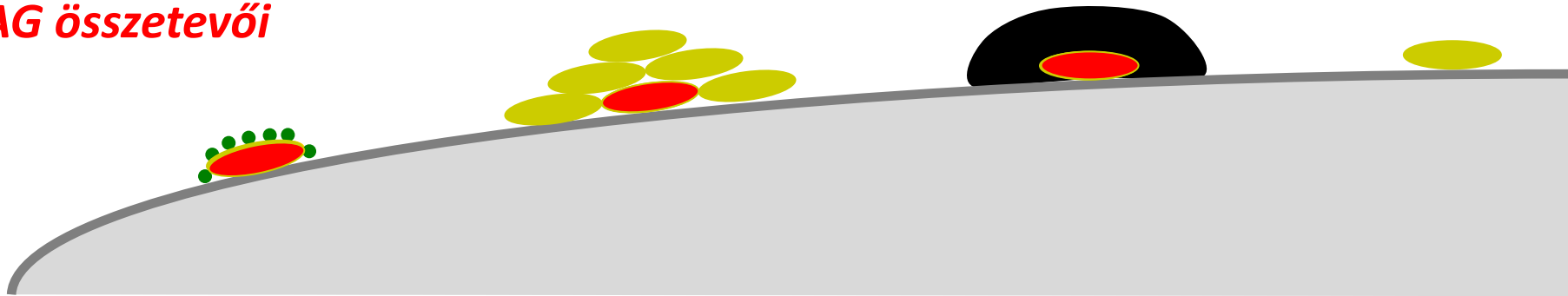
   Üzemeltetési körülmények függvényében

3. **Irreverzibilis mérgeződés:**
   - Fém szennyezők (V, Fe, As, Hg)
   - Szilicium, nátrium…
   - Kén (Ni…)

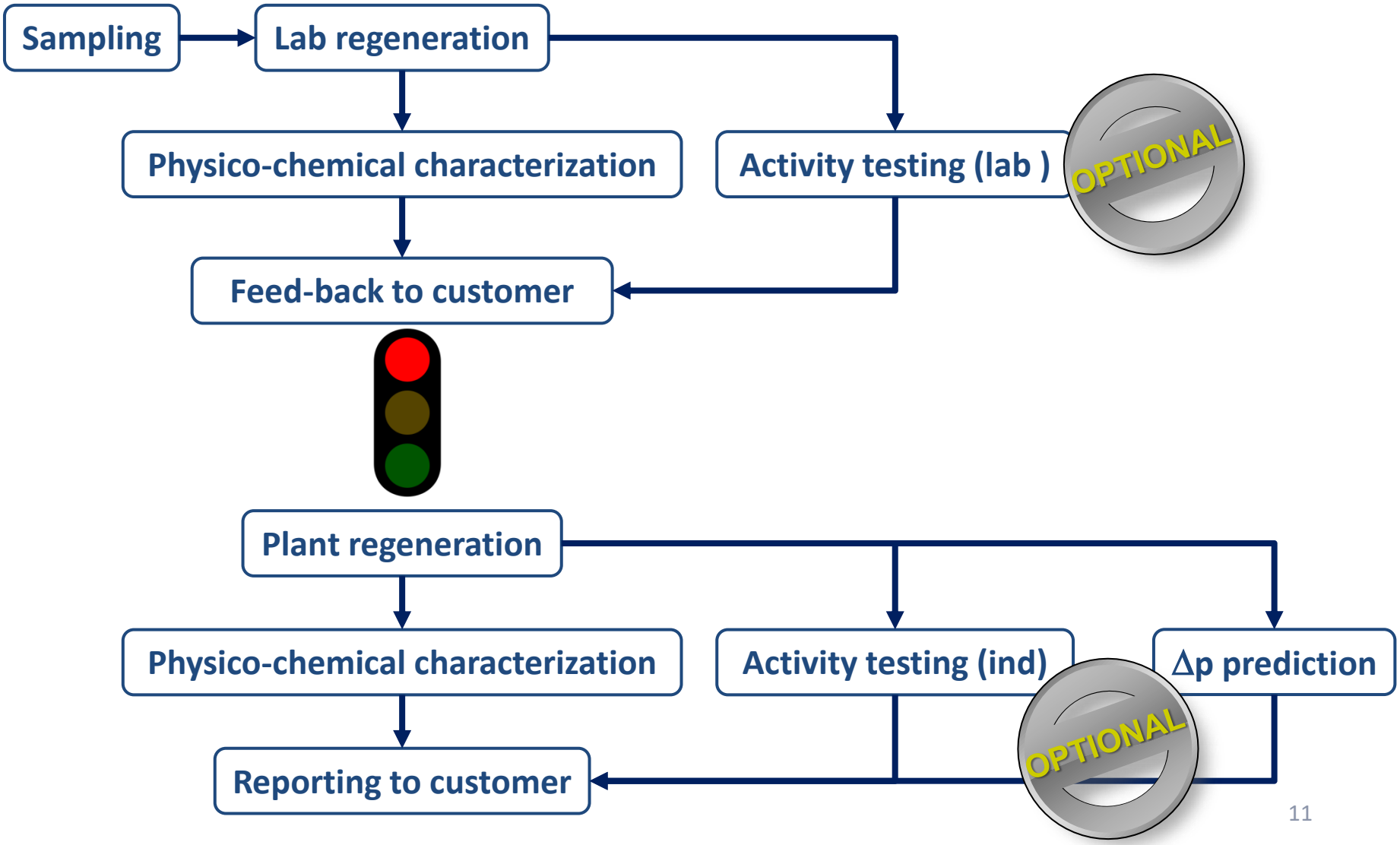
AAG összetevői

1. **Reverzibilis mérgeződés:**
   - Szén: 5-25%
   - Bázikus nitrogén (Hidrokrakk)

Mellékreakciókból



HPC TREATMENT



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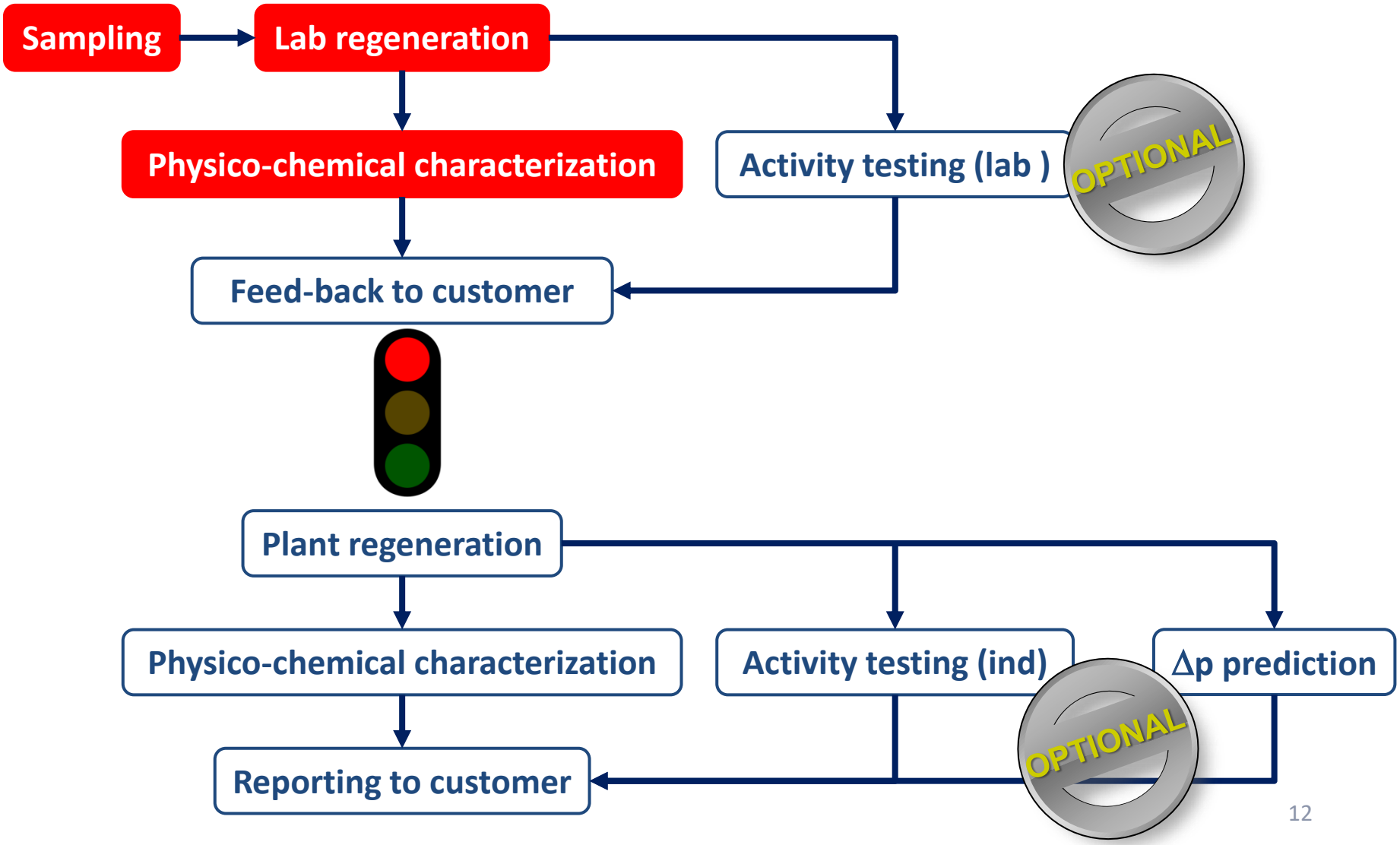


MINTÁZÁS/ANALÍZIS

1 reprezentatív ágyminta

Standard

mintavételi eljárás

(mindegyik ágyból):

15-30 mintát vesznek,

összekeverik, homogenizálják az

elemzés előtt

Ágy profil

Dedikált SAS

mintavételi eljárás

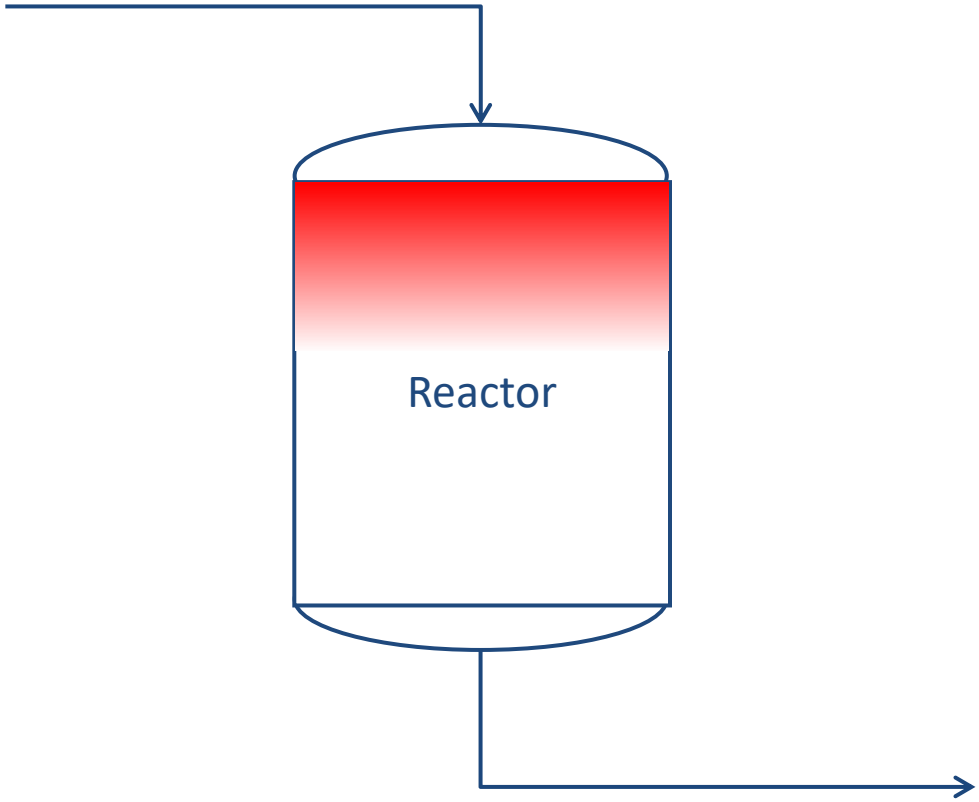
(mindegyik ágyból):

15-30 mintát vesznek, amiket

megtartanak egyedi elemzés

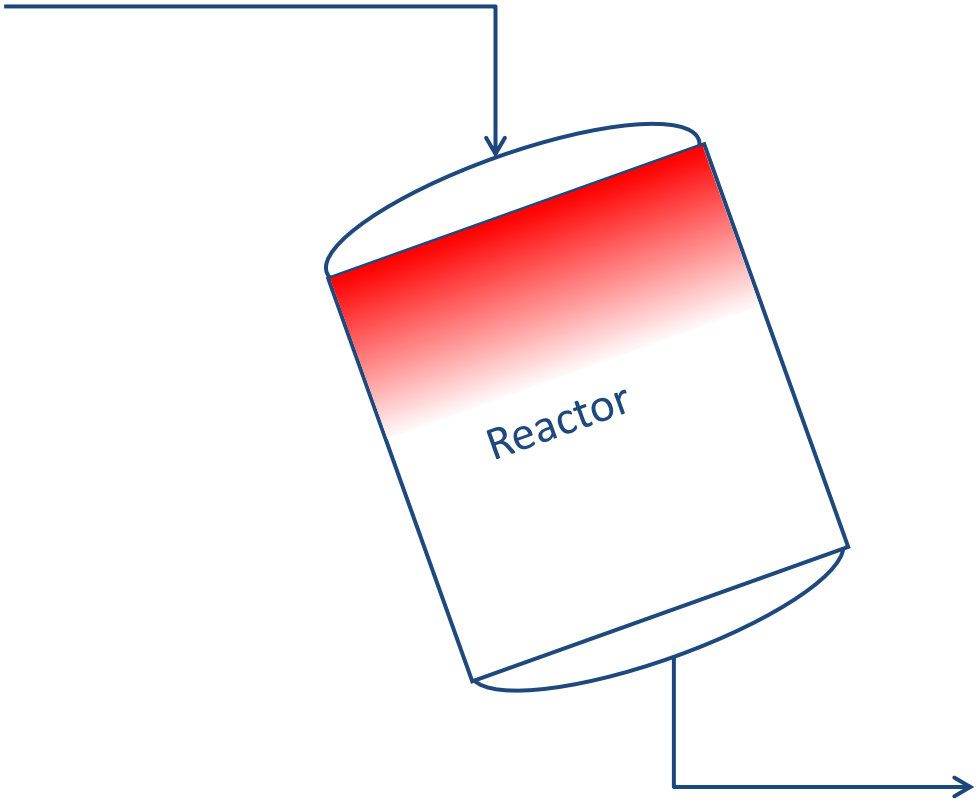
céljára

MAXIMISE RECOVERY SAS (SAMPLE/ANALYSE/SEGREGATE)



Catalyst is often partly contaminated, partly in good shape

SAS: SAMPLE/ANALYSE/SEGREGATE



Catalyst is often partly contaminated, partly in good shape

- **Allows to trace metals contamination vs height in reactor.**

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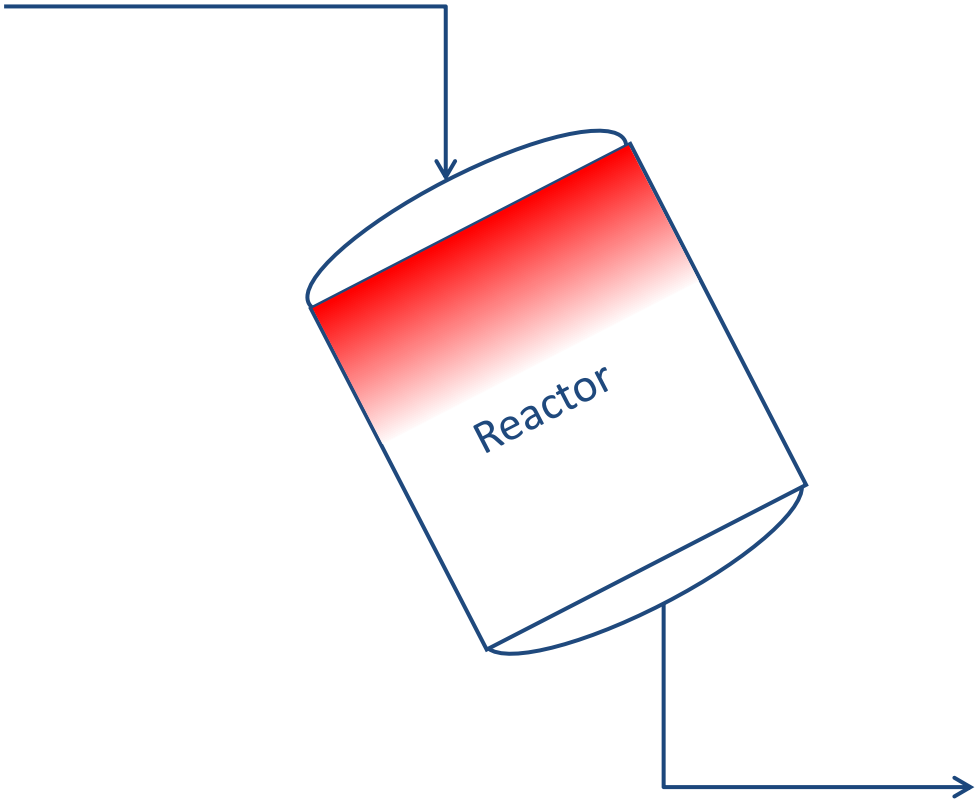

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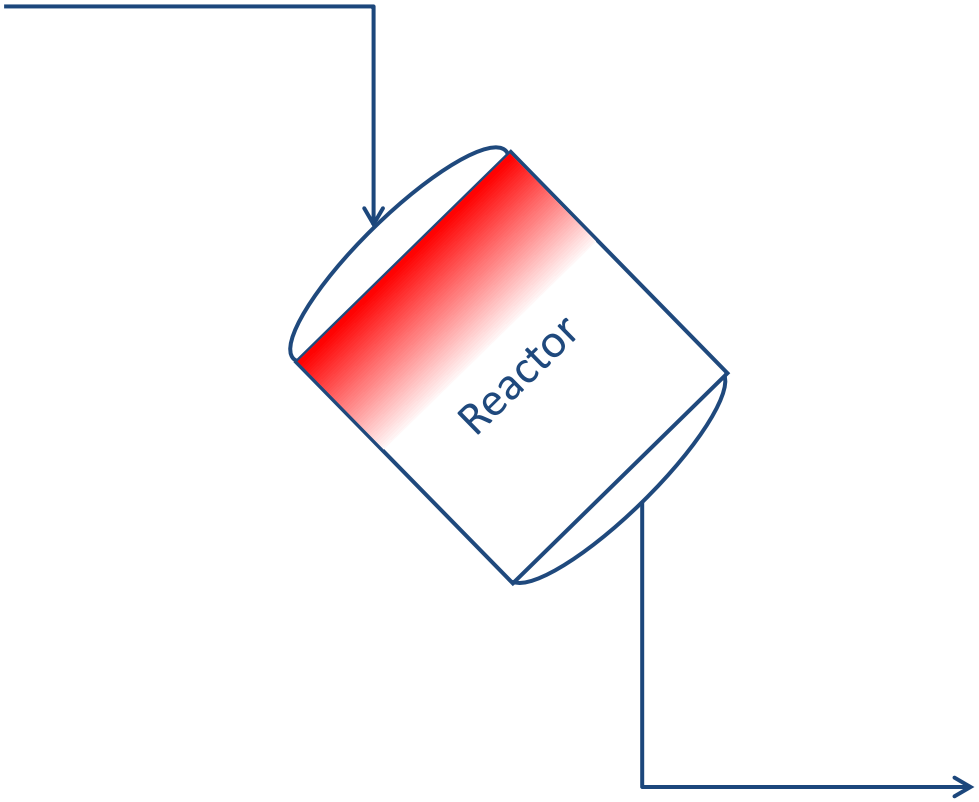

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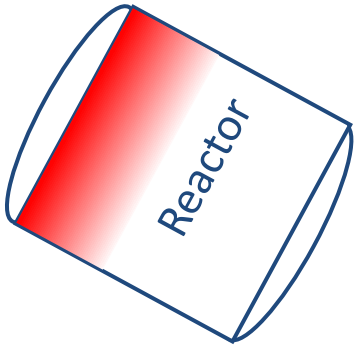

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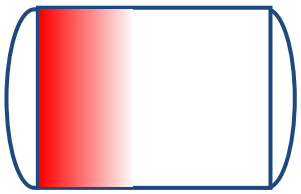

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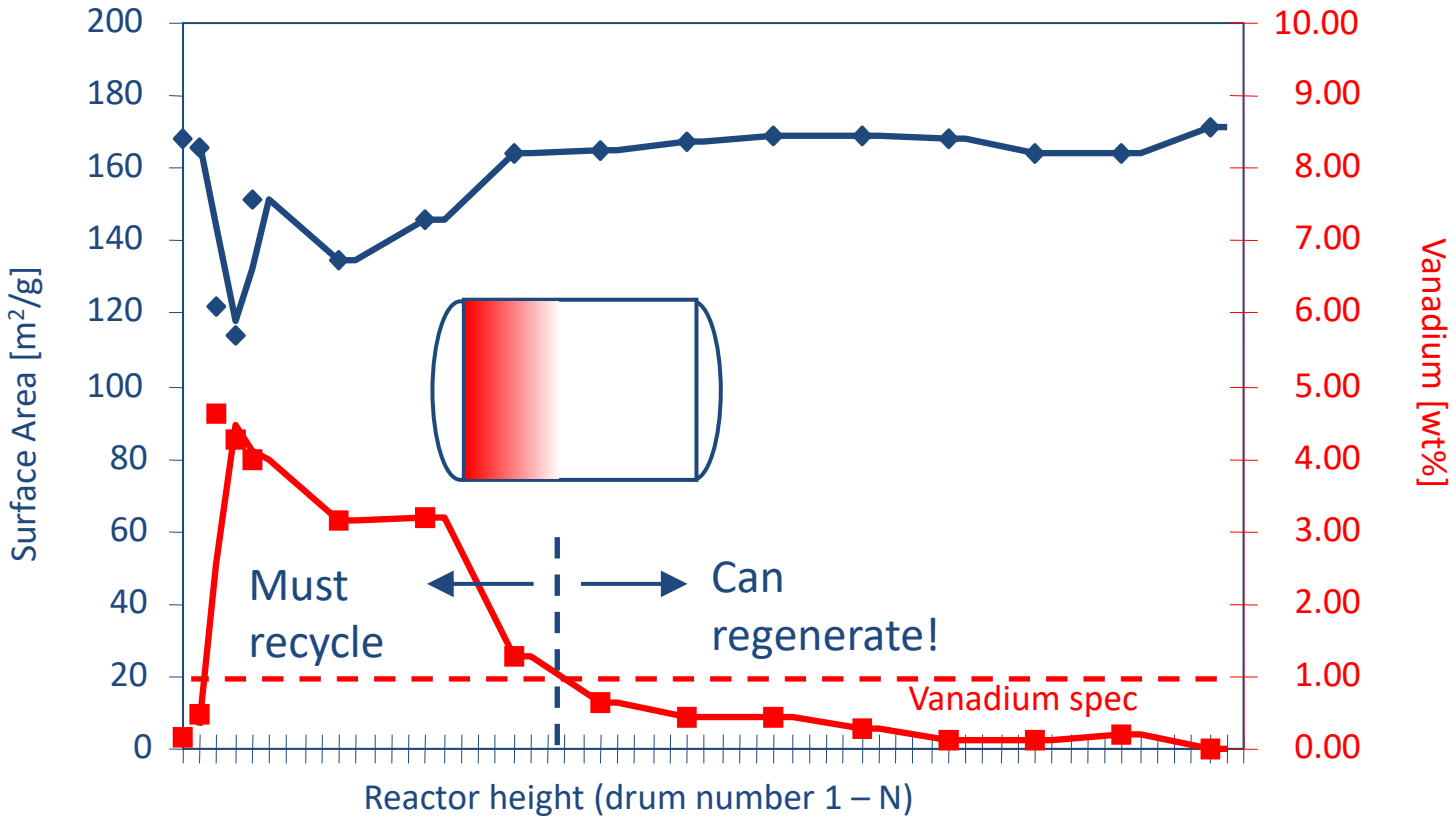
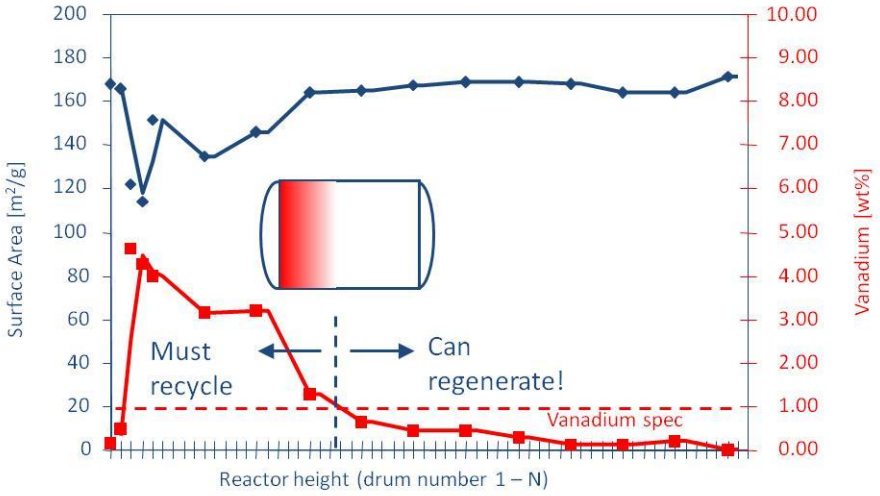


- **Hordókat/konténereket számozni kell a leürítés során!!!**
- **Nyomonkövethető a fémszennyezés a reaktor hosszában**

SAS: SAMPLE/ANALYSE/SEGREGATE



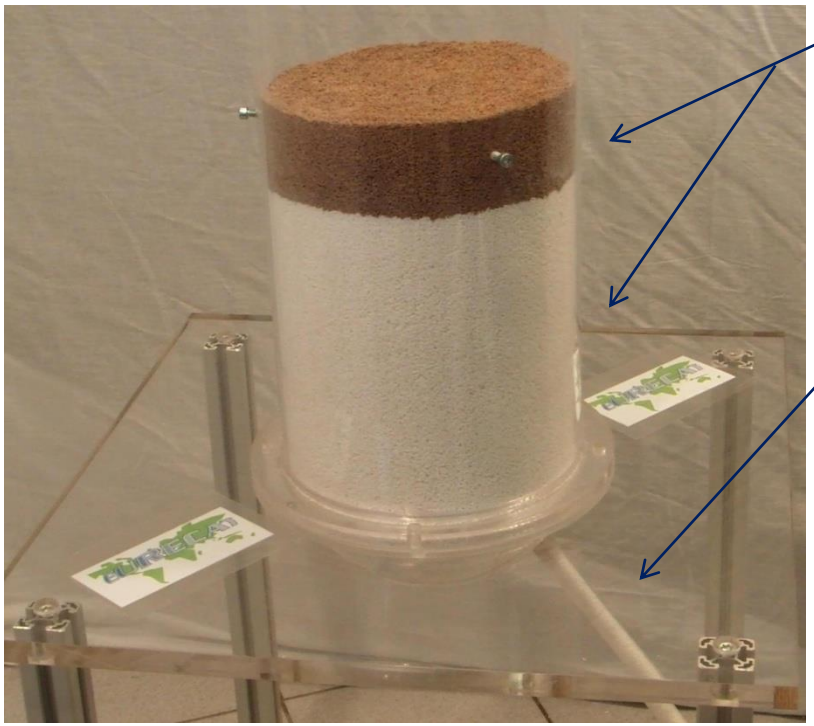
Case study of 96 tons reactor

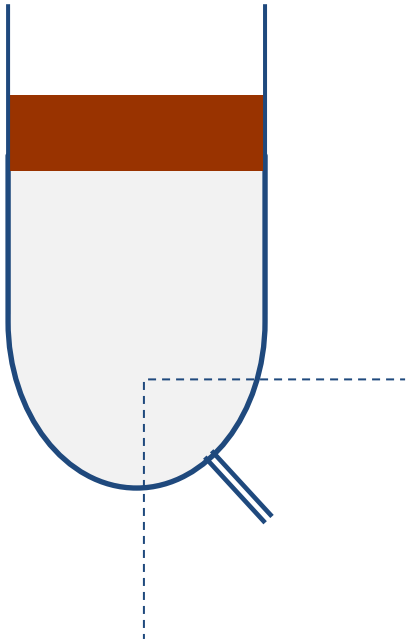
- Average analyses slightly off-spec
- Refiner wanted to send batch for metals recovery.
- SAS pointed out contamination was very local
- 75% of catalyst could be recovered
- 1.3 M$ savings

FROM GRAVITY DUMPING TO SAS

2 stacked beds – dense load

1 dump pipe (45°)

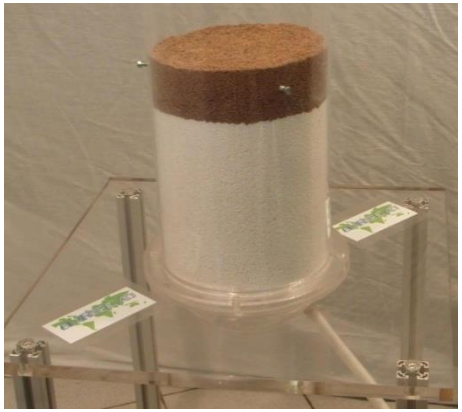




Diameter = 24 cm

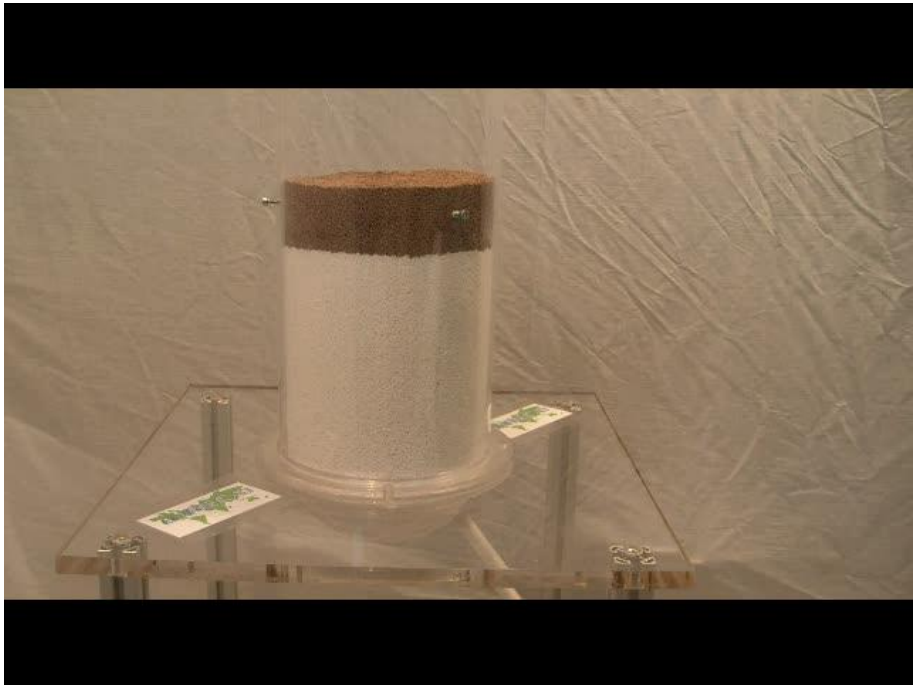
Height = 1 m

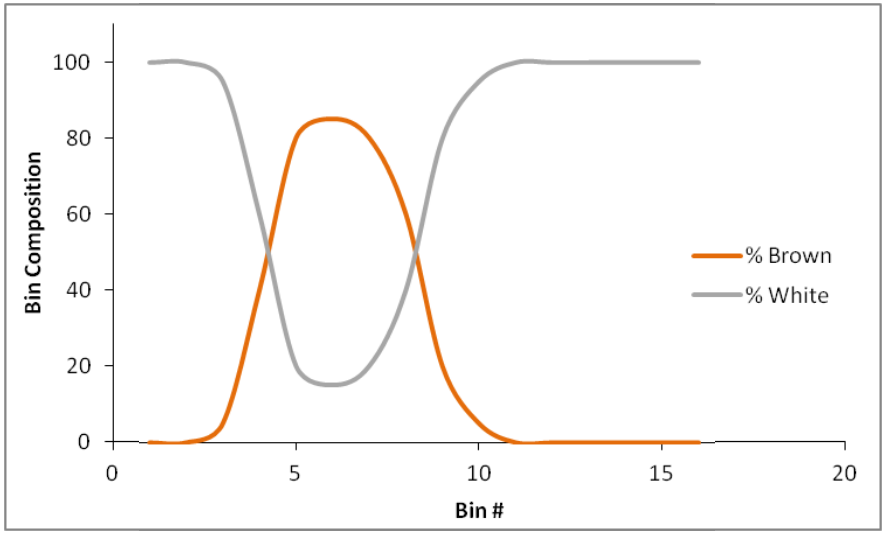
FROM GRAVITY DUMPING



Side view

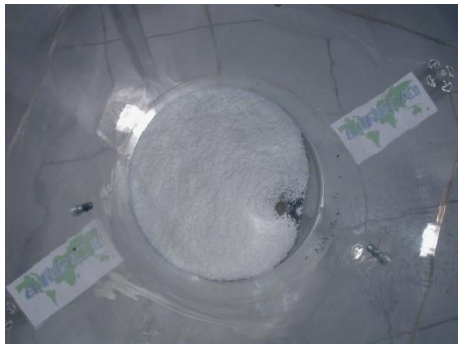
Front view





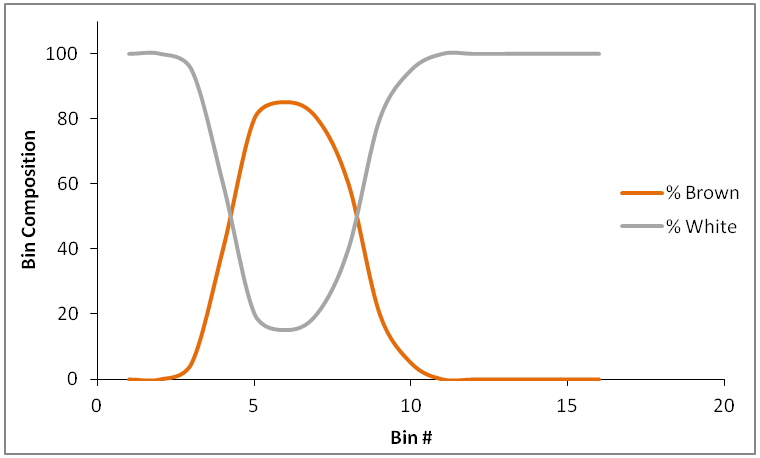
TO SAS

Dig out
Conical shape



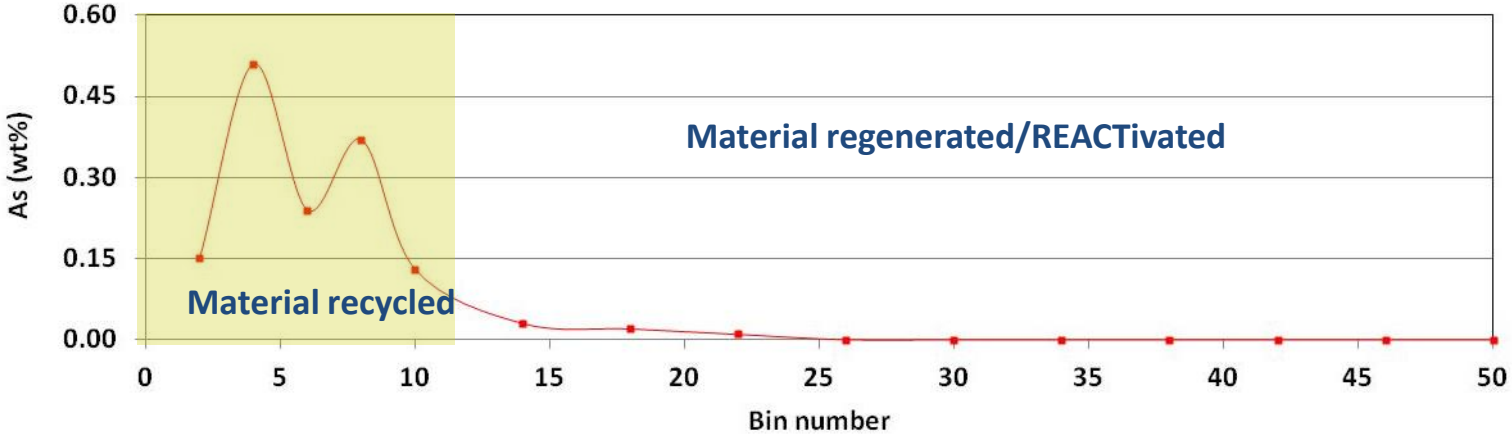
SAS

Gravity Unloading



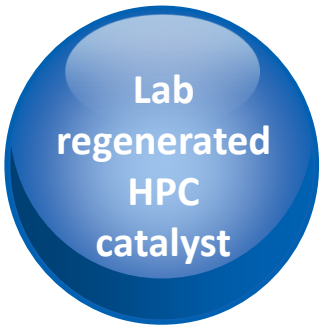
- After Gravity dump, SAS can trace back the position of the catalyst in the reactor.

TO SAS



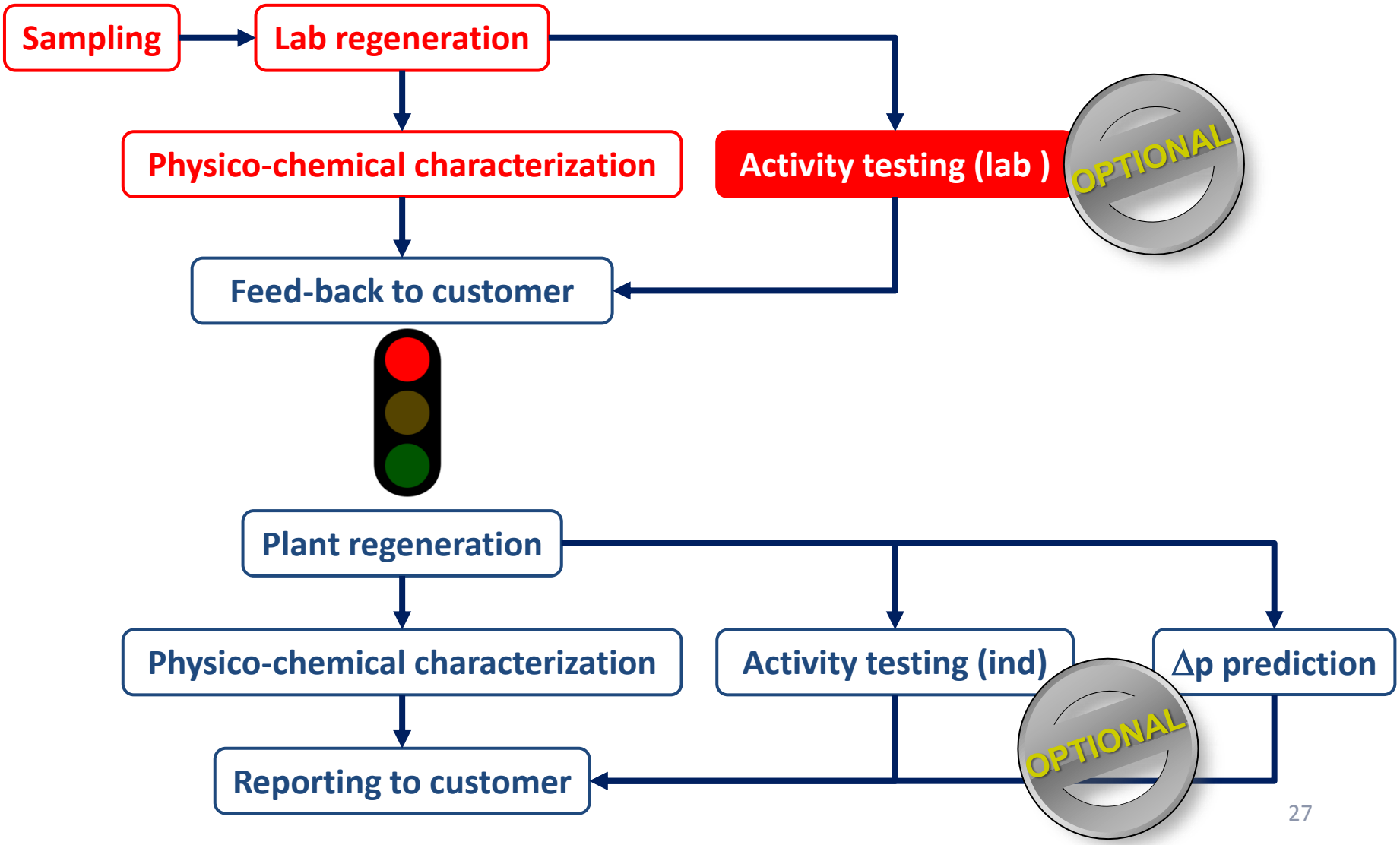
N° BINS	As (wt%)	V (wt%)	Fe (wt%)	Si (wt%)	Na (wt%)	C spent (wt%)	S spent (wt%)	C lab reg (wt%)	S lab reg (wt%)	SA (m^2/g)	Lmm (mm)	%grains < 1.5mm	%grains < 2.0mm
2	0.15	0.00	0.12	0.15	0.09	10.8	13.6	0.1	0.6	141	2.67	6	22
4	0.51	0.00	0.13	0.17	0.07								
6	0.24	0.00	0.13	0.17	0.11	9.5	14.3	0.1	0.8	143	3	4	18
8	0.37	0.00	0.14	0.17	0.14								
10	0.13	0.00	0.11	0.15	0.08	9.1	13.9	0.1	0.6	142	3.04	2	10
14	0.03	0.00	0.10	0.15	0.08	9.5	13.5	0.1	0.6	142			
18	0.02	0.00	0.08	0.16	0.06	10.2	14.2	0.1	0.4	151	2.84	4	15
22	0.01	0.00	0.08	0.15	0.08	10.9	14.1	0.1	0.3	154			
26	0.00	0.00	0.07	0.14	0.05	11.0	13.7	0.1	0.4	160	3.31	3	9
30	0.00	0.00	0.06	0.15	0.05	11.0	13.8	0.1	0.3	162			
34	0.00	0.00	0.06	0.14	0.04	10.3	14.0	0.1	0.3	152	2.85	4	15
38	0.00	0.00	0.07	0.15	0.05	11.4	14.1	0.1	0.3	162			
42	0.00	0.00	0.06	0.16	0.05	12.0	13.4	0.1	0.2	158	3.00	5	14
46	0.00	0.00	0.06	0.15	0.04	12.0	14.0	0.1	0.3	153			
50	0.00	0.00	0.06	0.15	0.05	12.0	13.7	0.1	0.3	160	2.66	10	26
Average	0.10	0.00	0.09	0.15	0.07	10.7	13.9	0.1	0.4	152	2.89	5	16

LAB REGENERATED CATALYST ANALYSIS



- Carbon
- Sulfur
- Surface area
- BCS (Bulk Crushing Strength) and/or SCS (Side Crushing Strength)
- Abrasion loss
- Average length
- PSD (Particle Size Distribution)
- Poisons (As, V, Fe, Si, Na, Ni,…)

HPC TREATMENT



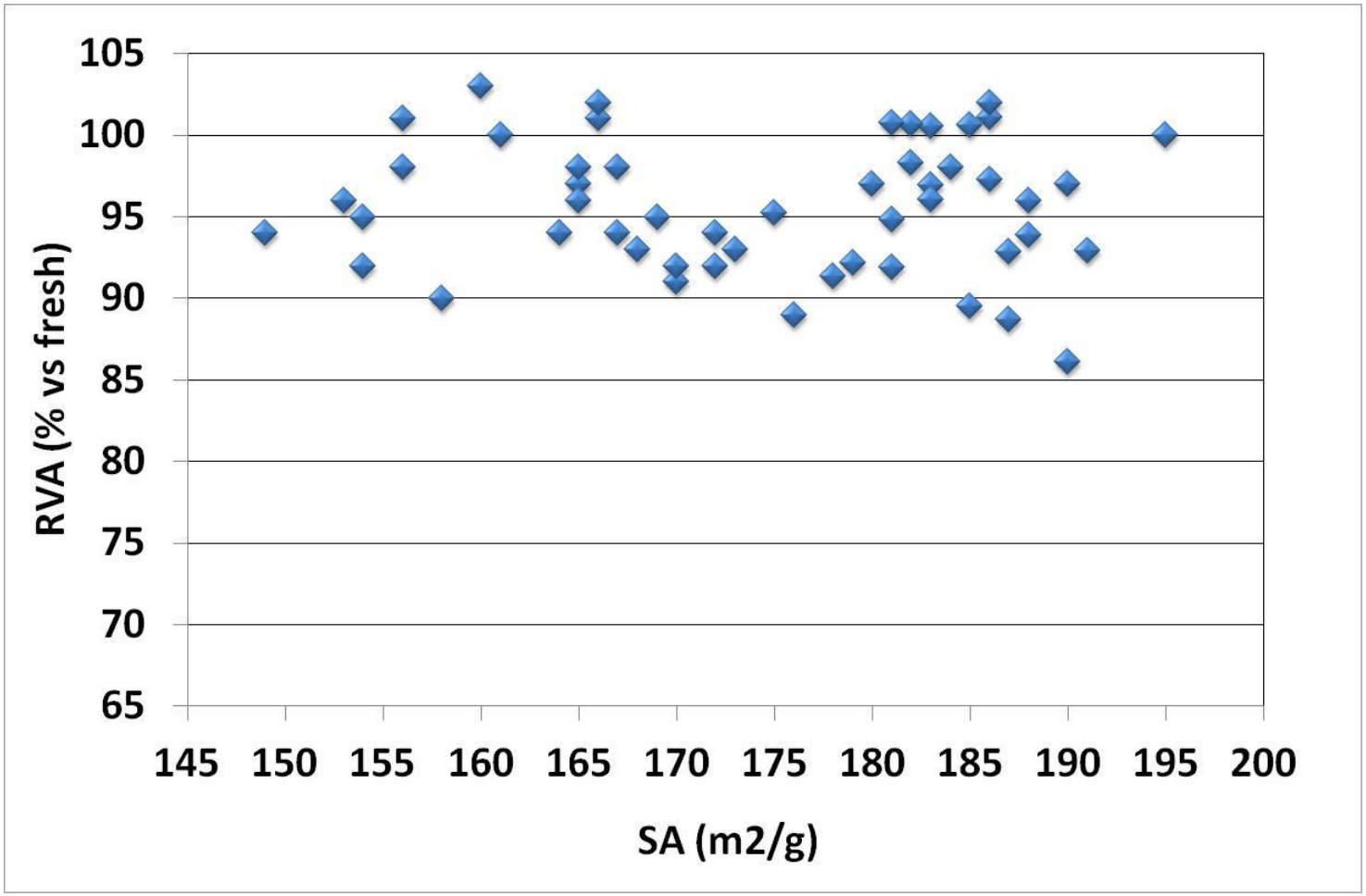
AKTIVITÁS TESZT

- Az aktivitás helyreállíthatósága nagyban függ a használt katalizátor állapotától.
- A katalizátor aktivitása rosszul számszerűsíthető az analitikai eredmények alapján.

A közvetlen aktivitásmérés elengedhetetlen az újrahasznosíthatóság megállapításához.

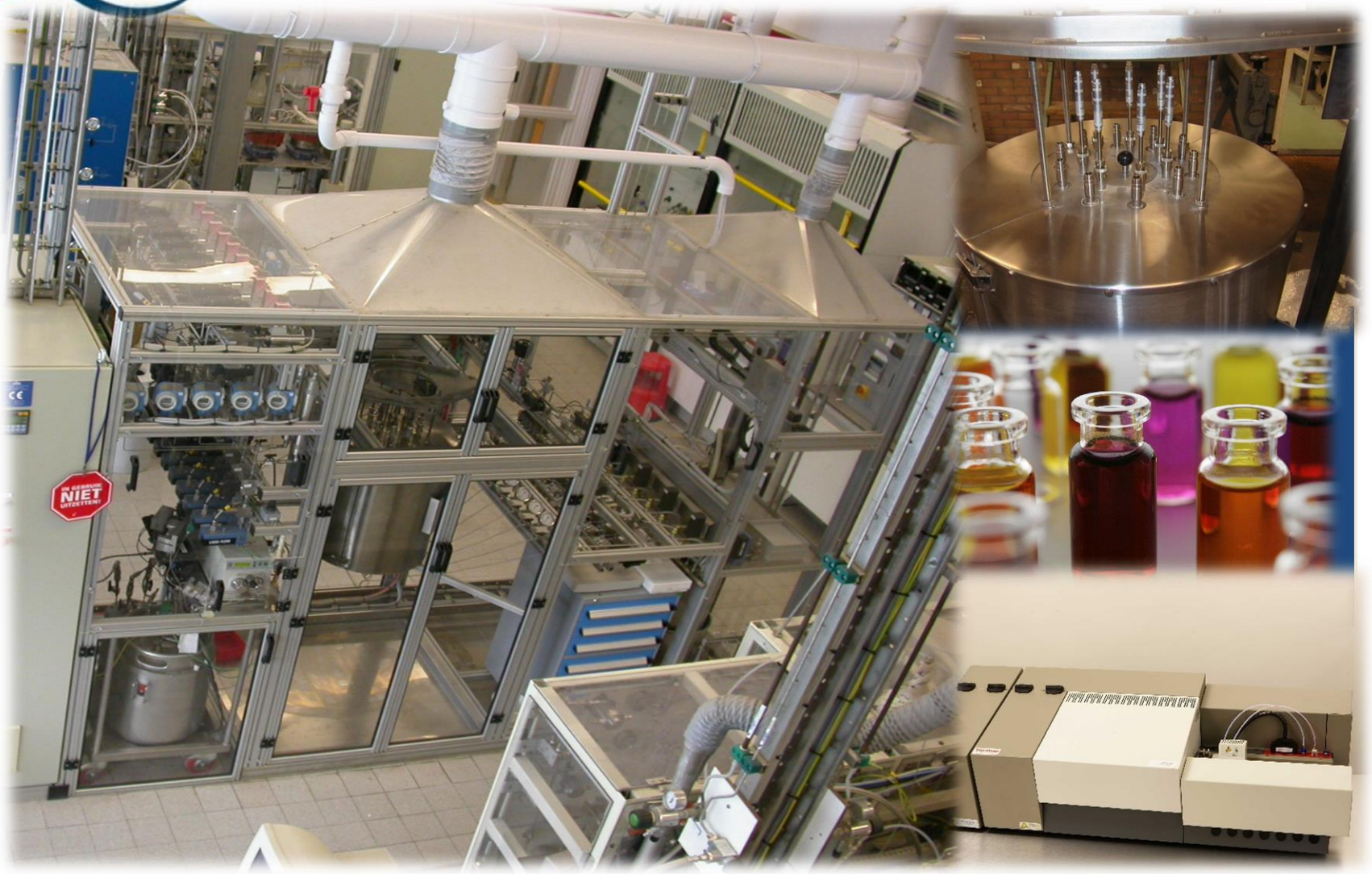
RVA / RWA (%) measured correlates directly to reactor WABT (°C).

ACTIVITY VS SURFACE AREA KF757



No relevant metals contamination on every batch

ACTIVITY TEST



MTU OVERWIEV

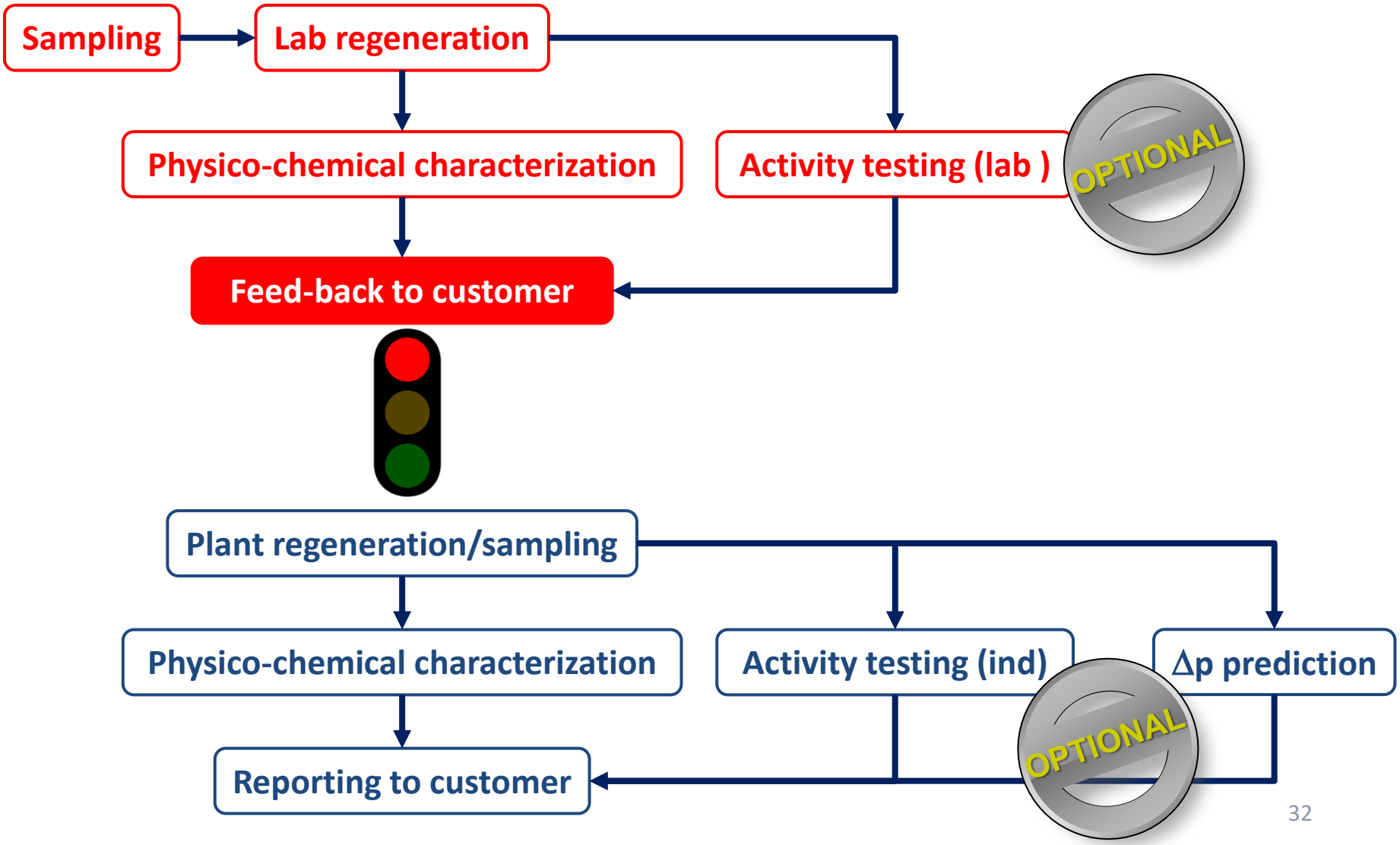
Furnace and 10 reactors in parallel

Sampler (and degassing section)

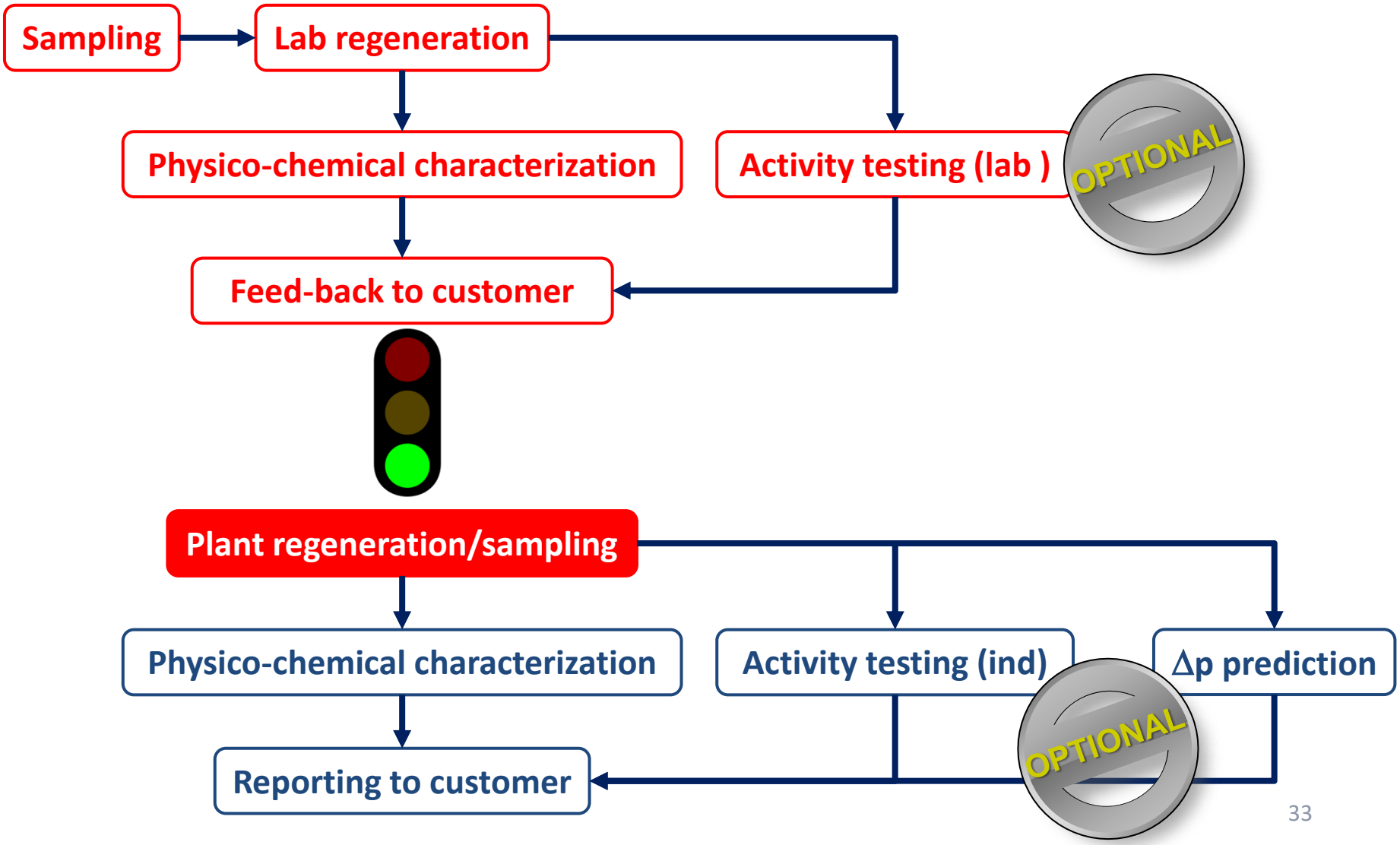
HDS – ULSD (10 -100 ppm S)

Capacity: 80 samples/month

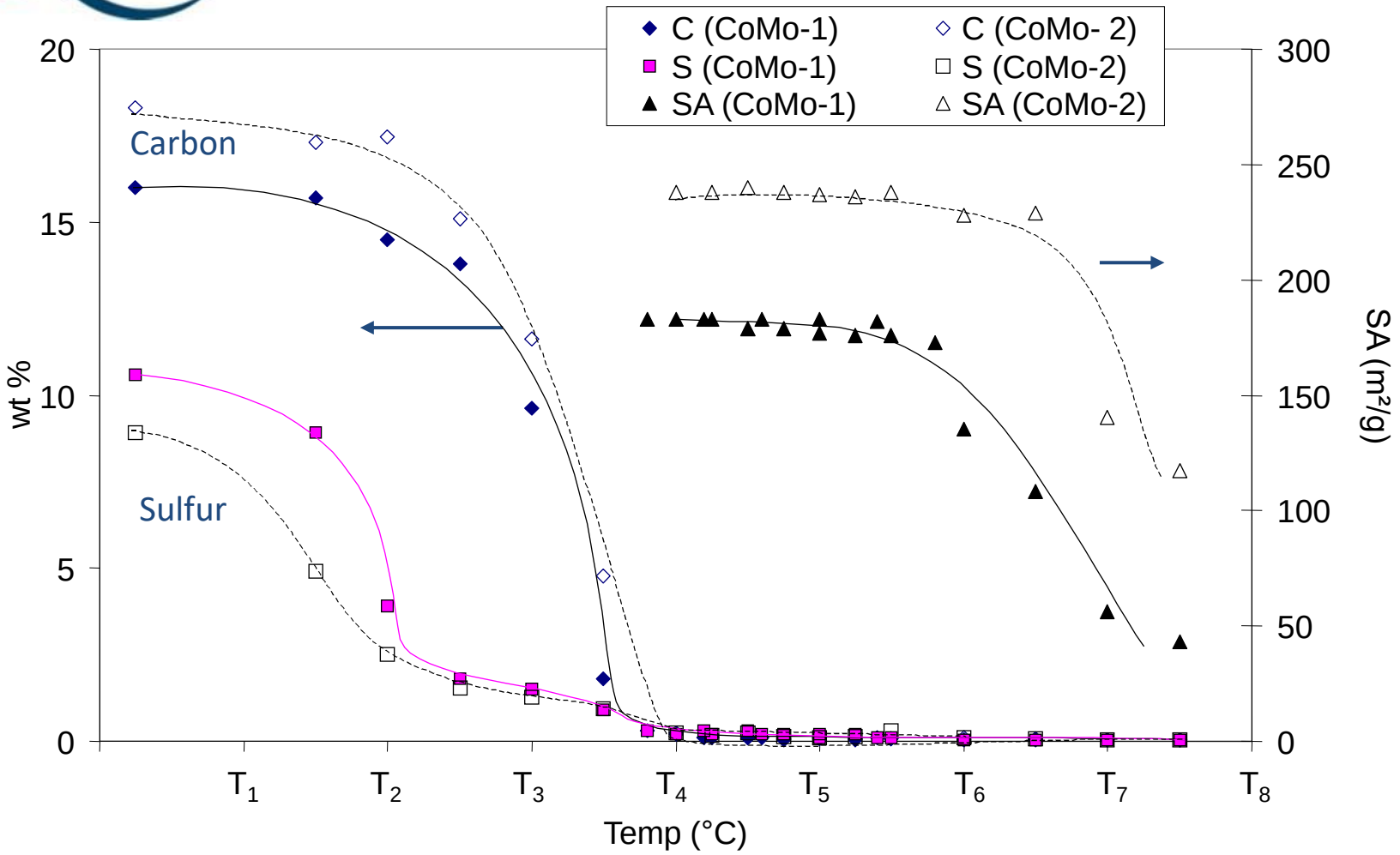
HPC TREATMENT



HPC TREATMENT



REGENERATION PARAMETERS OF HPC



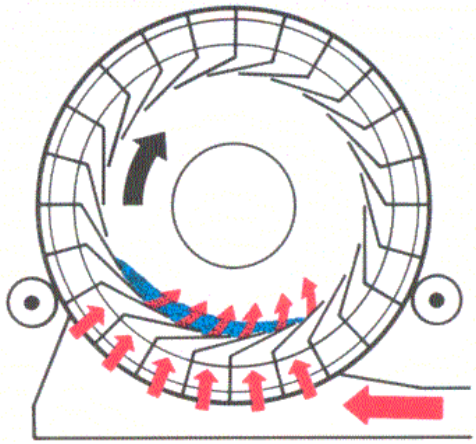
2 Commercial Hydrotreating catalyst - CoMo on Al_2O_3

Tailor-made recipes for each individual catalyst

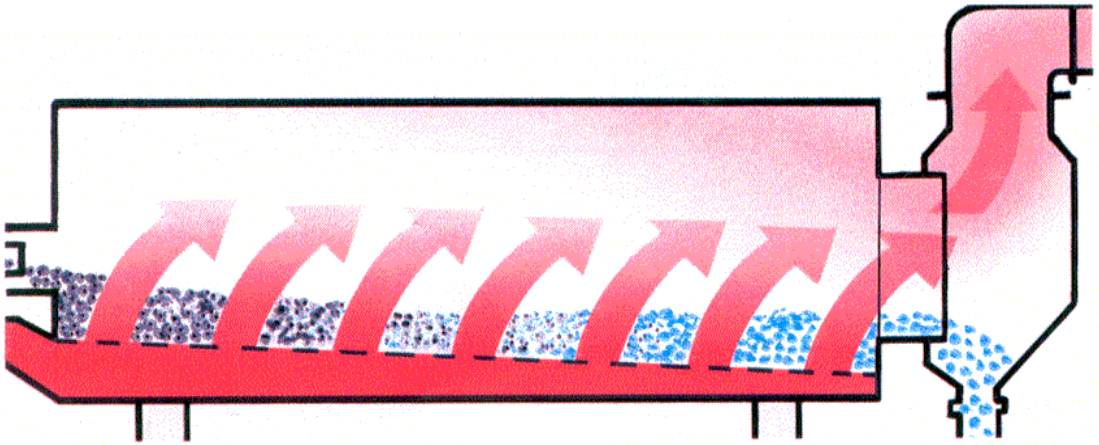
ROTO-LOUVRE

Maximális aktivitás visszanyerés:

- homogén regenerálás
- szigorú hőmérséklet kontrol
- Gyors hő és H_2O/SO_2 eltávolítás

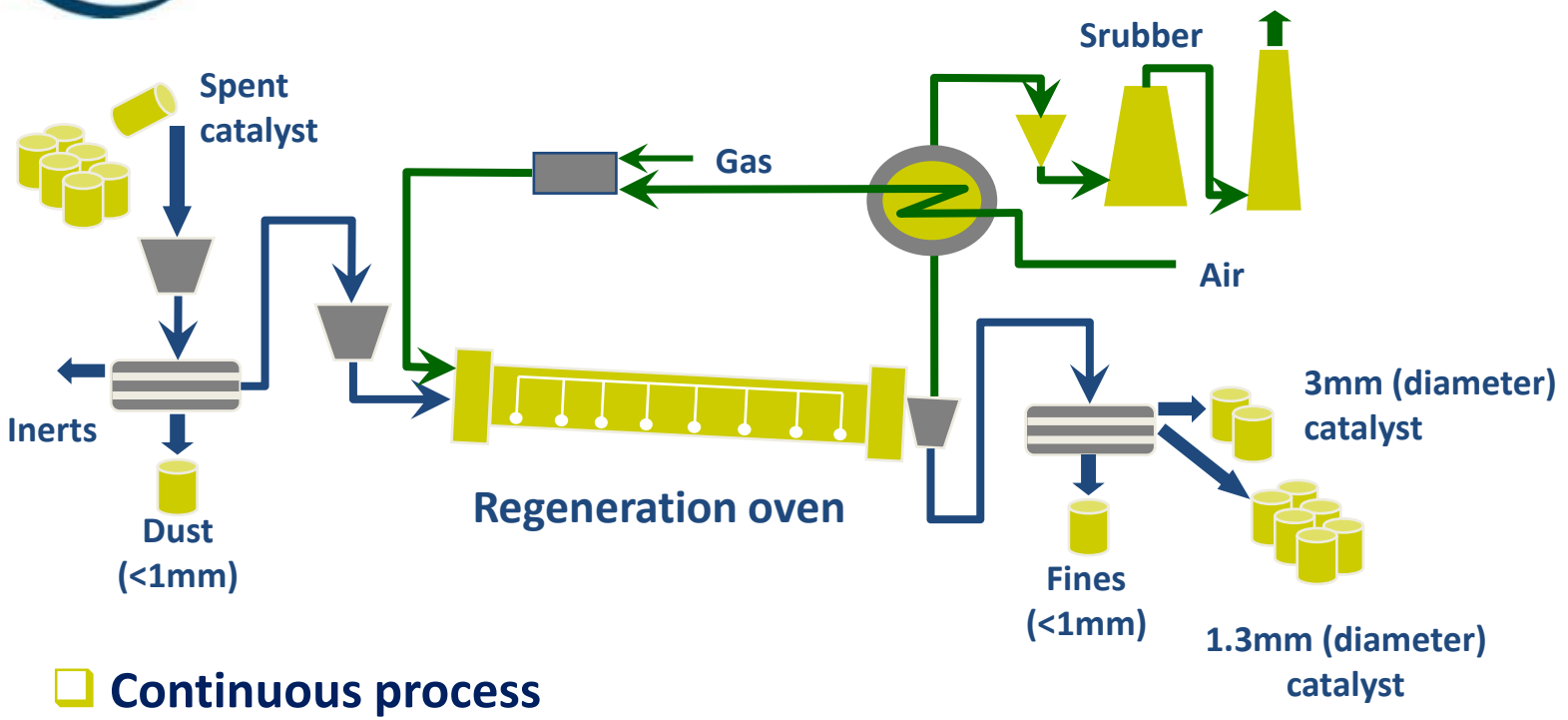


Cross view



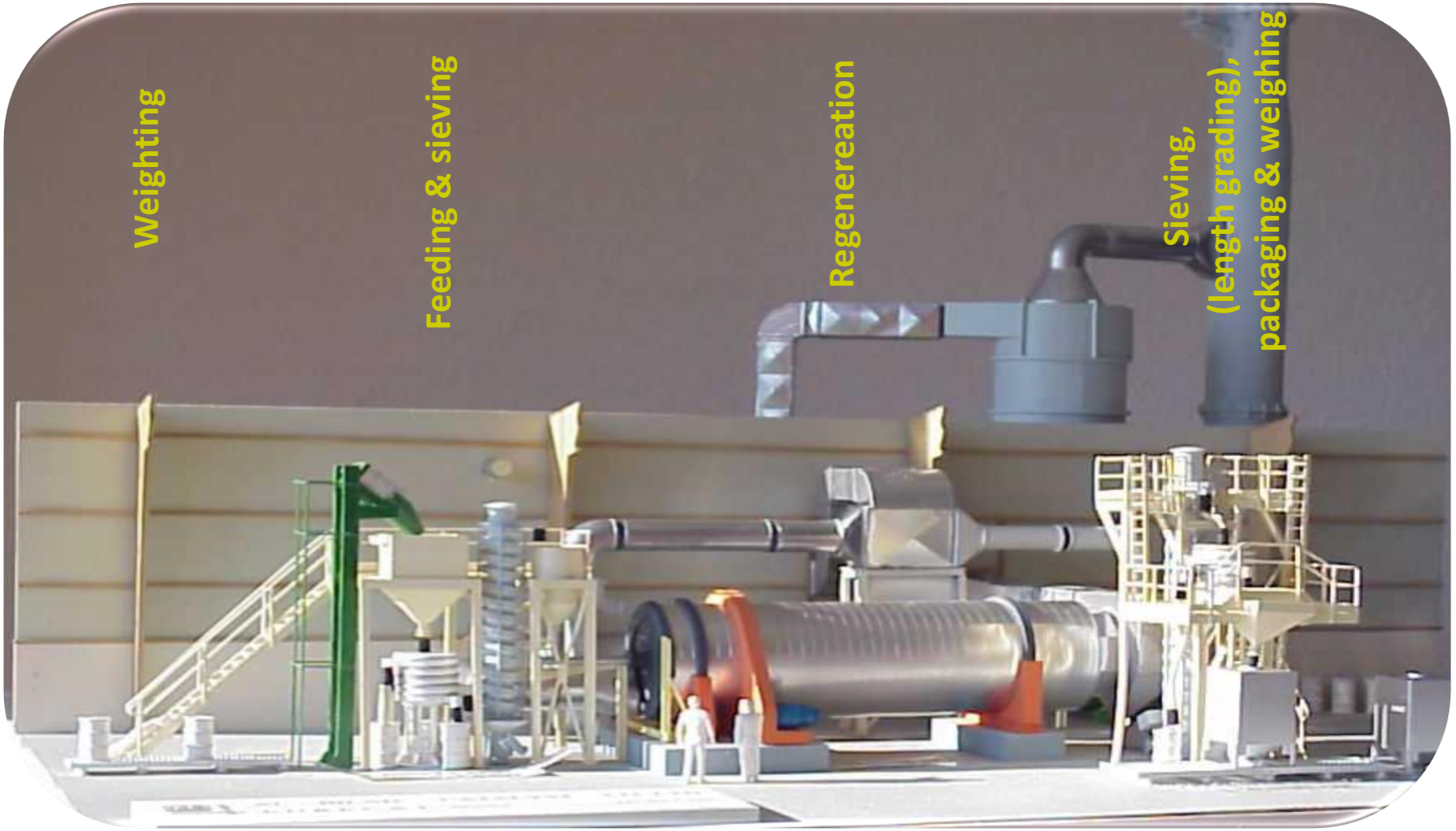
Side view

REGNERATION FLOW SHEET



- Continuous process
- High activity recovery (≥ 95 % SA retention)
- Fines guaranteed to be less than 1 wt%
- Fine-tuned to each different catalyst
- Catalyst evaluation (QC)

PLANT REGENERATION



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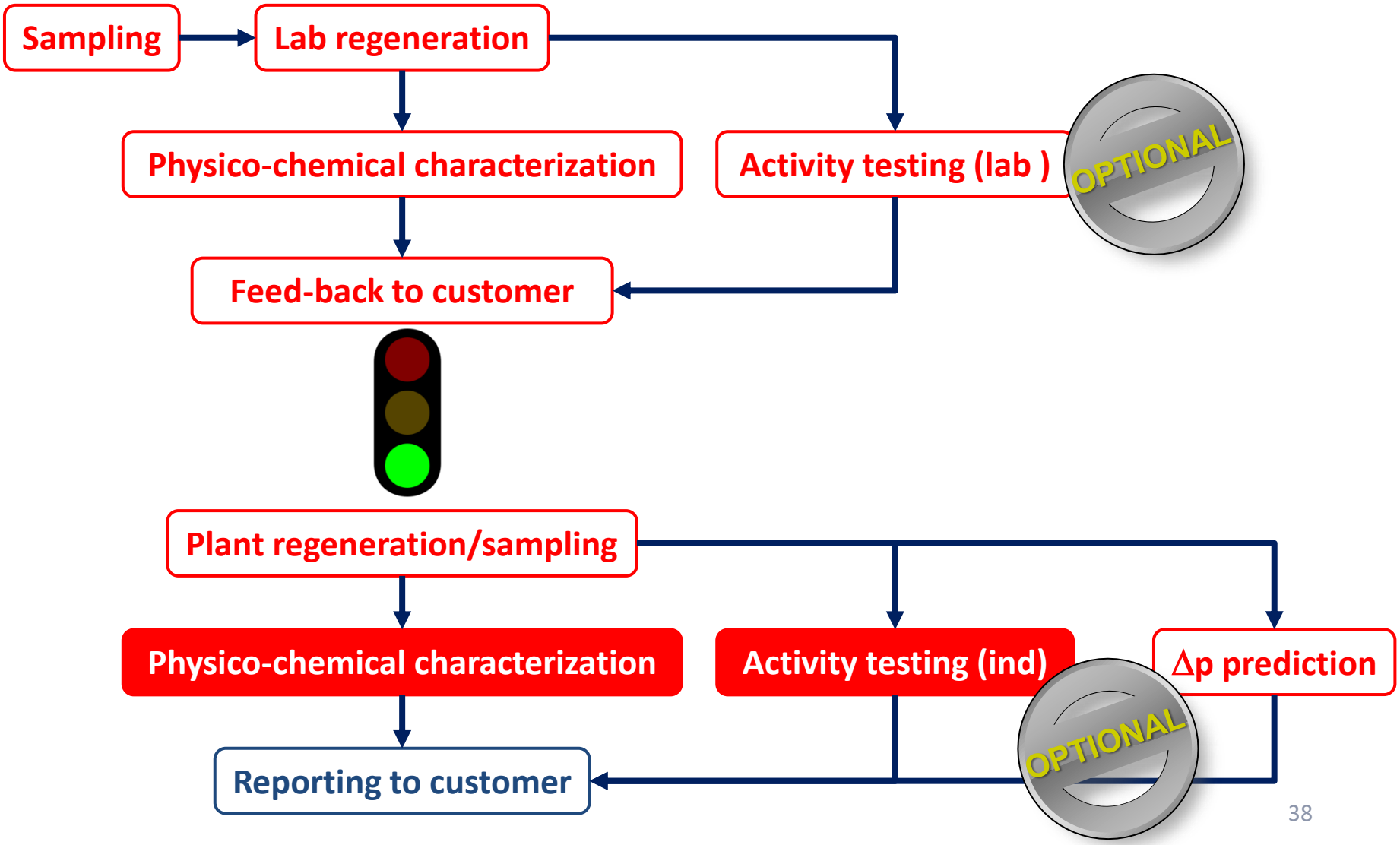

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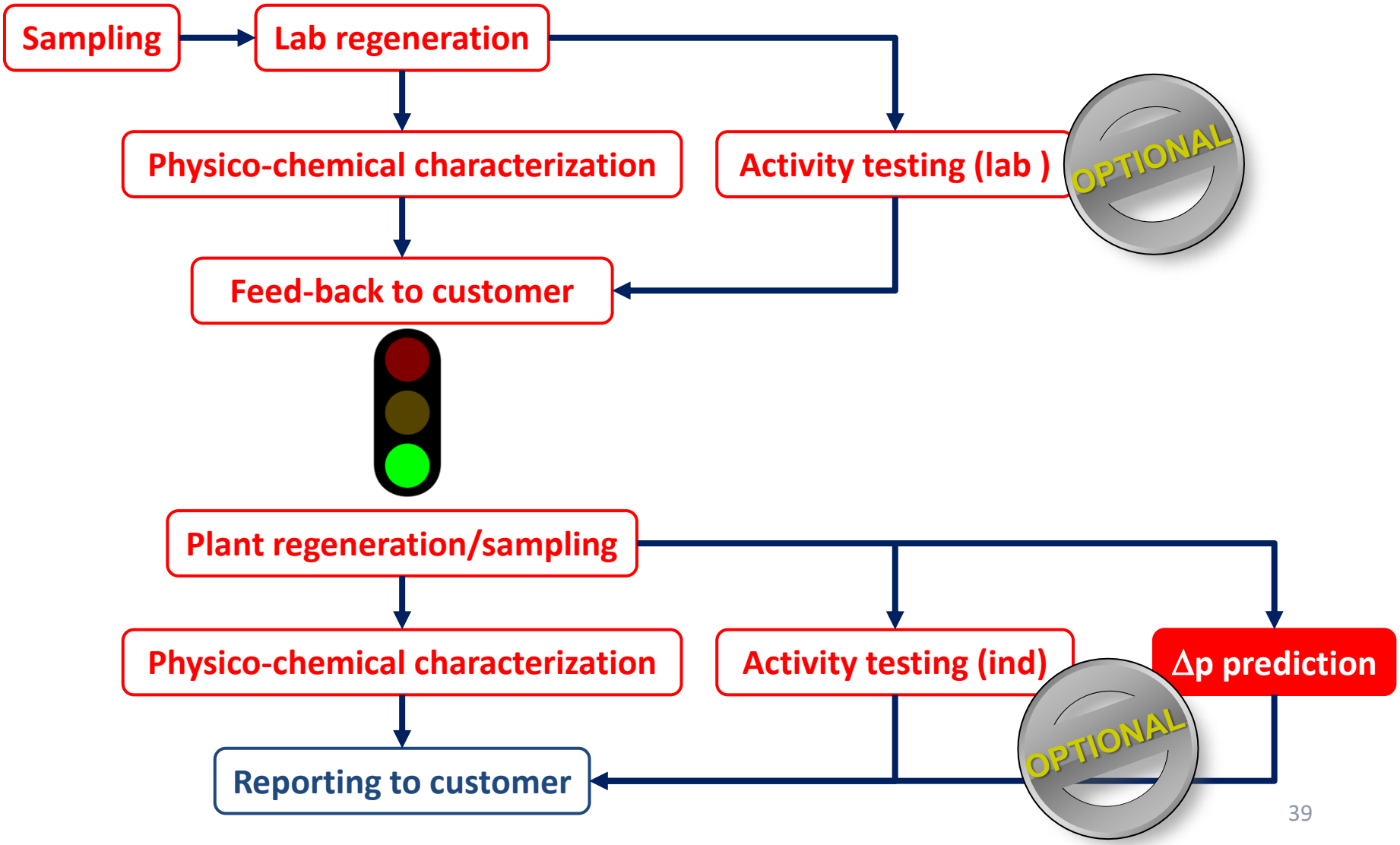


Catalyst Performance: Pressure drop

- Eurecat has developed unique pressure drop measurement to predict mixed phase delta P in refinery reactor conditions.

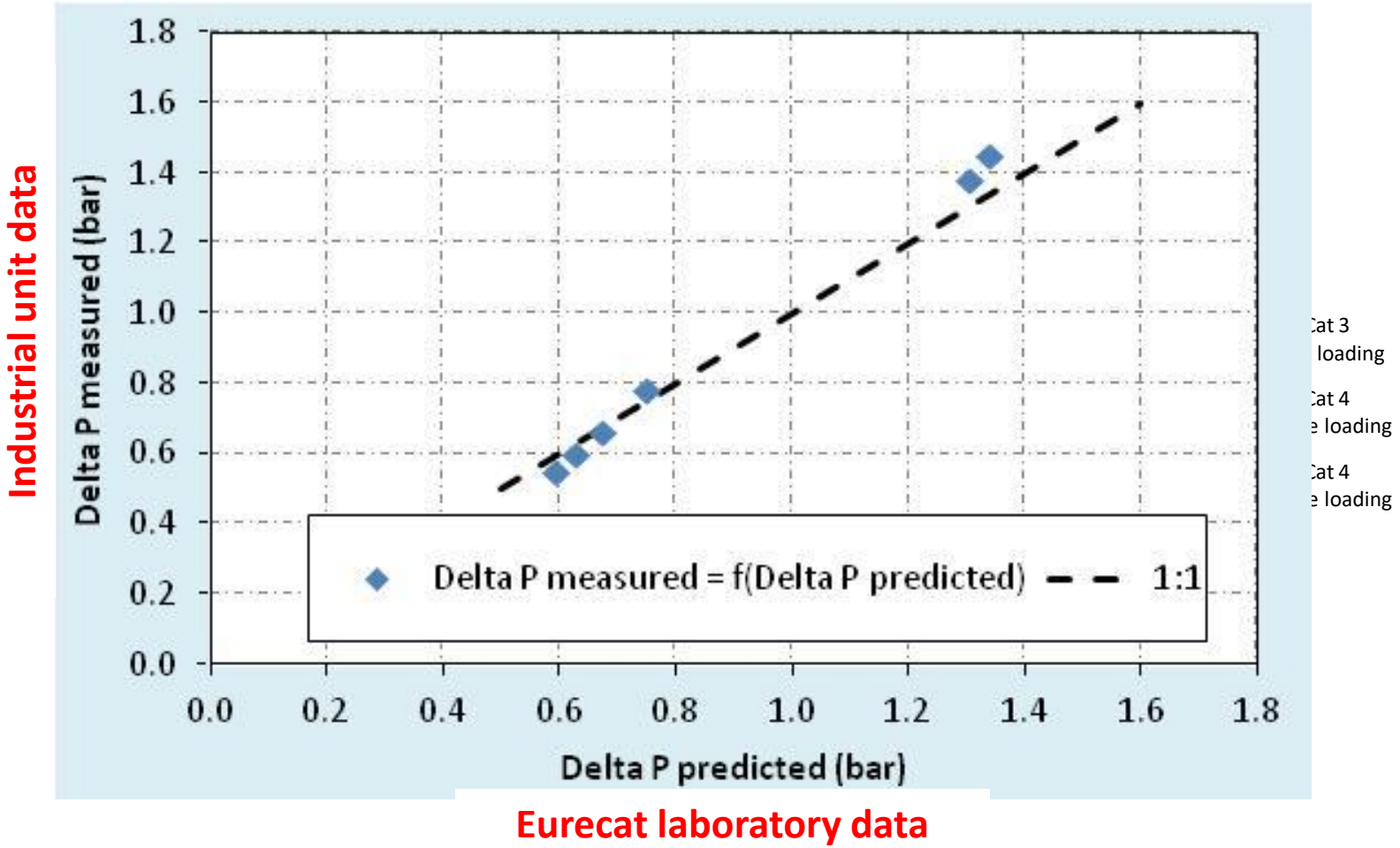
Eurecat
Lab gas phase ΔPref measurement
+ Lab gas phase ΔP measurement
+ Correlation

Commercial
ΔPref (bar)

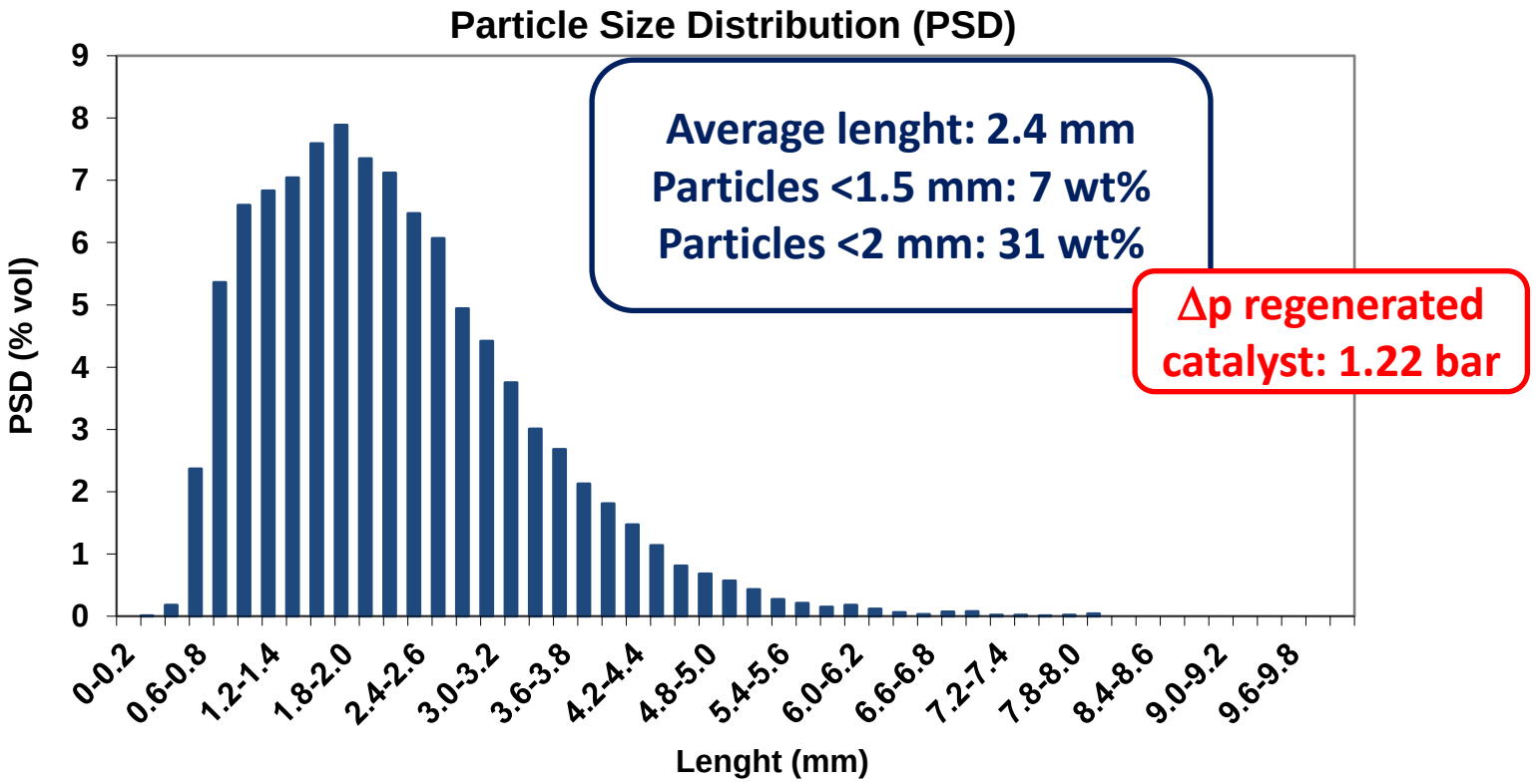
Predicted ΔP (bar)

Catalyst Performance: Pressure drop

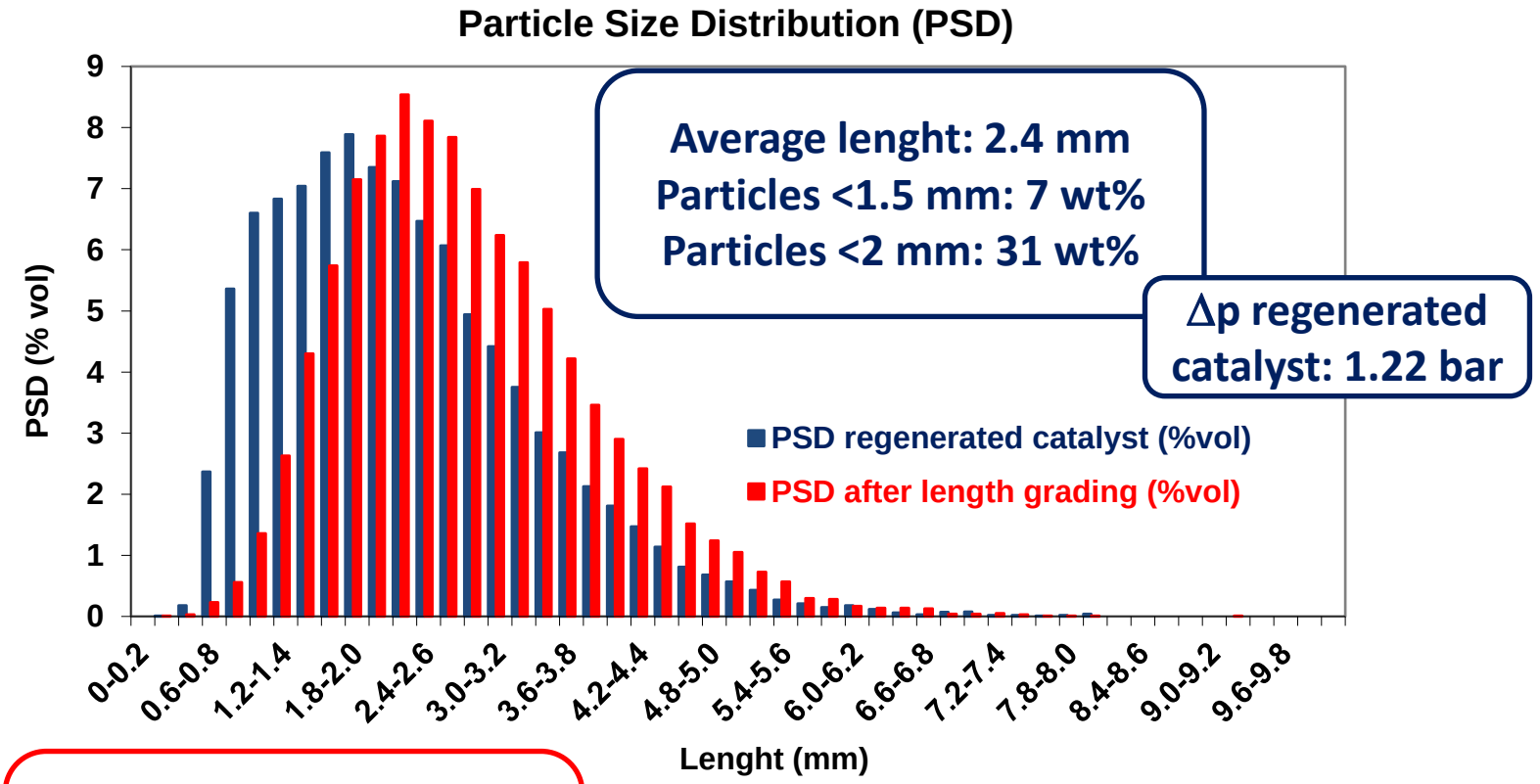
- Tool has been validated in refinery units (multibed, dense/sock):



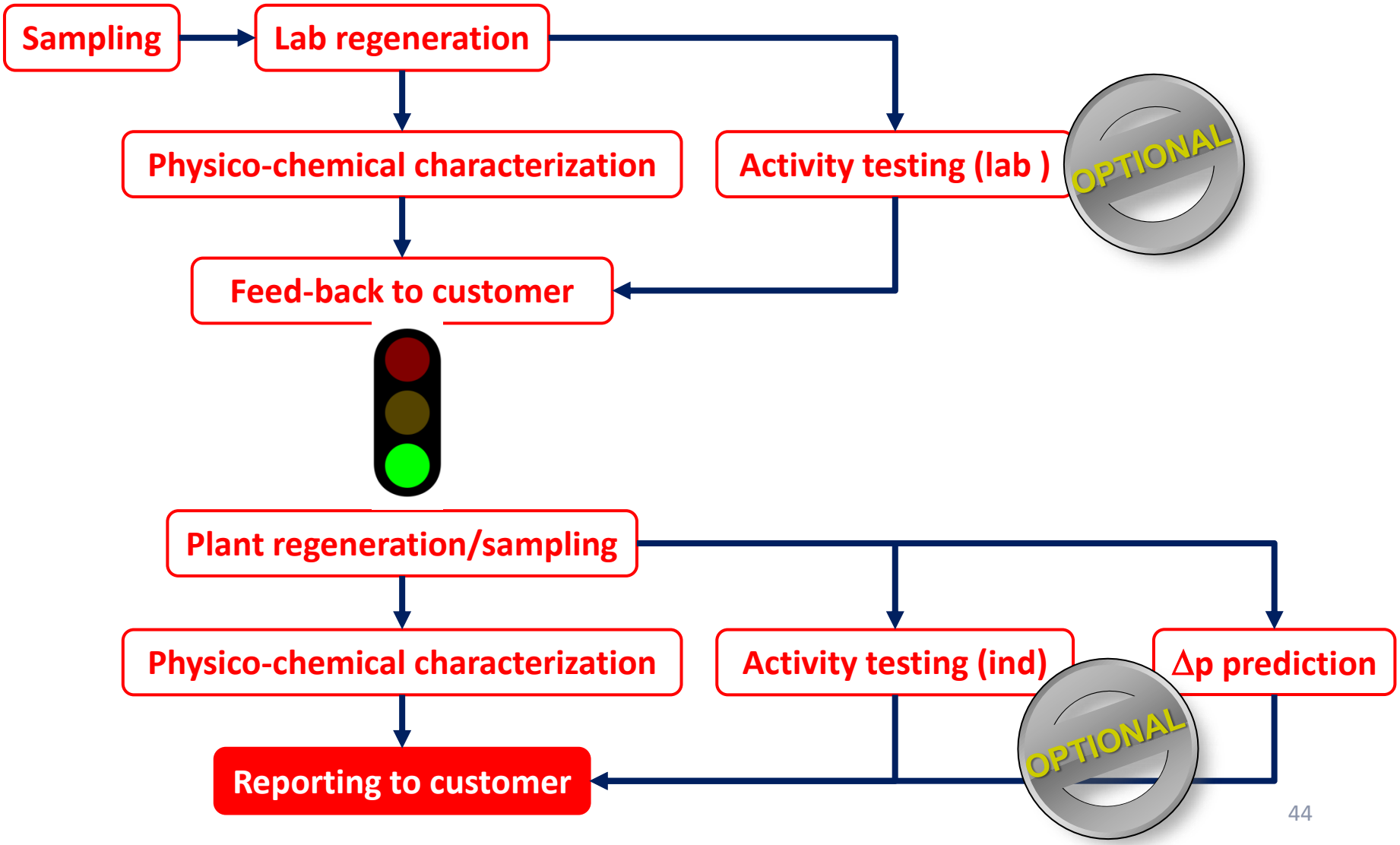
PARTICULE SIZE DISTRIBUTION VS PRESSURE DROP



LENGTH GRADING VS PRESSURE DROP



HPC TREATMENT



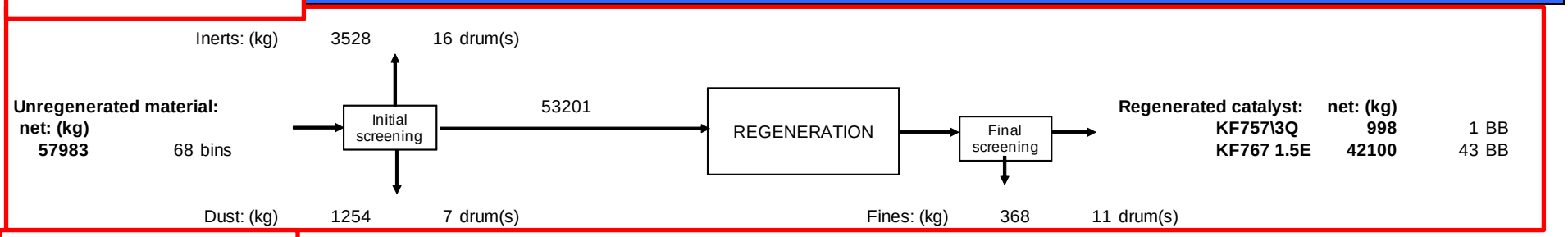
Regeneration report

1XXXX/A KF757 3Q

Comments:
FB 66-68 : 40 wt % dust

PO number:
End of treatment: 31/1/2013

Mass balance



Catalyst characteristic

		ab regen:	Spec:	Regen:	Notes:
LOI (wt%):	18.3	-	-	-	
Smoke test:	Yes	-	-	-	
HC/other volatiles (wt%)	2.9	-	-	-	
C (wt%):	14.2	0.1	0.5	0.1	
S (wt%):	12.0	0.4	0.9	0.5	
Surface area (m²/g):	-	140	137	140	
BCS (MPa):	-	1.60	1.44	1.58	
Average length (mm):	-	3.0	2.8	3.0	
< 2 mm (%):	-	17	20	15	
< 1.5 mm (%):	-	6	10	5	
Fines (wt%):	-	-	-	<0.1	

Metals contaminants:		Notes:
As:	<0.01	
V:	<0.01	
Fe:	0.2	
Si:	0.1	
Na:	0.11	
Ni:	<0.01	
Regenerated densitie		Notes:
CBD (t/m³)	0.843	
SD (t/m³):	0.784	
PDLD (t/m	0.903	
VF (-):	0.325	

Regen/Lab REACT™:		Notes:
RVA (%):	92	vs fresh
RWA (%):	98	vs fresh
Δp (bar):	1.01	

Performance prediction

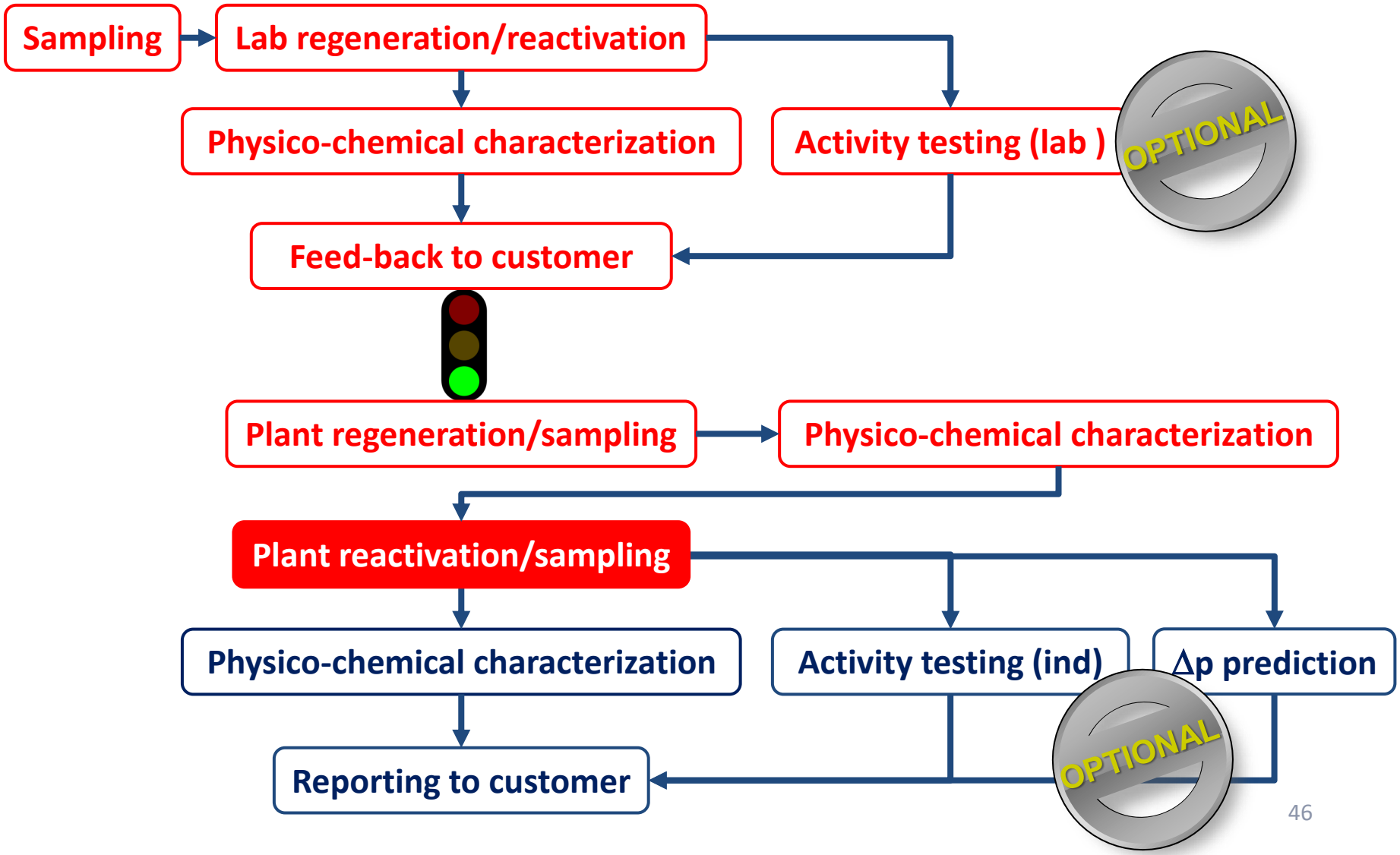
Loading parameters

Additional analysis results can be published via a stock database directly accessible via the Internet

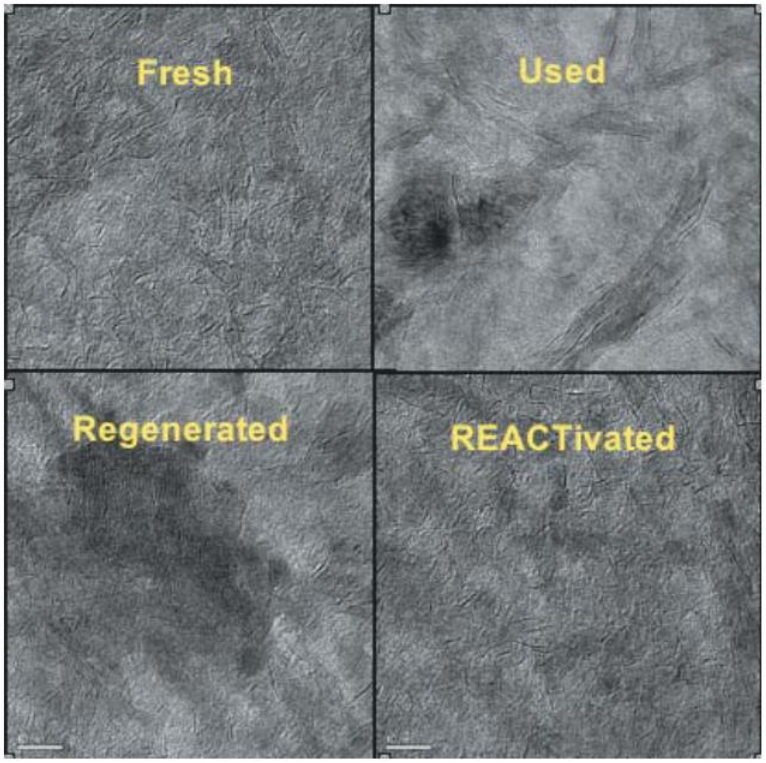
1XXXX/A KF757 3Q	Spent:	Lab regen:	Spec:	Regen:	Notes:
LOI (wt%):	18.3	-	-	-	
Smoke test:	Yes	-	-	-	
HC/other volatiles (wt%):		-	-	-	
C (wt%):	14.1	0.1	-	0.1	
S (wt%):	12.7	0.3	-	0.8	
Surface area (m²/g):	-	176	-	170	
BCS (MPa):	-	-	-	1.2	
Average length (mm):	-	-	-	4.8	
< 2 mm (%):	-	-	-	1	
< 1.5 mm (%):	-	-	-	0	
Fines (wt%):	-	-	-	<0.1	

Metals contaminants:		N
As:	<0.01	
V:	0.01	
Fe:	0.12	
Si:	0.15	
Na:	0.15	
Ni:	<0.01	
Regenerated densitie		Notes:
CBD (t/m³)	0.727	
SD (t/m³):	0.676	
PDLD (t/m	0.801	
VF (-):	0.382	

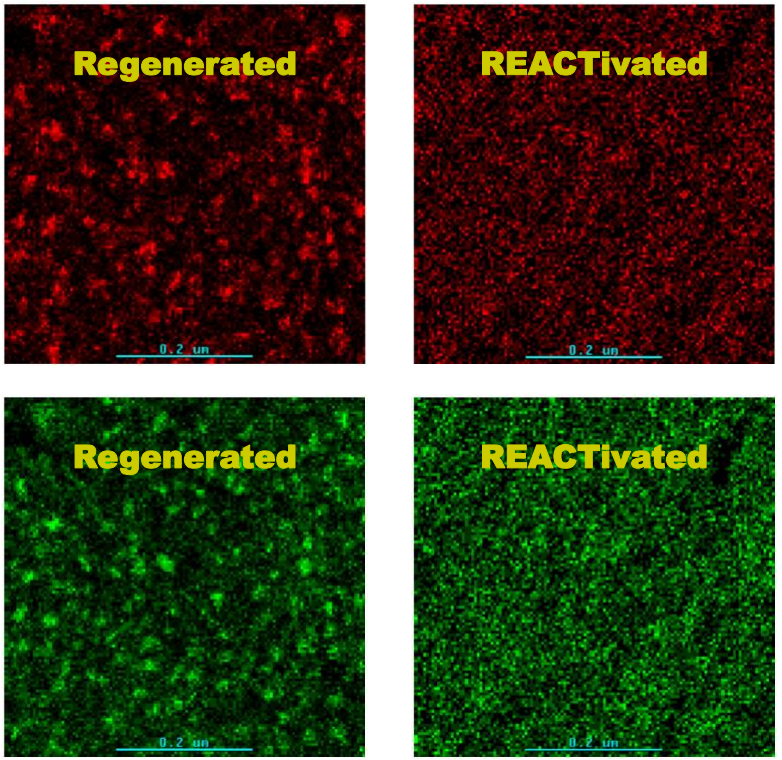
HPC TREATMENT



REACTIVATION: STARS CATALYST



TEM micrograph of CoMo catalyst



STEM-EDX maps of Co (red) and Mo (green)

➡ **Redispersion of the active phase**

E. Brevood, Catalysts Courier 60 (2005) 4.
S. Eijsbouts, A.A. Battiston, G.C. van Leerdam, Catal. Today 130 (2008) 361.

EURECAT REACT™ STEPS

Regenerated STARS Catalyst

REACT

Sampling & analysis

Sieving

Fines

Packing & weighting

REACT STARS Catalyst

Experience: more than 25 000 tons delivered

REACT recipie cannot be disclosed

REACT report

1XXXX/A KF767 1.5E

Comments:

PO number:

End of treatment: 1/2/2013

Mass balance

Oxidic catalyst:
net: (kg)
42100 43 BB

→ REACT™ REJUVENATION →

REACT™ catalyst:
net: (kg)
50054 43 BB

Catalyst characteristic

1XXXX/A KF767	Specs:	REACT™:	Notes:
C (wt%):	-	-	
S (wt%):	-	-	
Surface area (m^2/g):	-	144	
Average length (mm):	2.8	2.85	
< 2 mm (%):	20	19	
< 1.5 mm (%):	10	6	
Fines (wt%):	<1.0	0.2	
BCS (MPa):	-	1.55	

Loading parameters

REACT™ densities:		Notes:
CBD (t/m^3)	1.039	
SD (t/m^3):	0.966	
PDLD (t/m^3)	1.111	
VF (-):	0.338	

Performance prediction

	REACT™	Notes:
RVA (%):	95	vs fresh
RWA (%):	101	vs fresh
Δp (bar):	1.04	

SPECIFIC EURECAT TECHNOLOGIES

Total sulfiding:

- CoMo, NiMo, NiW catalysts (TOTSUCAT)

Reduction:

- Pd, Pt, PtPd, PtRe, PtSn, Ni, Cu, CuZn...

Selectivation:

- Pd, Pt, PtRe, Ni, Activated carbons

Chlorination:

- Pt, PtRe, PtSn, PtPd

Customized treatments:

- Confidential (treaments for various manufacturers and end-users)

SPECIFIC EURECAT TECHNOLOGIES

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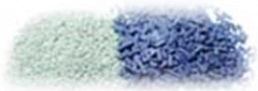
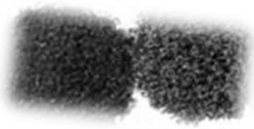
UNIT OPERATION/CATALYST LIFE CYCLE





Metal reclaim

Fresh → Loading → Startup → Run → Unloading → Regeneration Reactivation

~~In-situ sulfiding~~

Activity
Δp

SMARTDUMP
CARBODUMP

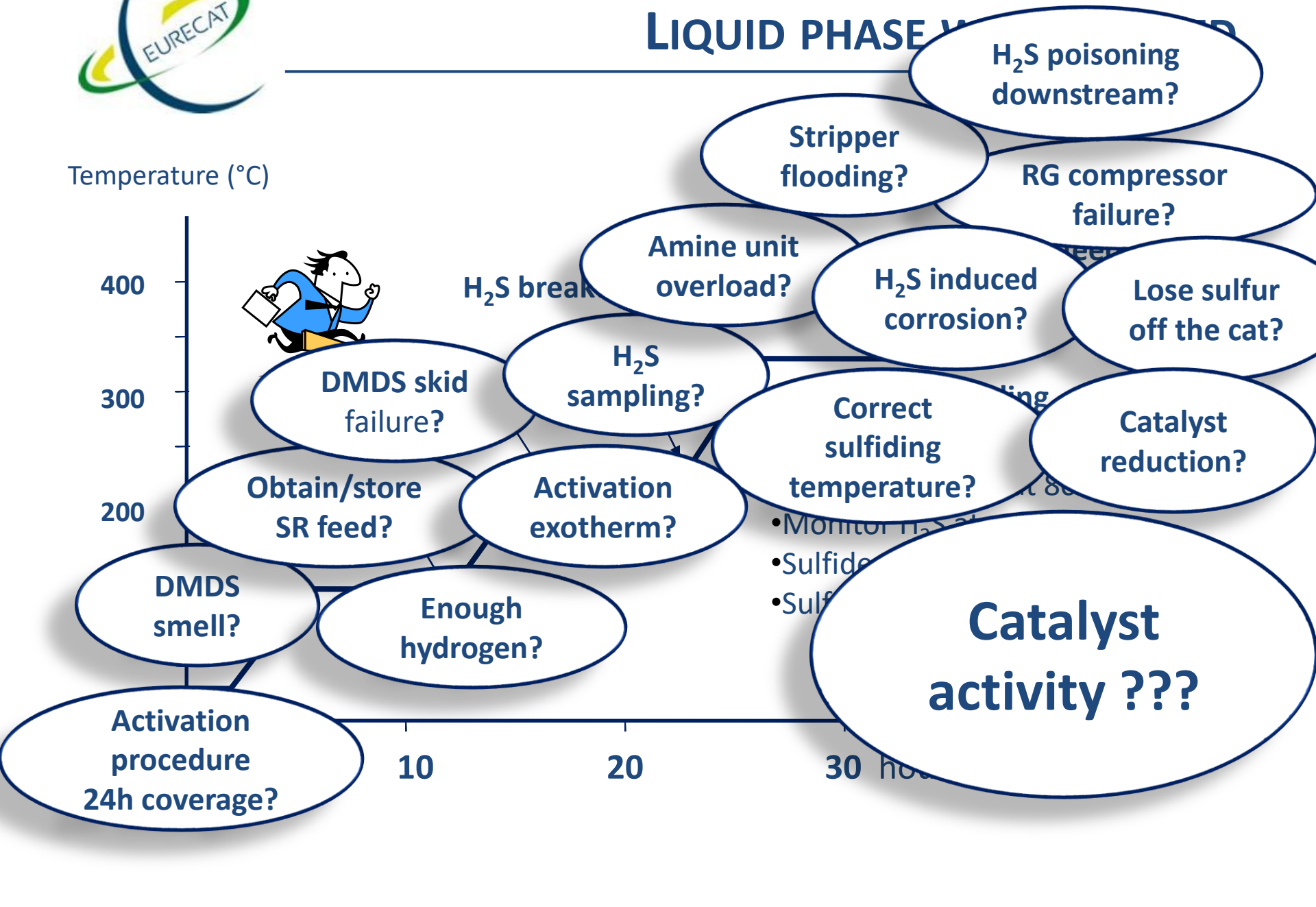
Ex-situ Sulfiding: TOTSUCAT

Initial activity Δp prediction

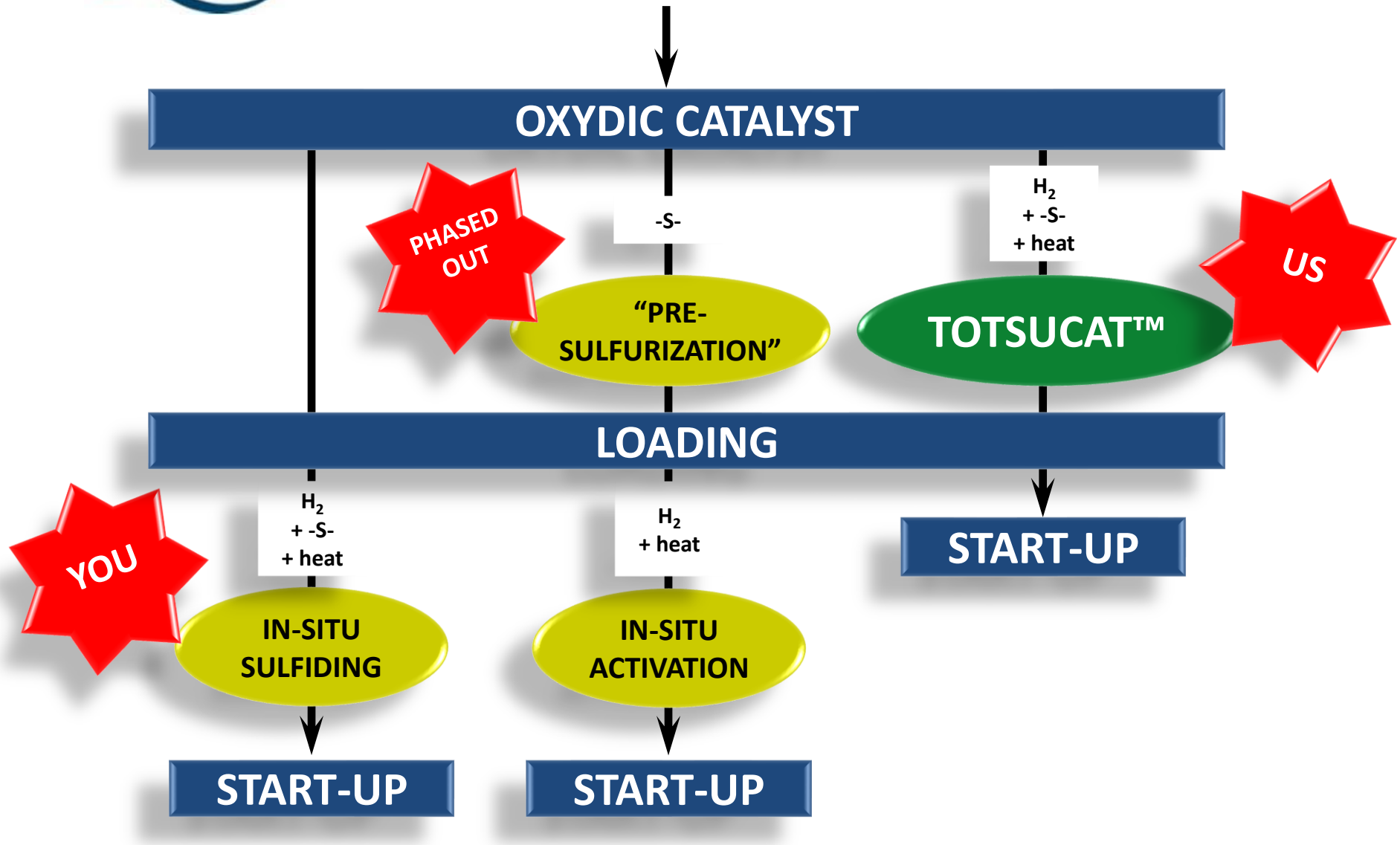
REUSE
CASCADING
POOL
RESALE

STARTUP WITH IN-SITU SULFIDING

LIQUID PHASE



CATALYST SULFIDING



TOTALLY SULFIDED CATALYST (TOTSUCAT) VS PRESULPHIDING (ACTICAT, SULFICAT…)

	TOTSUCAT™	Presulphiding
Catalyst delivered already at full activity?	YES	NO
In situ activation required?	NO	YES
Sulphiding conditions?	Optimized for each catalyst type	Fixed by in situ procedure
Typical start up time	8h	24h+
Need to perform final sulphiding step at 320-350°C (CoMo/NiMo)?	NOT APPLICABLE	YES
Activation exotherm?	NOT APPLICABLE	YES
Additional H_2 required for activation?	NOT APPLICABLE	YES
Typical H_2S level in recycle gas during startup	< 500 vppm	1-2 vol%

TOTSUCAT® BENEFITS

Easy and quick

No DMDS or other sulfiding chemical to handle

No additional H_2 needed at startup

No activation exotherm

No need to reach final sulfiding temperature

No recycle gas sampling / H_2S monitoring

No impact of H_2S on downstream units

Fail-proof: upsets will not damage the catalyst

Maximum catalyst performance

Typical TOTSUCAT® Applications

Critical Path Units

- Downtime cost for in situ sulfiding often outweighs Totsucat cost.
- Totsucat with Amine passivation for Hydrocrackers saves precious startup time.

Temperature Limited Units

- Some units cannot achieve correct sulfiding temperature: in situ sulfided catalysts will not perform as designed.

Before Sulfur Sensitive Units

- Reformers and Isom units contain precious metal catalysts that are sensitive to H_2S contamination.

Hydrogen Limited Units

- Some units are difficult to sulfide, as there is not enough H_2 available at the time of startup.

TOTSUCAT® COMMERCIAL EXPERIENCE

Hydrotreating
- CoMo, NiMo, NiCoMo

Selective Hydrogenation
- NiMo and CoMo

Pyrolysis Gasoline
- NiMo and CoMo

Hydrocracking
- NiMo and NiW

Lube / Wax Hydrofinishing
- NiMo and CoMo
- NiW

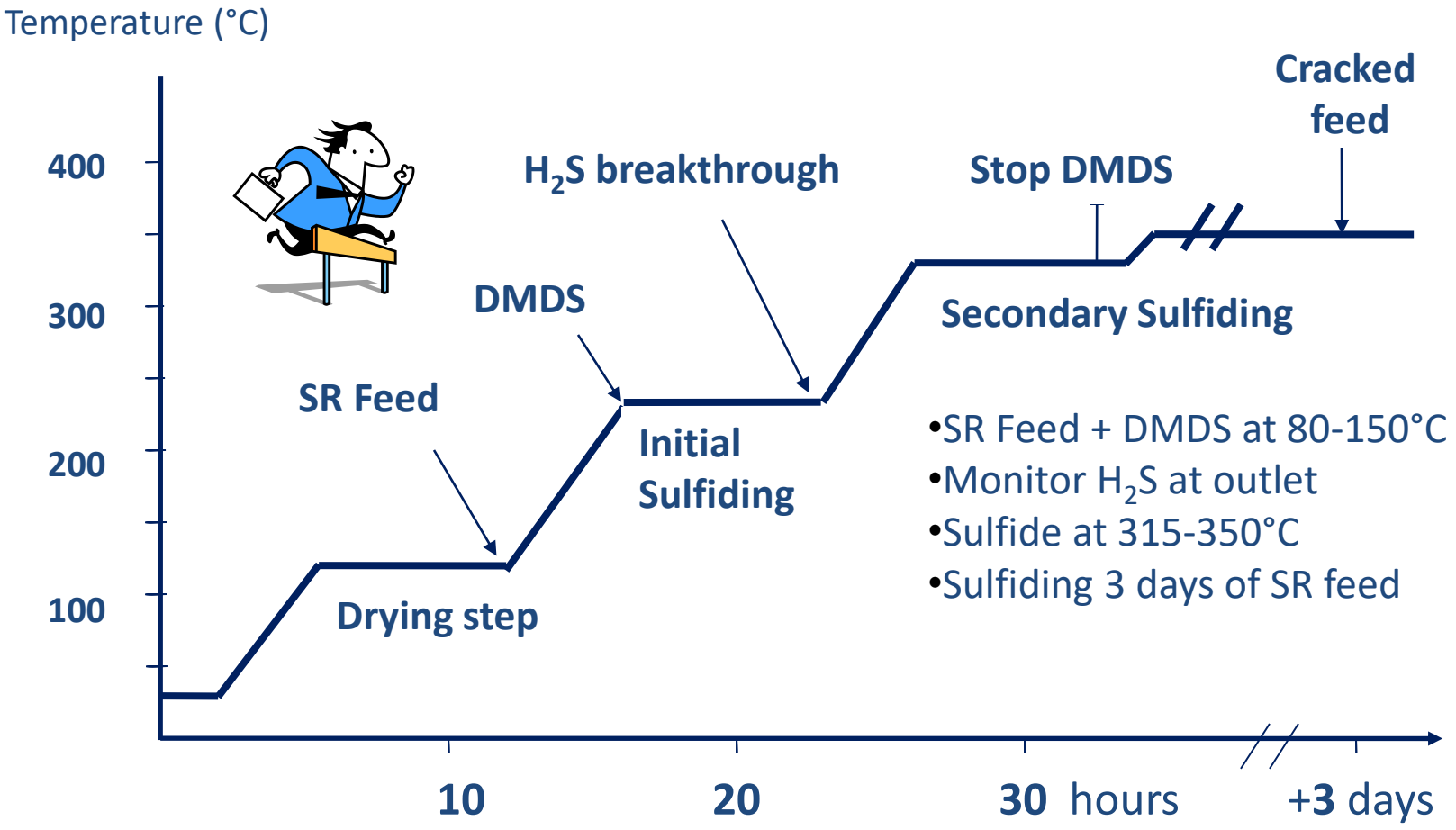
Other…
- Biofuels
- Contaminant traps
- …..

Over 1200 Totsucat lots treated worldwide: 30 000 tons of catalyst

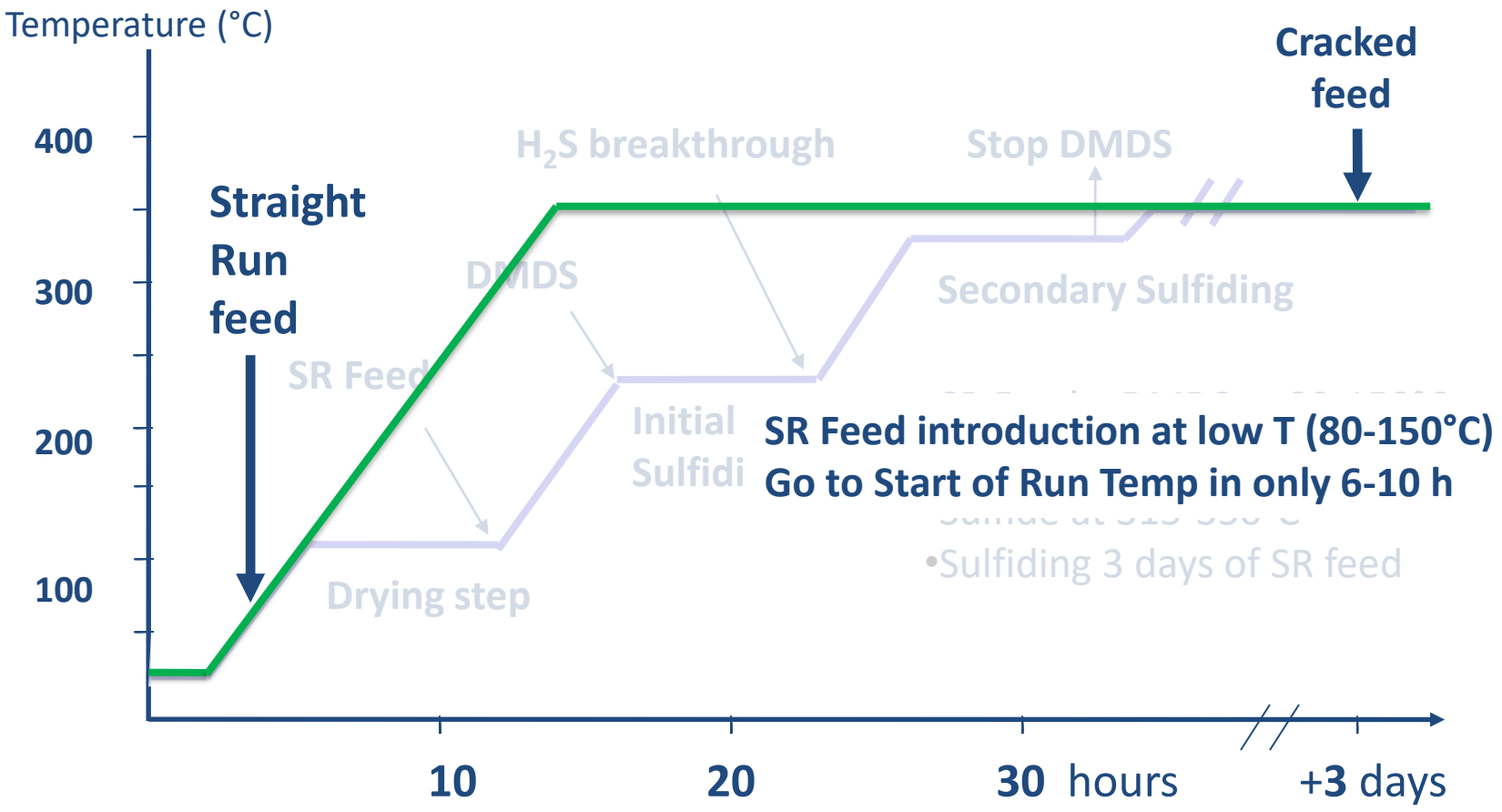
TOTSUCAT® BRANDS

TOTSUCAT – G	• Naphtha, Kero, FCC gasoline, Tail Gas, Lubes
TOTSUCAT – D	• (Ultra Low Sulfur) Diesel
TOTSUCAT – N	• Hydrocracking Pretreat
TOTSUCAT – E	• FCC Pretreat
TOTSUCAT – HC	• Hydrocracking
TOTSUCAT – CFP	• Light Cycle Oil, Heavy Cycle Oil • Coker and Visbreaker Naphtha / Diesel

STARTUP WITH IN-SITU SULFIDING



STARTUP WITH TOTSUCAT® G/D/E/N/HC

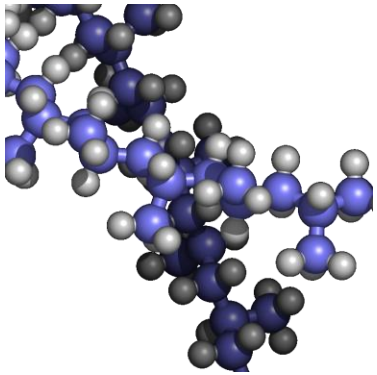


- **Startup Liquid phase with SR Feed**

A KRAKKOLT ALAPANYAG PROBLÉMÁJA

A krakkolt alapanyag (DCU, FCC)
(di)olefineket és aromásokat tartalmaz

Ezek a komponensek frissen szulfidált katalizátorral érintkezve **polimerizálódnak**



A polimer gyanta lerakódás **blokkolja** a katalizátor pórusait és aktív helyeit

Ez végleges katalizátor aktivitás **veszteséghez** vezet

❑ **Katlizátor gyártók általában minimum 3 nap „betörési" időszakot javasolnak „straight run" alapanyaggal.** ➡ **Solution: TOTSUCAT CFP**

TOTSUCAT® CFP

CFP = Cracked Feed Protection

- TOTSUCAT®-CFP mimics the 3 days break-in period
- Catalytic acidity is reduced no gums formation
- Can inject cracked feed immediately
- Proven in refineries: 60+ references

Example case of 75 tons reactor treating 30% cracked feed

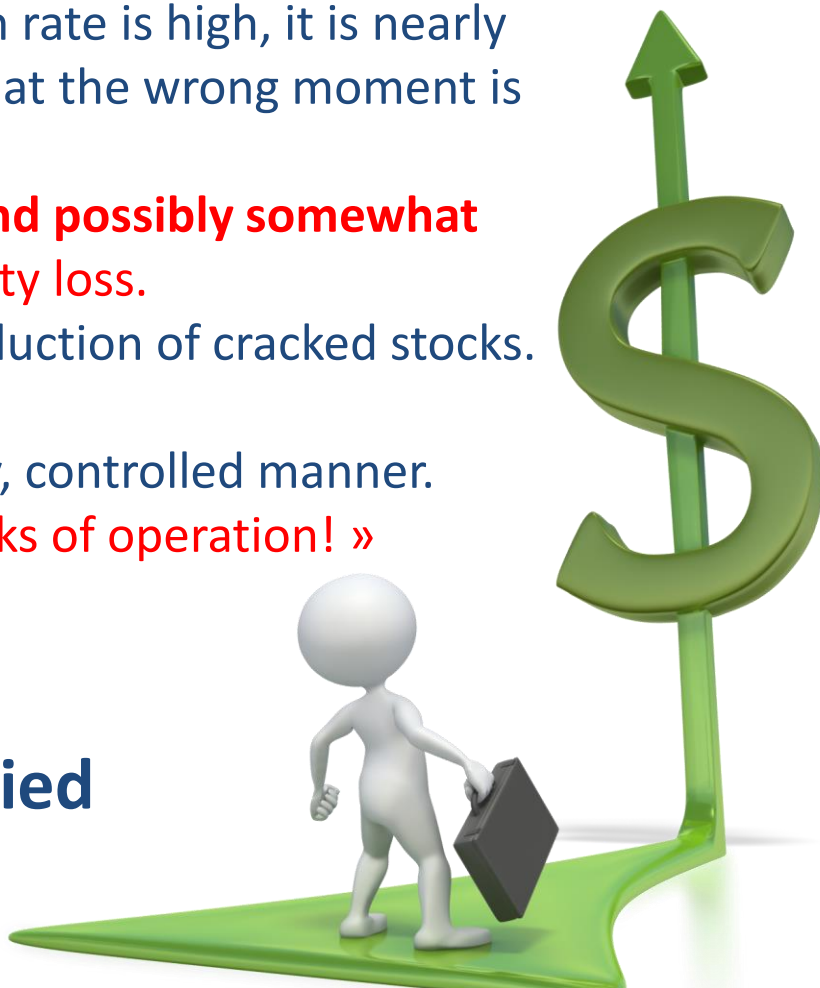
- lost opportunity in order of ~500 k$
- plus associated logisitics cost.

CASE STUDY

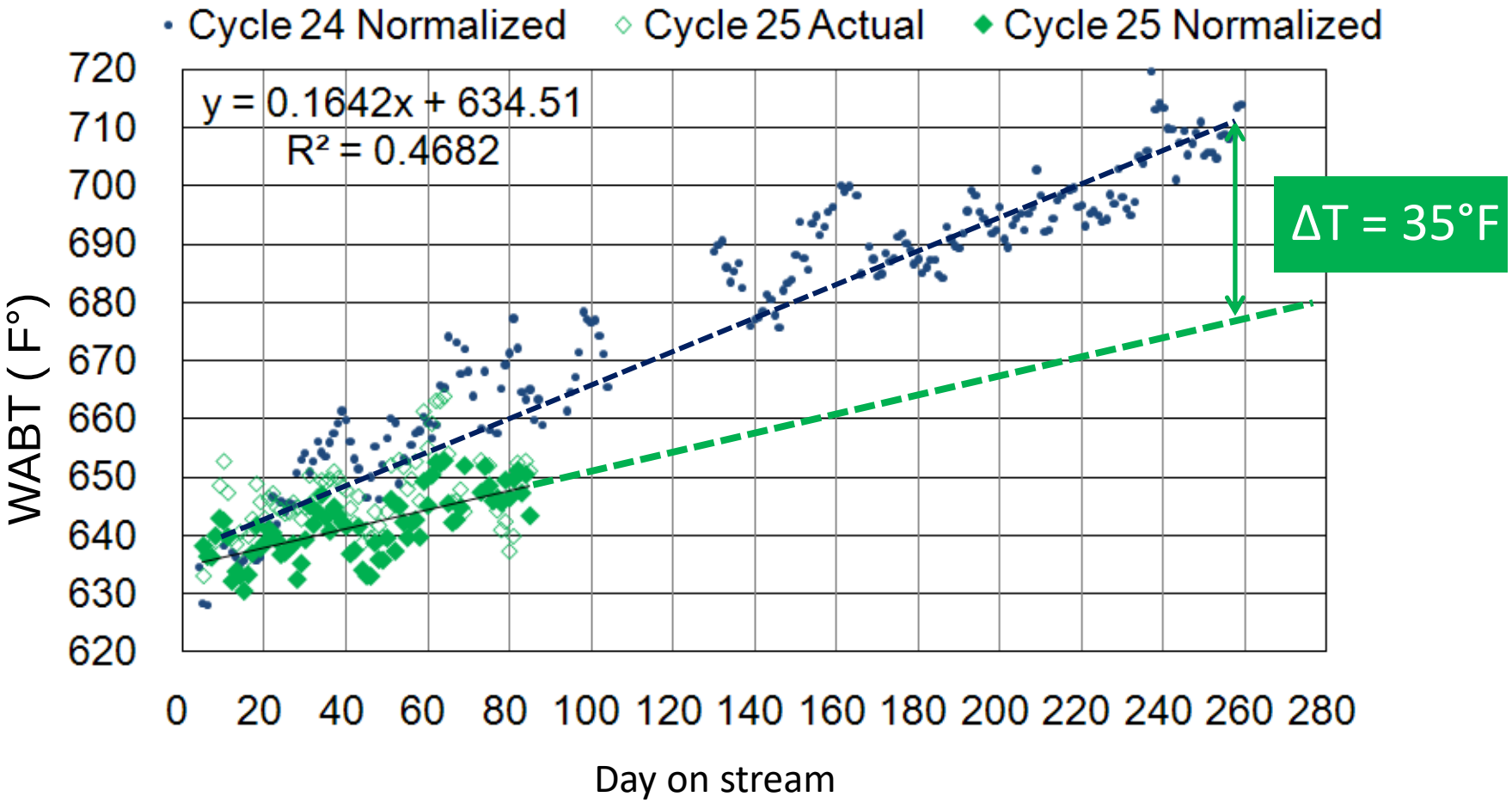
Recommendations from Catalyst Supplier:

- « In a stressed unit, once the deactivation rate is high, it is nearly impossible to reduce it. 2-3 % LCO added at the wrong moment is sufficient to double the deactivation rate.
- Therefore, it is **critical to have a gentle and possibly somewhat lengthy startup** to avoid premature activity loss.
- Run 3 days on virgin oil prior to the introduction of cracked stocks. Longer is better!
- Introduce cracked stocks in a slow, steady, controlled manner.
- **Do not push the unit** in the first two weeks of operation! »

Instead, the refinery applied Totsucat CFP

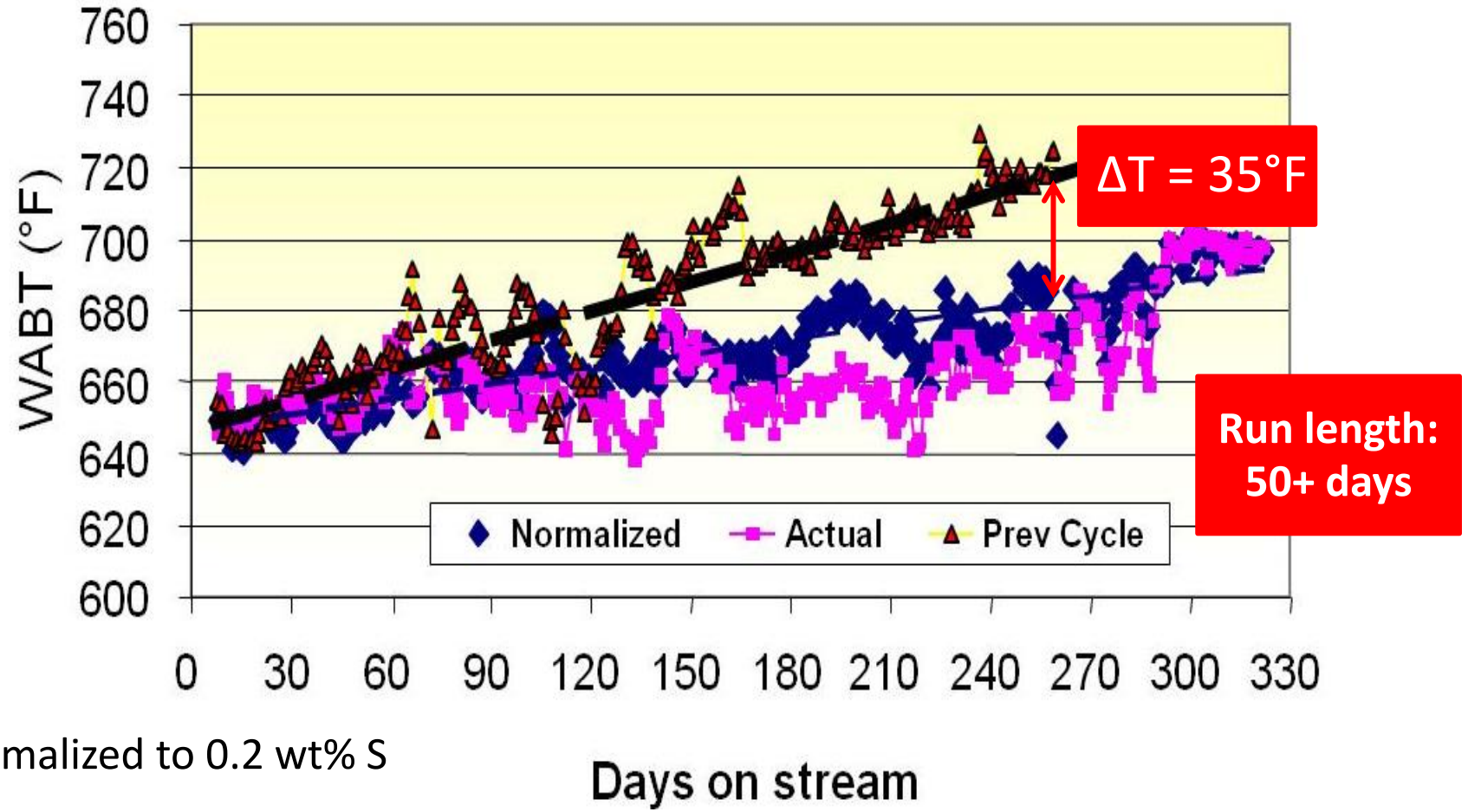


CASE STUDY - TOTSUCAT® CFP IN ULSD
SERVICE AFTER 90 DAYS



Normalized to 0.2 wt% S

CASE STUDY - TOTSUCAT® CFP IN ULSD
SERVICE AFTER 320 DAYS



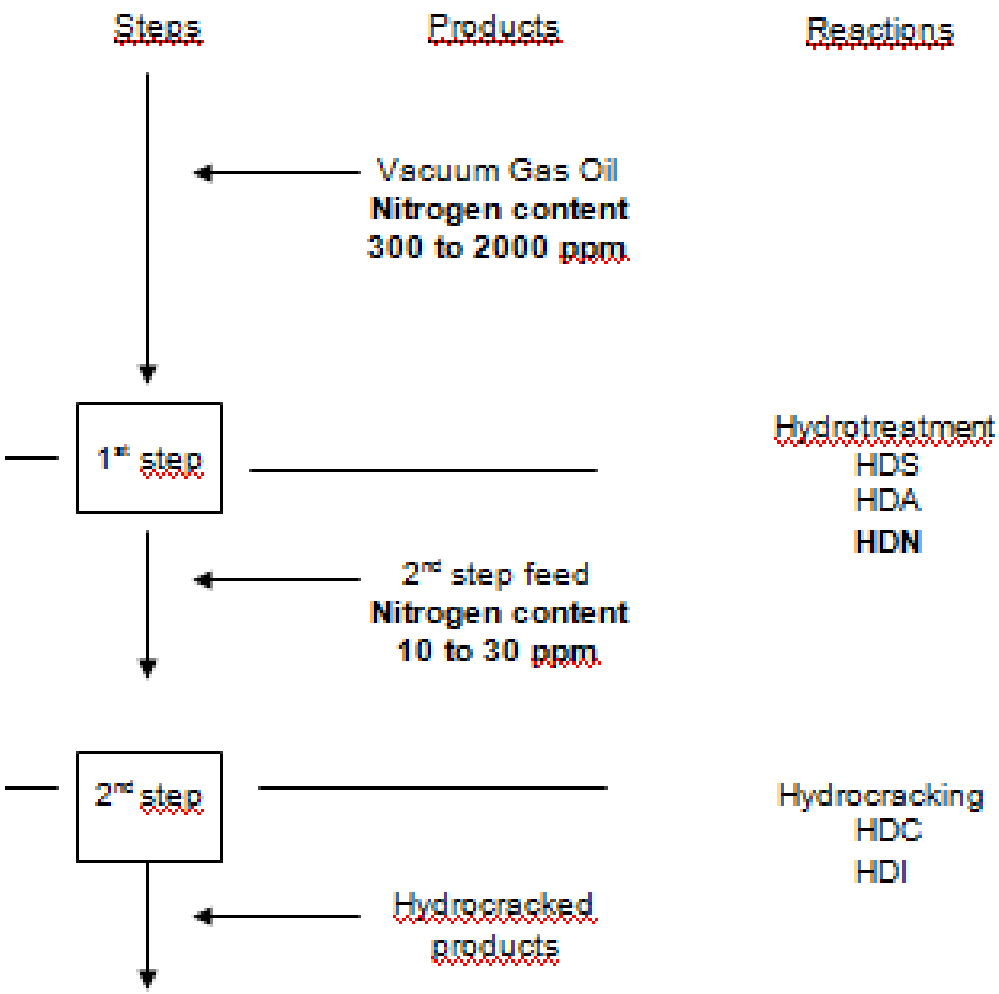
Normalized to 0.2 wt% S

- **Deactivation significantly lower with TOTSUCAT-CFP than with in situ!**

TOTSUCAT® BRANDS

TOTSUCAT – G	• Naphtha, Kero, FCC gasoline, Tail Gas, Lubes
TOTSUCAT – D	• (Ultra Low Sulfur) Diesel
TOTSUCAT – N	• Hydrocracking Pretreat
TOTSUCAT – E	• FCC Pretreat
TOTSUCAT – HC AP Acidity protection	• Hydrocracking
TOTSUCAT – CFP	• Light Cycle Oil, Heavy Cycle Oil • Coker and Visbreaker Naphtha / Diesel

HYDROCRACKING PROCESS: 2 STEPS OF REACTIONS

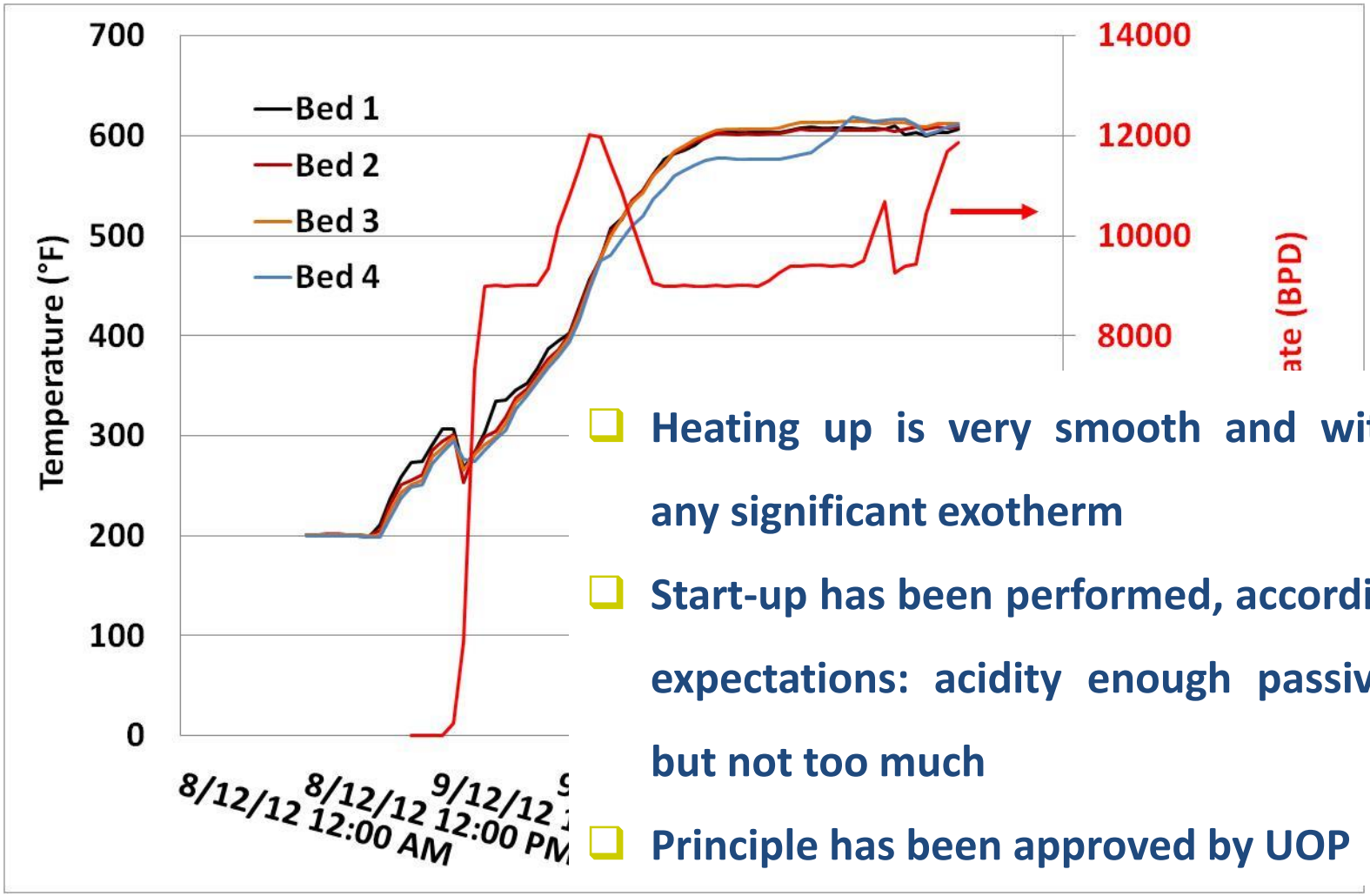


HDC: ISSUE OF START-UP

- **Fresh vs steady state catalysts**
  - Zeolite catalyst contains very strong acidic sites: can crack hydrocarbons already around 250°C
  - In steady state, the strongest acidic sites are essentially covered with N containing compounds (mainly ammonia) cracking temperatures are between 300 and 350°C.

- **How to go from a fresh to a steady state catalysts?**
  1. Wait for a long time that N in the feed, transformed by HDN to NH_3, slowly saturates the acidic sites
  2. Inject during sulfiding procedure NH_3 or another N containing compound
  3. **Use the TOTSUCAT HC-AP**

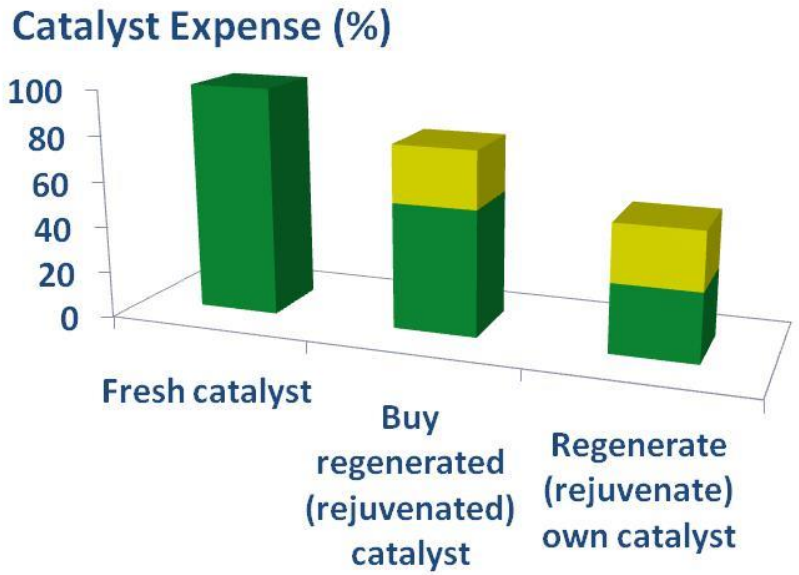
HYDROCRACKER START-UP WITH TOTSUCAT HC-AP



- Heating up is very smooth and without any significant exotherm
- Start-up has been performed, according to expectations: acidity enough passivated, but not too much
- Principle has been approved by UOP

Konklúzió

- A katalizátor regenerálása és ismételt felhasználása jelentős költségcsökkentést eredményez:



- TOTSUCAT:
  - Optimise catalyst performance
  - Reduce unit downtime

REGULAR CUSTOMERS

Catalyst manufacturers

ALBEMARLE®
Axens IFP Group Technologies
UOP A Honeywell Company
GRI
HALDOR TOPSØE CATALYSING YOUR BUSINESS
GRACE
BASF The Chemical Company
JM Johnson Matthey
CLARIANT
ExxonMobil Chemical

Catalyst end-users: oil refiners & (petro)chemicals companies

ExxonMobil
Shell
TOTAL
Chevron
CALTEX
bp
Eni
REPSOL
CEPSA
Statoil
preem
OMV
KNPC
VARO ENERGY
ارامكو السعودية Saudi Aramco
ADNOC
BR PETROBRAS
PEMEX
INEOS THE WORD FOR CHEMICALS
VALERO
PETRON
Reliance
petroineos REFINING AND TRADING
ESSAR
MOL Group
LUKOIL
سابك sabic
DOW
lyondellbasell
ARKEMA
STYROLUTION
SCG
ptt GLOBAL CHEMICAL
PCS
EASTMAN
BOREALIS
Rhodia
SOLVAY
HUNTSMAN
sasol
AIR LIQUIDE
THE LINDE GROUP Linde

REGULAR CUSTOMERS

Catalyst manufacturers

ALBEMARLE® Axens UOP GRACE

THANK YOU FOR YOU ATTENTION!

ies

ExxonMobil KNPC VARO ENERGY preem OMV MOL Group LUKOIL

Saudi Aramco PETROBRAS INEOS PETRON ESSAR

sabic DOW lyondellbasell ARKEMA STYROLUTION SCG ptt GLOBAL CHEMICAL PCS

EASTMAN BOREALIS Rhodia SOLVAY HUNTSMAN sasol AIR LIQUIDE Linde