

Pilot Plants

Bench Scale Cyclic Deactivation (CD) Unit

360[®]
KAS

The method to obtain a resembling equilibrium catalyst (E-cat) at laboratory scale by simulating the cyclic nature of commercial fluid catalytic cracking (FCC).

During its lifetime a Fluid Catalytic Cracking (FCC) catalyst is subjected to numerous repeated cycles of cracking, stripping and regeneration. It thereby endures a variety of physical and chemical changes that cause deactivation.

Deactivation is firstly caused by hydrothermal deactivation due to the presence of steam at elevated pressures. This results in zeolite de-alumination, zeolite decomposition and matrix collapse. Secondly, the deposition of metals entrained in the oil feed have deleterious selectivity effects:

- Vanadium (Va) blocks pores, destroys acid sites and has some dehydrogenation activity leading to poor coke and gas selectivity.
- Nickel (Ni) blocks pores, is macroscopically immobile and has dehydrogenation activity.
- Sodium (Na) blocks pores and destroys acid sites.
- Iron (Fe) blocks pores and causes a large drop in catalyst accessibility.

When evaluating FCC catalysts at laboratory scale, it is of imperative importance that fresh catalyst is properly deactivated to a so called equilibrium catalyst (E-cat) sample. E-cat is a mixture of fractions with a distribution of age, metals and activity. Some particles are loaded with metals or carbon and have lost most of their surface area. Other particles are relatively fresh with extremely high surface area and unit cell size.

To obtain a mimic E-cat sample at laboratory scale both cyclic hydrothermal deactivation and cyclic metal deposition must be simulated in a well-controlled manner closely resembling deactivation in a commercial FCC unit.

The activity, metal content, metal oxidation state and textural properties like surface area and micro-pore volume of the deactivated catalyst must be created as close as possible to the commercial E-cat.

This technology was pioneered by Albemarle® and is brought to market by 360°KAS since 2003 in the form of our bench-scale Cyclic Deactivation (CD) Unit.

Based upon the amount of E-cat you require for performance testing, our CD unit can be supplied with either one (CD), two (MCD-2) or four (MCD-4) reactors



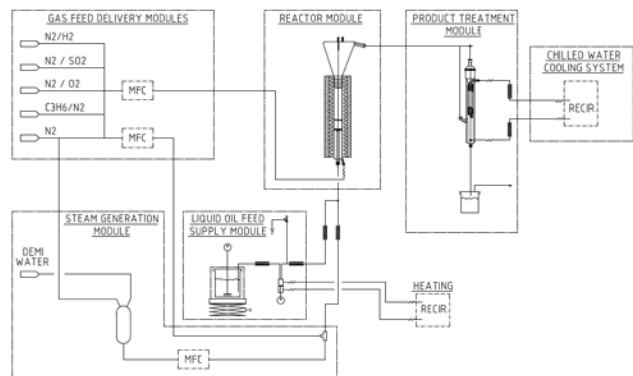
MCD-2 unit before shipment to Abu Dhabi Oil Refining Company (Takreer) R&D center in Abu Dhabi, UAE.

The 360°KAS Cyclic Deactivation unit has proven to be the only method to reproduce all the fundamental phenomena that simulate the aging process in a commercial FCC operation.

Functionality

Next to the common utilities, each CD reactor is supplied with the following process modules:

- Gas feed delivery modules for N_2/H_2 , N_2/O_2 , N_2/C_3H_6 , N_2/SO_2 and N_2 .
- A steam generation module with a de-mineralised water feed.
- Heated liquid oil feed supply module with injection system.
- Reactor module.
- Product treatment module (condenser).
- Closed chilled water cooling system.



Process modules per CD reactor.

The system is built into a ventilated enclosure with polycarbonate sheets for use in safe area. Provision for forced ventilation is foreseen at the top of the unit. This is required to provide a safe environment when using hazardous and flammable gases and liquids.

360°KAS supplies the CD unit in accordance with the following technical specifications:

Cyclic Deactivation Unit	
Type of Reactor	Quartz Glass Fluidized Bed
Catalyst Intake per reactor	Min 75 g Max 200 g
Fluidizing Medium	Nitrogen / Reactant gas
Feed of Oil	(Ni or Va spiked) VGO
Steam Make-Up	Max 1 kg/hr
Type of control	Sequence control and temperature control PLC/PC
Gas flow control	Instrument-based
HMI & Data Acquisition	In Touch - Wonderware
Approximate Dimensions (WxDxH in mm)	CD: 2160 x 860 x 2080
	2-MCD: 2700 x 1400 x 2080
	4-MCD: 4200 x 1250 x 2080

Utilities (required)	
Power	220-230/380-400 VAC, 50/60 Hz, 3-phase + neutral (other are possible upon request)
Instrument Air	5 - 7 barg, HC free, dew point < -30 °C, max consumption 4-8 L/h
Nitrogen	bottle or clean supply network, 20-200 barg, min 7 barg, 99.99% purity, max consumption: 60 nL/hr at peak/per reactor
Demineralized water	3 Barg, consumption max 1L per 24/hr per reactor section, this can also be added manually
N ₂ /H ₂ (60/40)	20-200 Barg, peak 60 nl/hr per unit per reactor section
N ₂ /O ₂ (60/40)	20-200 Barg, peak 60 nl/hr per unit per reactor section
N ₂ /C ₃ H ₆ (95/5)	20-200 Barg, peak 60 nl/hr per unit per reactor section
N ₂ /SO ₂ (B/3000 ppm)	20-200 Barg, peak 60 nl/hr per unit per reactor section



MCD-4 unit including gas mixing station on the left before shipment to Indian Oil Company Ltd. (IOCL) R&D center in Faridabad, India.

The reactor is constructed of inert Quartz glass instead of steel for the following main reasons:

- Exclusion of Nickel influence and other metal parts especially at high temperatures (700-900 °C).
- Fluidisation process inside the reactor can be monitored and afterwards proper emptying of the reactor can be visually checked.

The prepared E-cat can be used for consequent performance testing in our Short Contact Time Riser Test (SCT-RT) Unit. This Albemarle® developed unit simulates the behaviour of a commercial FCC riser with a short oil injection time of about one second. For more information, see our dedicated 360°KAS Short Contact Time Riser Test (SCT-RT) Unit product sheet.

Control system

A CD unit is delivered as fully computer controlled system with all necessary monitoring and control functions, safety and alarm management and data acquisition with historical trending.

In the control system the following components can be distinguished:

- One independent control cabinet with PLC per reactor.
- One common control cabinet with PLC and power distribution box with main switch.
- One common SCADA PC to act as HMI and screen to display operational data and settings.

The CD unit is standard supplied with 13 “ready-to-use” preconfigured deactivation protocols built up with cycles of:

1. Metallisation

Cracking (feed spiked with Nickel and/or Vanadium) and Regeneration cycles, including steaming

2. Aging / Redox

Oxidation and Reduction cycles, including steaming

3. Pre- or Post-Treatment

Steaming, stripping, heat treating, oxidation or reduction step

Depending on the required target deactivation status, the standard protocols are built up with selected steps of metallisation, oxidation or hydrothermal deactivation.

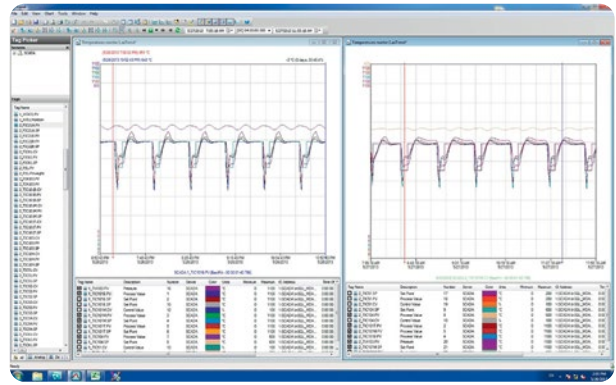
Within the standard methods/protocols, the client operator has a large degree of freedom to adapt the recipes. For example temperatures, gas flows, gas sources, number of cycles, running times and/or repeat cycles can be adjusted. The reactors of the MCD-2 and MCD-4 unit can be programmed to operate fully independent from the others following a different deactivation protocol. If required, client specific protocols can be added.

Two types of data collection are provided:

- Continuous I/O process data logging for graphic trends on the SCADA PC.
- Experiment snapshot I/O process data logging for data reporting and detailed analysis. This data will be available in CSV-file.

No.	Protocol Name	Description of Protocol
1.	Two-Step CD	Metallization followed by Aging
2.	Special Two-Step CD	Metallization followed by Aging
3.	SM-3 Mild-CD	Metallization (low metal content)
4.	SM-3 V-CD	Metallization (low Nickel, high Vanadium content)
5.	Ni-CD, known as 54-cycles	Post-treatment with H ₂
6.	Special Ni-CD	Metallization (high Nickel, low Vanadium) Post-treatment with H ₂
7.	AN-CD 1 Method	Metallization Post-treatment Propylene
8.	AN-CD 4 Method	Metallization Post-treatment Propylene
9.	CD-ALFA (not available on PSMCD)	Metallization with a high amount of cycles (300)
10.	CPS Method	Oxidation / Reduction
11.	CPS-5-2-2012-i-mCD1 Method	Oxidation / Reduction Post-treatment Propylene
12.	CPS3 Method	Oxidation / Reduction Post-treatment Propylene
13.	Post-treatment Propylene	Steaming only

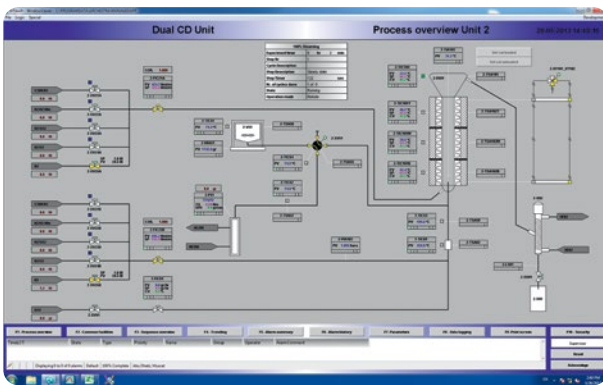
The standard “ready to use” deactivation protocols supplied with the 360°KAS Cyclic Deactivation unit.



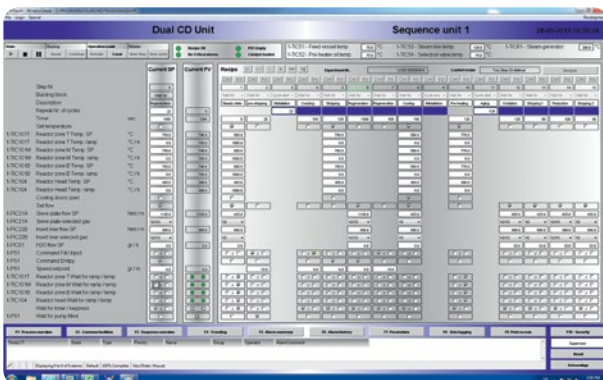
In the trend screen all historical I/O data can be plotted up to 6 months in the past.

Looking for a way to produce more than 800 gr of E-cat per experiment? 360°KAS also offers the Cyclic Deactivation technology in the form of a Pilot Scale Multi Cyclic Deactivation (PSMCD) unit that produces 2 – 4 kg of E-cat per reactor.

Interested? Contact us for more information via info@360KAS.com



The process overview screen shows the controls (flows, valve openings, pressures, temperatures) per CD reactor unit.



The sequence screen shows the method/protocol parameters per CD reactor unit.

Options

The following options can be quoted upon request:

- Interface with your local LIMS and prepare/write custom made reports.
- Gas mixing station for feed gases in case these cannot be supplied with a consistent compositional quality.
- Uninterrupted Power Supply (UPS).
- Semi-automatic catalyst operation with manual addition and full automatic withdrawal after cool down of the reactor.
- Laboratory equipment such as glass work, calciner, catalyst sieves and weighing scales.
- Analytical equipment such as an FTIR analyser for emission monitoring of exhaust gases.
- Consumables such as metal naphthalenes to spike VGO.
- Services such as site installation, installation supervision, commissioning, start-up and training.
- Annual maintenance.
- Spare parts for 2 year operation.
- Extended warranty.

The following Pilot Plant product sheets are available:

- Cyclic Deactivation (CD) Unit
- Short Contact Time – Riser Test (SCT-RT) Unit
- Hot Attrition Test (HAT) Unit



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