

REINHOLD ENVIRONMENTAL Ltd.



2012 NO_x-Combustion Round Table & Expo Presentation

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REINHOLD ENVIRONMENTAL
2012 NO_x-Combustion/PCUG Conference

Laboratory Testing in Support of SCR Catalyst Management

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What Tests are Performed ?

- 1. DeNOx Activity**
- 2. SO₂ Conversion**
- 3. Geometry**
- 4. Pressure Drop**
- 5. Chemical Composition**
- 6. Special Analyses**

BENCH-SCALE REACTOR TESTS

- **DeNO_x ACTIVITY (K)**
- **SO₂ CONVERSION**

ACTIVITY EQUATIONS

$$\text{Activity: } K = - AV \ln(1-\Delta\text{NO}_x)$$

Where ΔNO_x is the NO_x reduction measured in the apparatus and AV is the area velocity at which the test is conducted (expressed at standard conditions). Activity is usually expressed in units of m/hr.

$$\text{Relative Activity: } (K/K_0) = K_\tau/K_0$$

Where K_τ is the activity at exposure time (τ) and K_0 is the activity at exposure time zero. Note that relative activity may be expressed as either a fraction or a percentage, depending on preferred convention.

SO₂ CONVERSION EQUATION

$$\text{SO}_2 \text{ Conv.}(\%) = \frac{(\text{SO}_3\text{out} - \text{SO}_3\text{in})}{\text{SO}_2\text{in}} * 100\%$$

Where SO₃out and SO₃in are the gas-phase concentrations of SO₃ at the catalyst outlet and inlet, respectively, and SO₂in is the gas-phase concentration of SO₂ at the inlet.

Activity and SO₂ Conversion Bench-Scale Laboratory Reactor

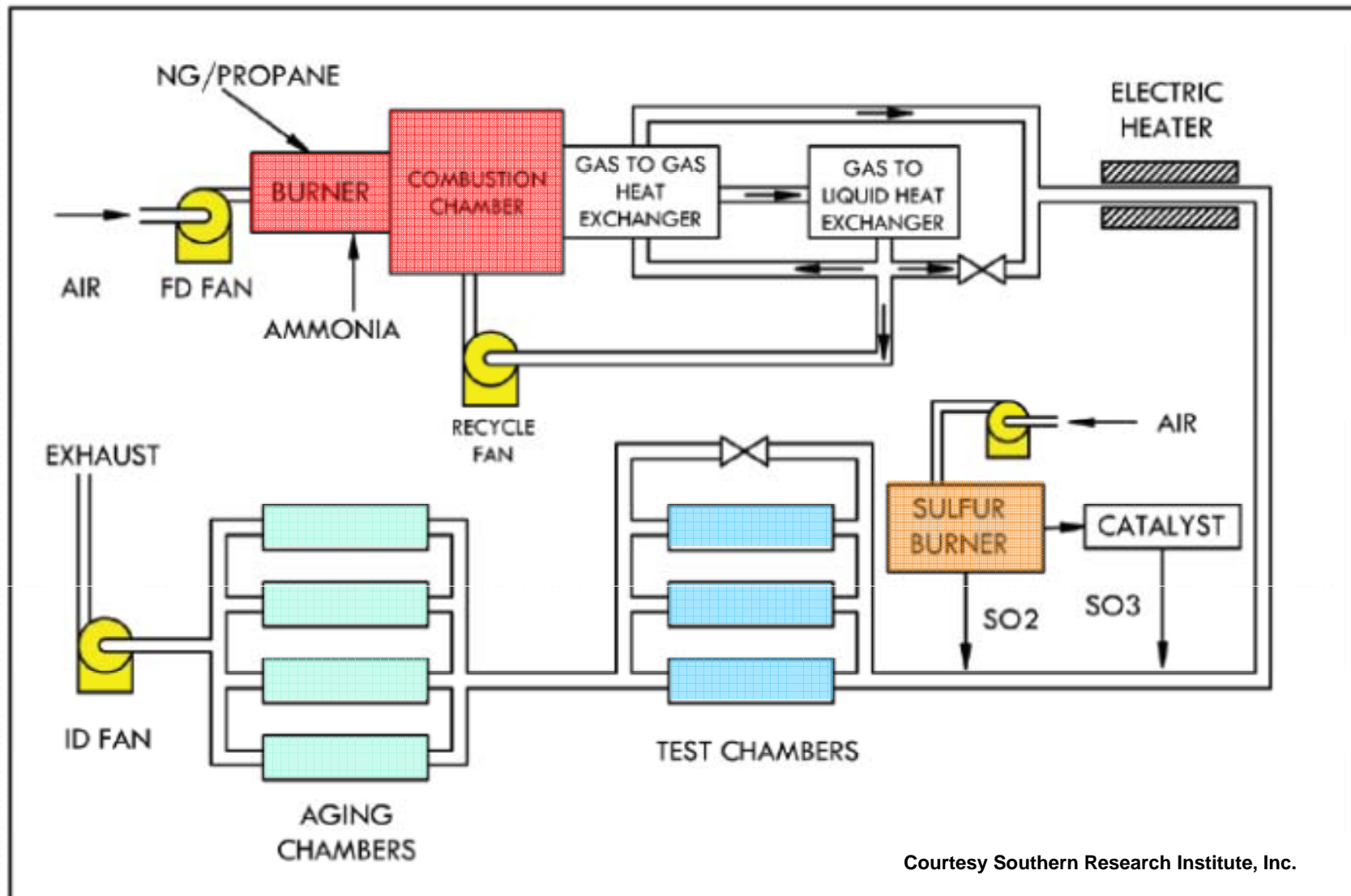


Photo of Lab Test Chambers



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CATALYST SAMPLING

- More is better
- For split reactors, 1 sample each side
- For single reactors, 2 samples adds a great deal of confidence
- Sampling every 4,000 hrs. (6 mo.) gives most timely info. and allows problems to be identified before they become critical
- Honeycomb – select samples that are most representative of entire layer, keep good records of where sampled to avoid repeat sampling
- Plate – sample from several modules to create a composite sample representative of the entire layer – usually about 16 plates needed, always removed in pairs
- Corrugated – usually a “block” is removed which is then cut to size – sampling considerations similar to honeycomb



Sample Preparation



Honeycomb: Full-length element sampled and tested 150mm x 150 mm CSA.

Plate: Test element constructed of approximate CSA of honeycomb element. “Half” layer often tested, 24-26 “mini” plates, ~16 full plates needed for element construction.

Corrugated: One block or test element sampled, cut to size to form element similar to honeycomb element – as with plate, half layer often tested.

2 PRIMARY USES OF LAB DATA (for Activity and SO₂ Conversion)

Absolute Measurements: Used in catalyst design, manufacturing, quality control, etc. Sometimes specifications/guarantees. If correction factors are not used – this should match exactly the condition of the field.

Relative Measurements: Catalyst tracking and management, deactivation trending, etc. Less stringent requirement to match field conditions, since it's a **relative** measurement, i.e. K/K_0 , but must match exactly the conditions of the K_0 testing to be valid.

SPECIFIED CONDITIONS

Parameter	Target for Absolute Activity	Target for Relative Activity
Temperature	field	field $\pm 10^\circ$ F
Linear Vel.	field	field
O ₂	field	field $\pm 1\%$, as long as at least 3%
H ₂ O	field	as generated
CO ₂	as generated	as generated
NO _x	field	field $\pm 10\%$
SO ₂	field	field $\pm 20\%$
SO ₃	field	0 or field
NH ₃ /NO _x ratio	1.0	1.0
N ₂	Balance	Balance

SUMMARY

SELECTION OF LAB TEST CONDITIONS

Absolute Measurements: MUST MATCH EXACTLY CONDITIONS OF APPLICATION IF CORRECTIONS ARE NOT USED.

Remember: If RFP requires guaranteed activity, exact conditions must be specified – absolute K-values are meaningless if conditions are not known/specified.

Relative Measurements: CAN DEVIATE SOME FROM APPLICATION CONDITIONS, BUT CONDITIONS FOR K_t MUST MATCH EXACTLY THOSE FOR K_0 .

Remember: You need K_0 for relative activity, best to collect sample when fresh catalyst arrives. Depends partly on what lab will be testing – independent vs. catalyst supplier.

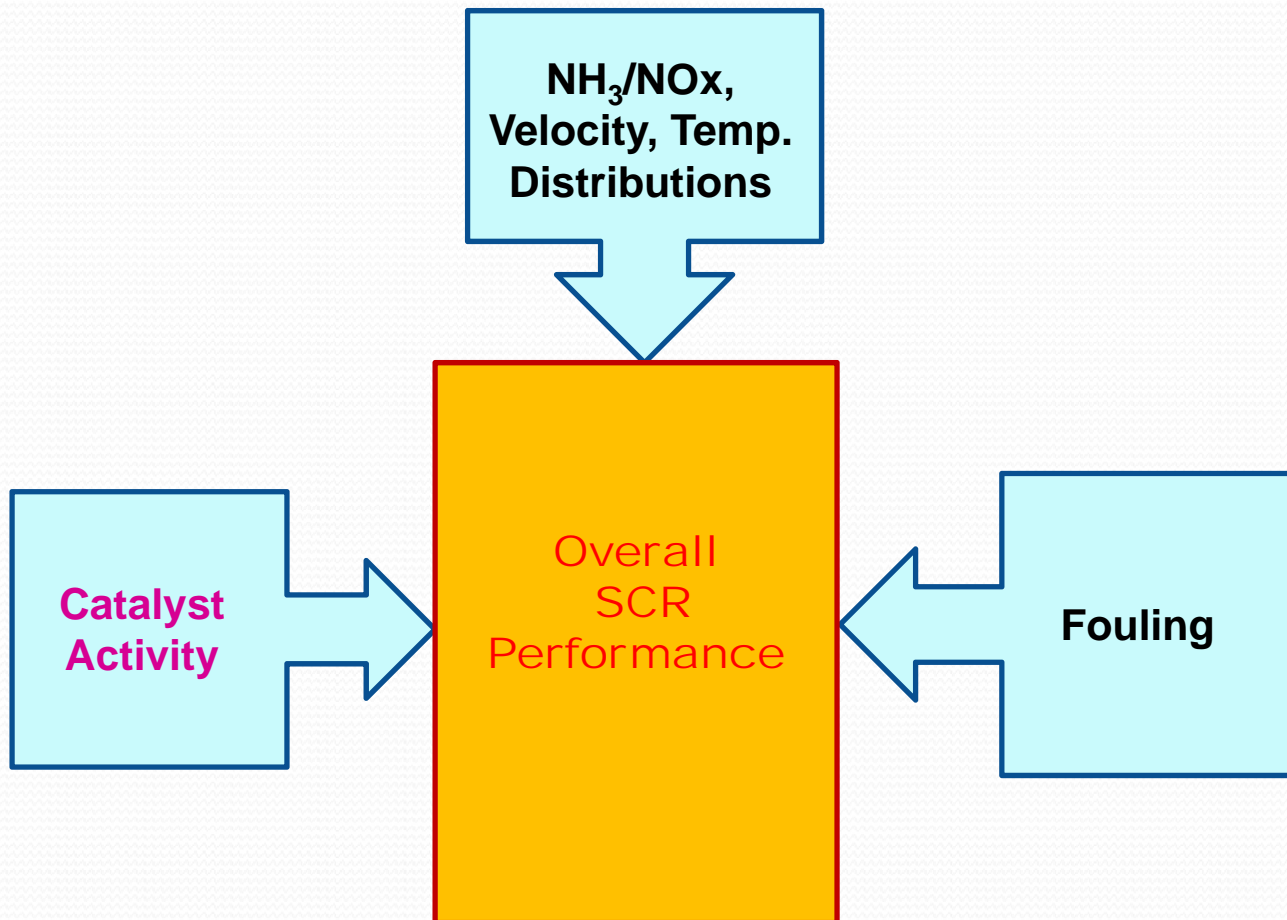
Catalyst Management and Deactivation Curves

- Management activities generally focused on maintaining some required minimum level of reactor potential (P). Remember that the required RP is a function of the assumed operating conditions.

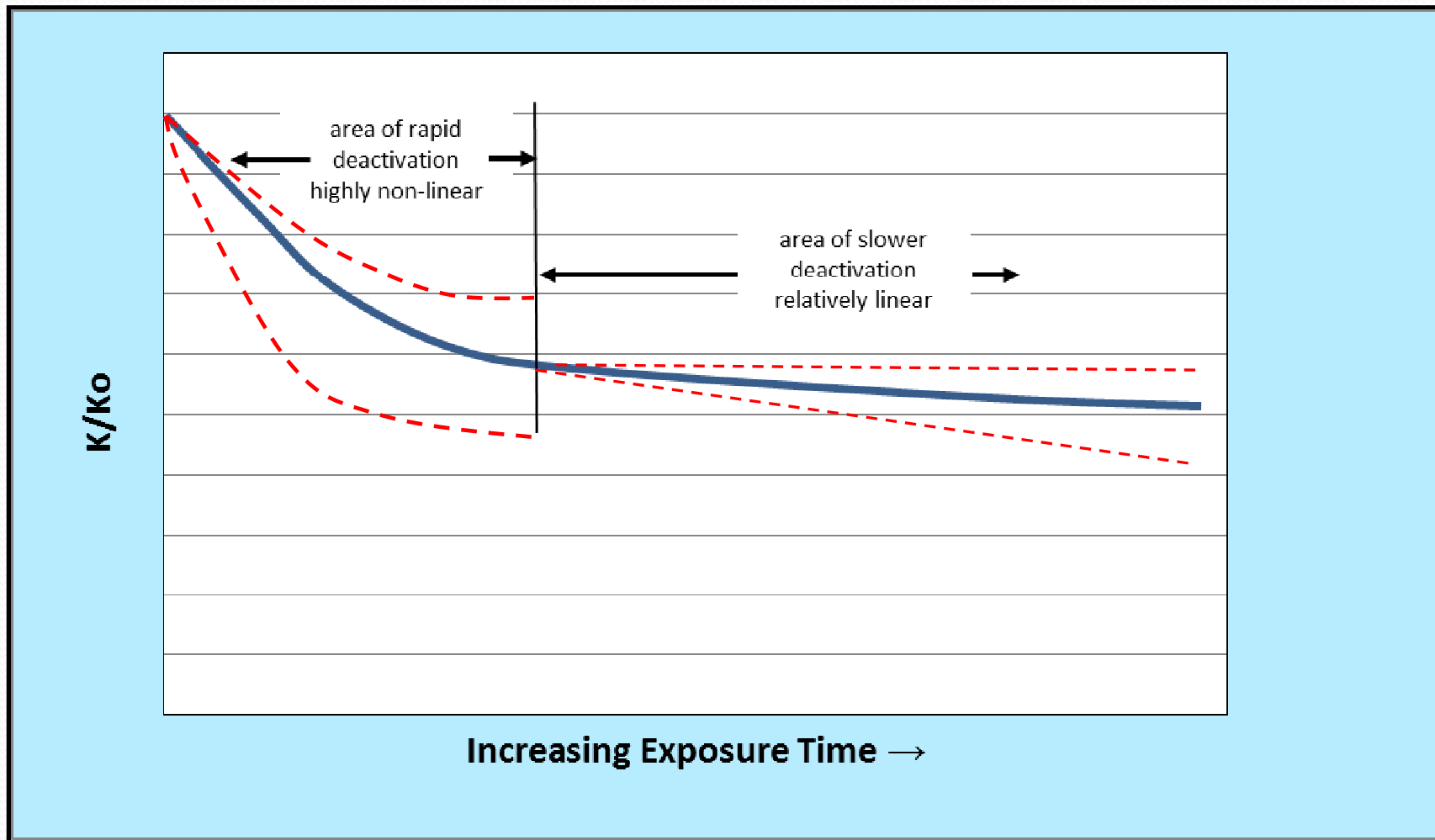
$$P = K/AV$$

- Reactor Potential is basically the activity times total surface area, per unit of flue gas flow rate. Usually calculated on a layer-by-layer basis which sum to give total reactor potential.

CATALYST ACTIVITY IS ONLY ONE PIECE OF THE PUZZLE !

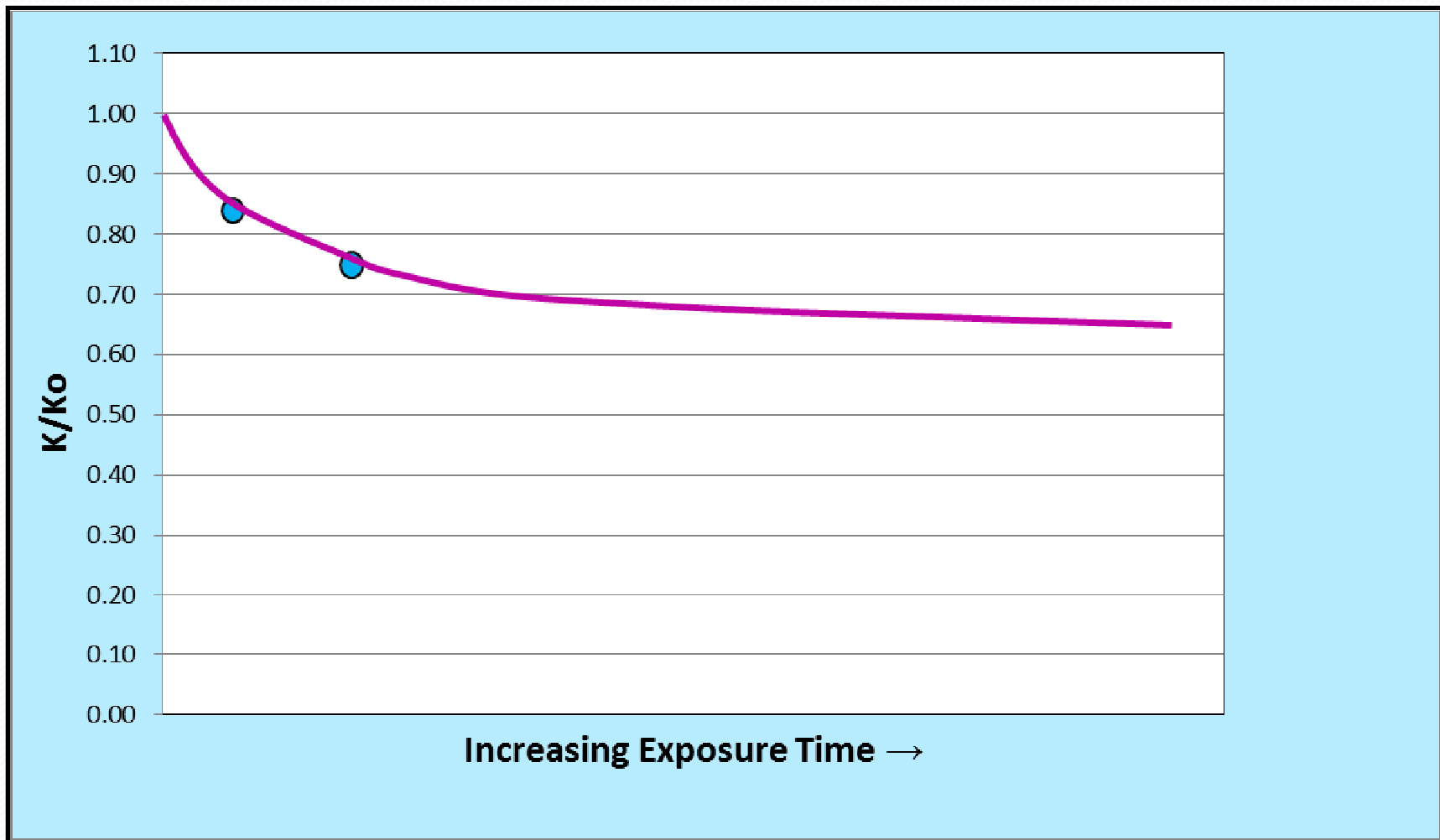


Typical Shape of Deactivation Curve



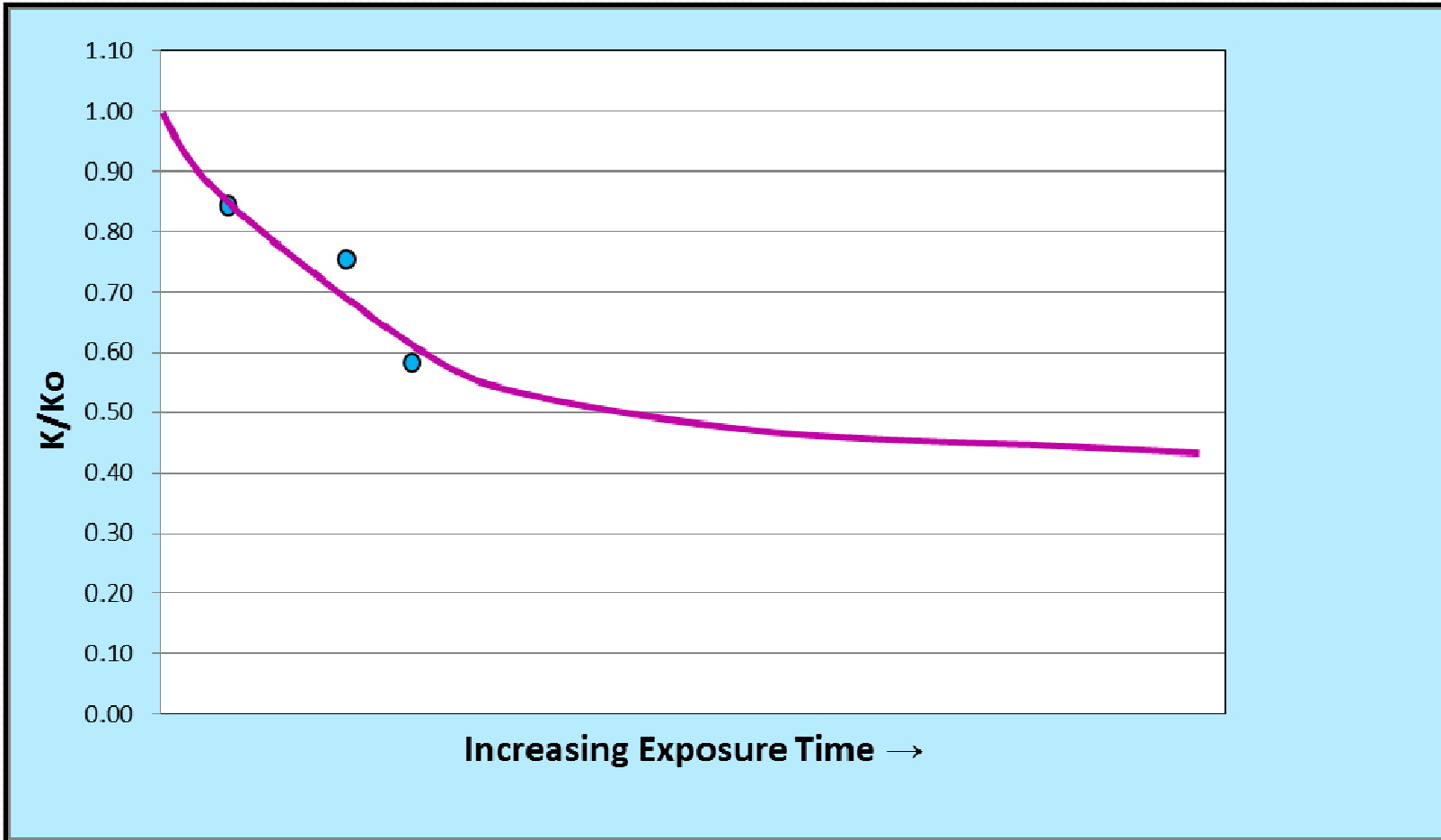
Prediction From Short-Term Data

$K/K_0 = 0.65$ at end of life ?



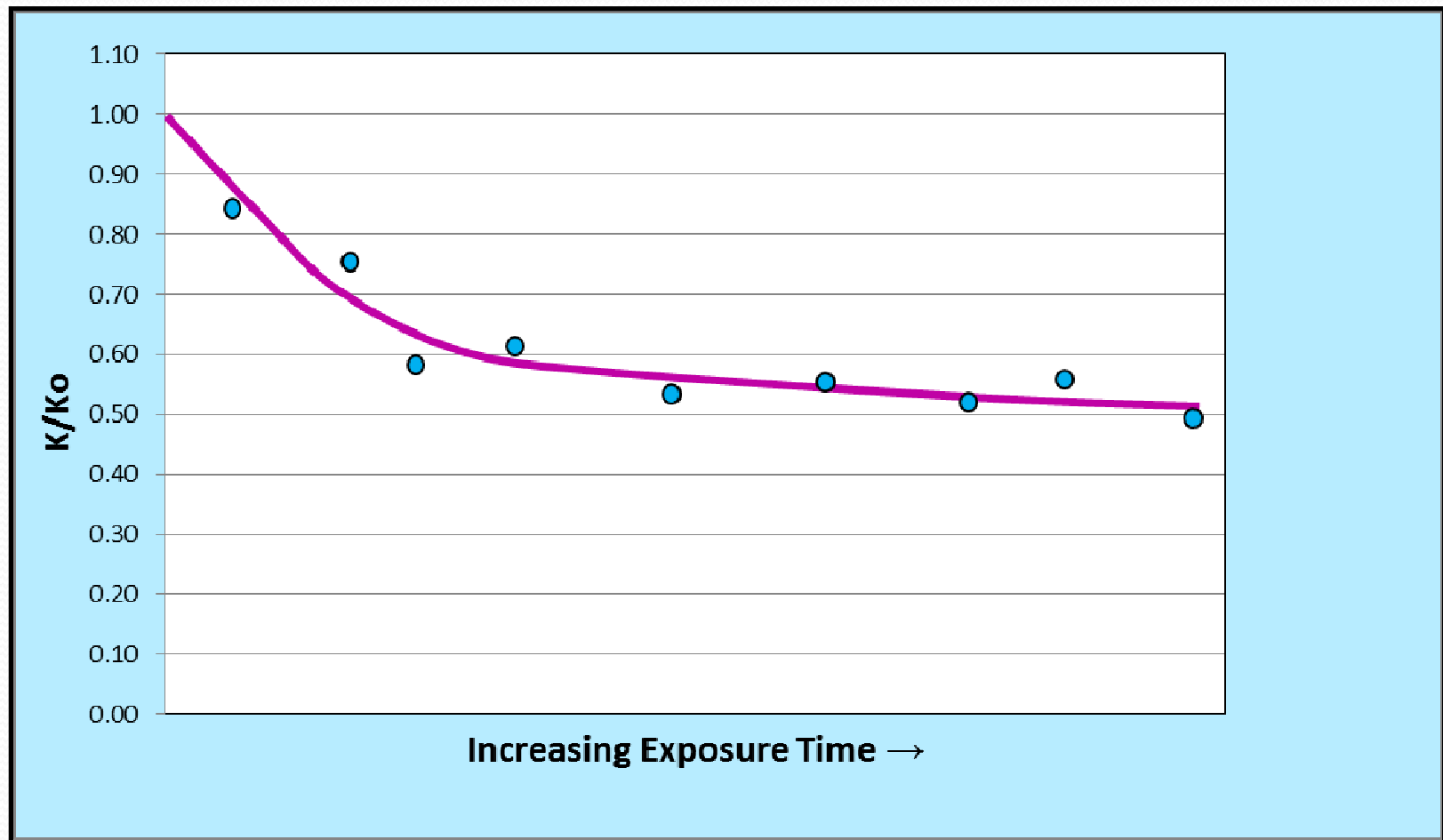
Prediction from Longer-Term Data

$K/K_0 = 0.40$ at end of life ?



Actual Shape of Example Deactivation Curve

$K/K_0 = 0.50$ at end of life

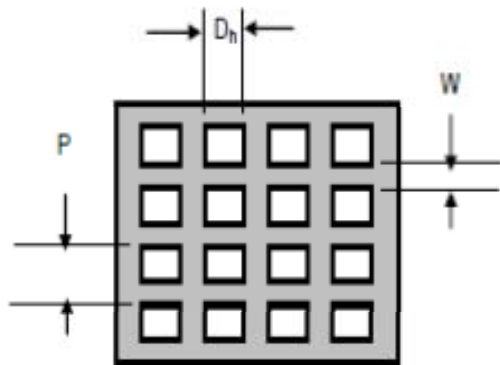


PHYSICAL AND CHEMICAL MEASUREMENTS

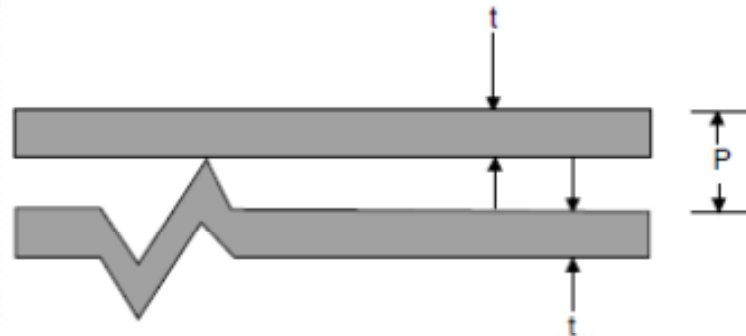
- **GEOMETRY**
- **PRESSURE DROP**
- **CHEMICAL COMPOSITION**
- **SPECIAL TESTS**

Geometry

Honeycomb



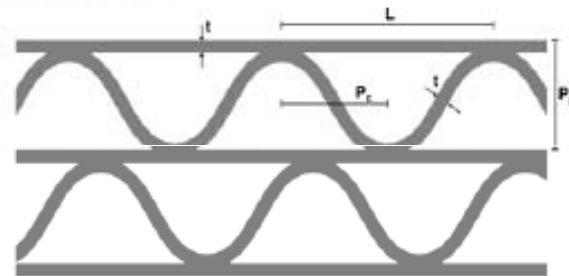
P = pitch, mm
 W = wall, mm
 D_h = hydraulic diameter, mm



Plate

t = plate thickness, mm
 P = pitch, mm

Corrugated



P_p = is the plate pitch, mm
 P_c = is the corrugated pitch, mm
 t = is the wall thickness, mm
 L = is the wave length, mm



Geometry Uses

- **Important in determining the specific surface area (m^2/m^3) of the catalyst, which in turn is needed for area velocity calculations ($AV = \text{Flow}/\text{surface area}$)**
- **Verifies catalyst type and pitch**
- **Helps to determine attrition rate (wall thinning)**
- **Determines clear cross-sectional-area/fouling**

Typical Measured Geometry Parameters

Honeycomb and Corrugated

Length	mm
Width / height	mm
Pitch	mm
Clear width catalyst channel	mm
Wall thickness	mm
Outside wall thickness	mm
Original No. of channels	-
No of channels	-
No of Plugged channels	-
Clear cross sectional area	%
Clear catalyst surface	m ²
Specific Catalyst Surface	m ² /m ³

Plate

Number of plates	
Length:	mm
Width Double Bead:	mm
Single Bead:	mm
Catalyst Cross Sectional Area:	mm ²
Thickness	mm
Catalyst Surface Area:	m ²
Specific Catalyst Surface Area:	m ² /m ³
Clear cross sectional area	%

Chemical Composition

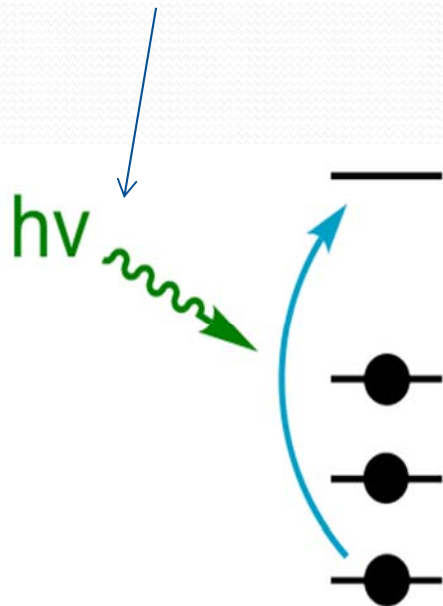
Why Test ?

- **Chemical composition helps to confirm poisoning mechanism (arsenic for eastern bituminous coals, and calcium/sulfur for PRBs).**
- **May be used to raise flag related to excessive poison uptake.**
- **Especially important for regeneration firms to confirm effectiveness of poison removal and active compound impregnation.**
- **Important troubleshooting tool.**

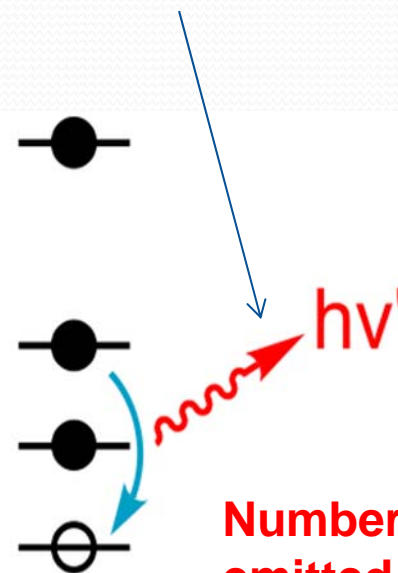
Chemical Measurements

X-Ray Fluorescence (XRF)

High-Energy X-ray ejects electron from atomic shell



Outer electron "falls" into vacant position emitting characteristic secondary (fluorescent) x-ray



Number and strength of the emitted X-rays determine concentration and element



X-Ray Fluorescence Options

Bulk – sample prepared by grinding the catalyst to produce an average/bulk concentration – AA/ICP and other methods can also be used for determinations.

Surface – only surface of the catalyst normally exposed to flue gas is analyzed – typically produces higher poison levels and may be more indicative of what the reactants “see”.

Inlet/Outlet – different levels of poisons may be present at the inlet compared to the outlet due to turbulent region at entrance and depletion of poisons in lower regions

SPECIAL TESTS

BET Surface Area – measures the actual microscopic surface area of the catalyst – not to be confused with “geometric” or “specific” surface area. Usually in units of m^2/g . Good for troubleshooting to determine causes of deactivation – shows loss of surface area due to sintering.

Mercury Porosimetry – gives pore size distribution of catalyst – good as compliment to BET surface area

Various bend and crush strength tests – important in determining the physical durability of the catalyst, especially important for catalyst regeneration.

Attrition Tests – complementary to bend and strength tests – focuses on the strength of the ceramic material itself.



Publication Resources

“Protocol for Laboratory Testing of SCR Catalyst: 2nd Edition,”
EPRI, Product ID: 1014256, Palo Alto, CA, 2007.

“Guideline for the Testing of DeNOx Catalysts – VGN-R302He 2nd Edition,” VGB – Technical Association of Large Power Plant Operators, Essen, Germany, 1998.

“Supplement to VGB-302He 2nd Edition – Common Best Practices for Bench Scale Testing and Chemical Analysis of SCR DeNOx catalyst,” STEAG AG, Essen, German, 2002.



QUESTIONS ?