REINHOLD ENVIRONMENTAL Ltd.

2018 NOx-Combustion Round Table & Expo Presentation

February 19-20, 2018, in St. Louis, MO / Hosted by Dynegy

All presentations posted on this website are copyrighted by Reinhold Environmental, Ltd (RE). Any unauthorized downloading, attempts to modify or to incorporate into other presentations, link to other websites, or obtain copies for any other uses than the training of attendees to RE's Conferences is expressively prohibited, unless approved in writing by RE or the original presenter. RE does not assume any liability for the accuracy or contents of any materials contained in this library which were presented and/or created by persons who were not employees of RE.

Innovative Combustion Technologies, Inc.

Lessons Learned/Getting the Most from Bench-scale Testing

Presentation Highlights

1. ICT Background
2. Bench-scale vs. Micro-scale
3. Catalyst Sample Prep
4. Catalyst Handling/Shipping
5. Target Testing Conditions
6. Testing Equipment
7. Value of Good Recordkeeping
8. Importance of Baseline Testing

Who We Are

- Focused on providing services to power plants to improve unit operations including:
  - Optimizing pulverizer performance (i.e. fuel fineness, air/fuel balance, etc.)
  - Boiler/SCR tuning programs to maximize operating efficiencies
  - Environmental testing for HAPs for compliance and APC performance testing

Catalyst Testing Facility

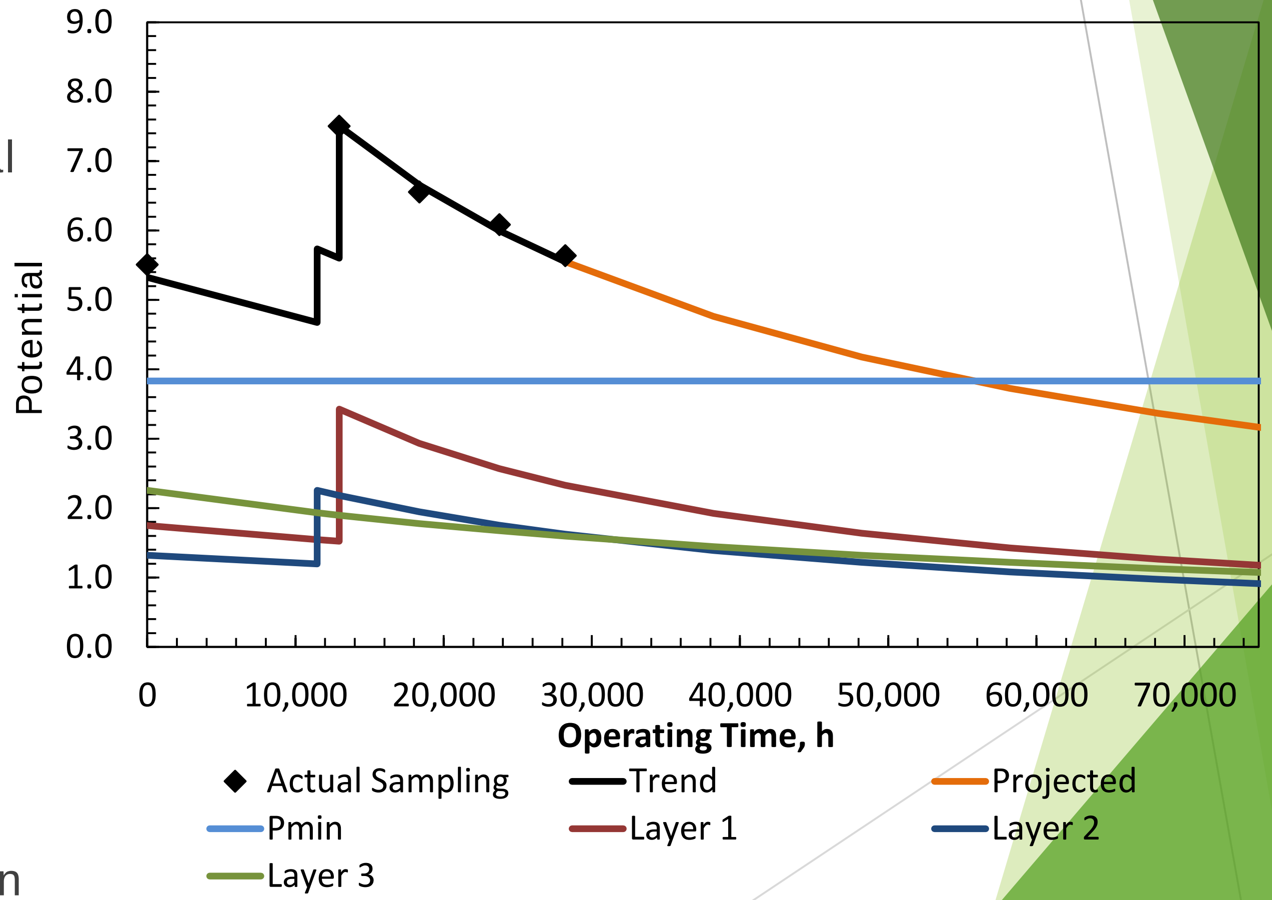
- In November 2016, ICT reached an agreement with Southern Research to take over management of their SCR catalyst testing lab
- Have tested over 800 catalyst samples at the facility for several major utilities/catalyst suppliers
- Will construct like-in-kind facility at Pelham office to be in operation late 2018

Catalyst Performance Testing: Typical Test Methods

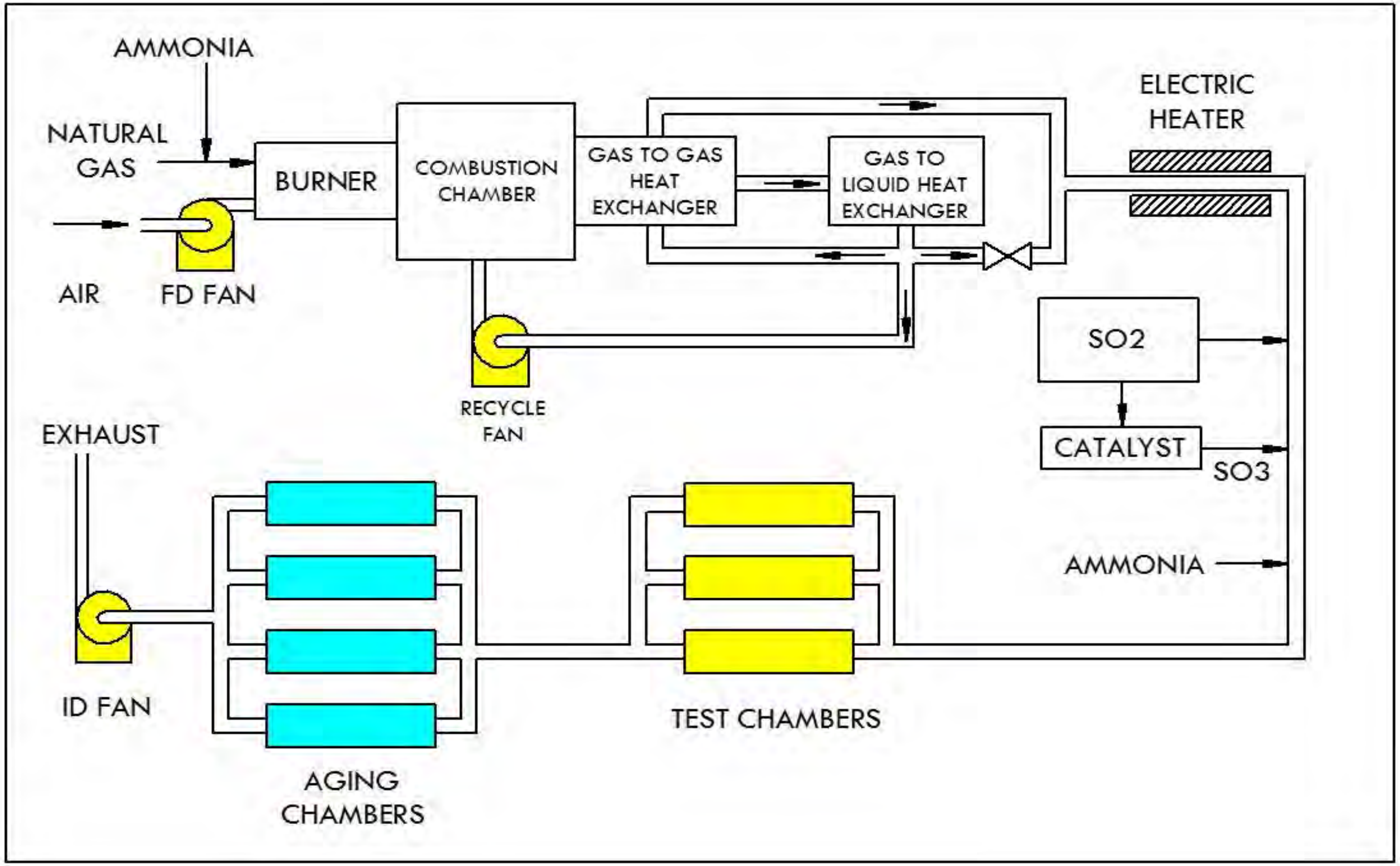
- deNO$_x$ Activity, k
  - Catalyst's ability to reduce NO$_x$
- SO_2-SO_3 Conversion
  - Monitor SO_2 oxidation to mitigate downstream equipment fouling and "blue plume" emissions
- Chemical Composition
  - XRF Analysis (surface and bulk) for formulation and poisoning/fouling species
- Physical Properties
  - BET surface area
  - Physical strength
  - Others

Catalyst Performance Testing: Value of Data

- deNO$_x$ Activity (k)
  - Track deactivation trends
  - Calculate total reactor potential
  - Project remaining catalyst lifetime
- SO_2 - SO_3 Conversion
  - Track changes in conversion performance
  - Schedule replacement before major corrosion/ABS event
- Chemical Composition
  - Determine primary causes of deactivation
  - Evaluate changes in deactivation mechanisms with fuel changes



Bench-Scale Schematic



Bench-Scale vs. Micro Scale

Bench-Scale Lab:

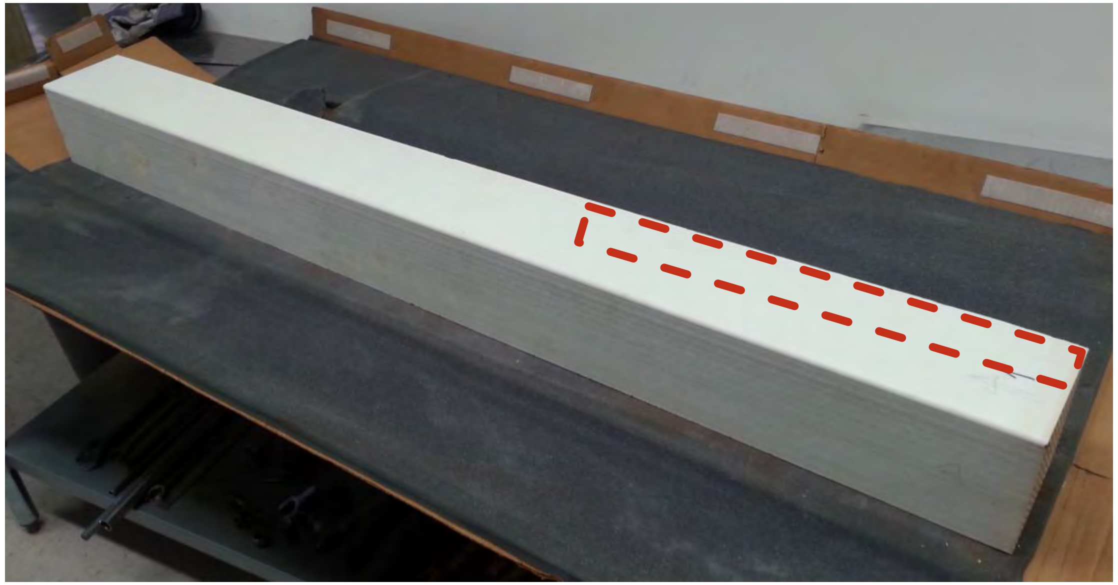
- Natural gas furnace for flue gas generation
- Sized for full-length sample elements, 150 x 150 mm cross-section
- Match all key flue gas parameters to full-scale; no corrections required
  - Moisture
  - Flow
  - Temperature
  - Composition (NO_x, SO_2, O_2)

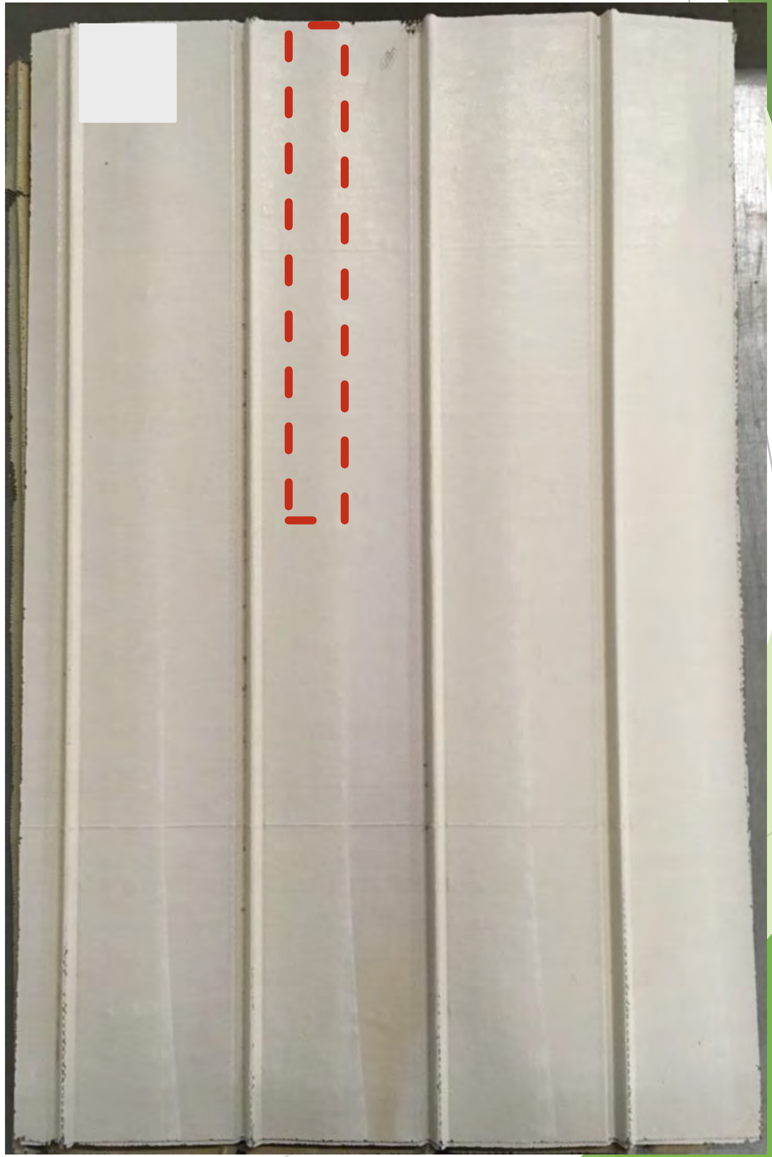
Micro-Scale/Semi-Bench Lab:

- Gas cylinder fed
- Reactor sized for smaller test elements, both cross-section and length
- Match most flue gas parameters; corrections must be applied to test results
  - Not full-scale length
  - Correct results for flow, moisture, etc.

Bench-Scale vs. Micro Scale

- Deactivation not uniform over length of catalyst
  - Leading edges deactivate more rapidly
- Micro-scale test elements not full length or cross-section
  - Results may not be representative



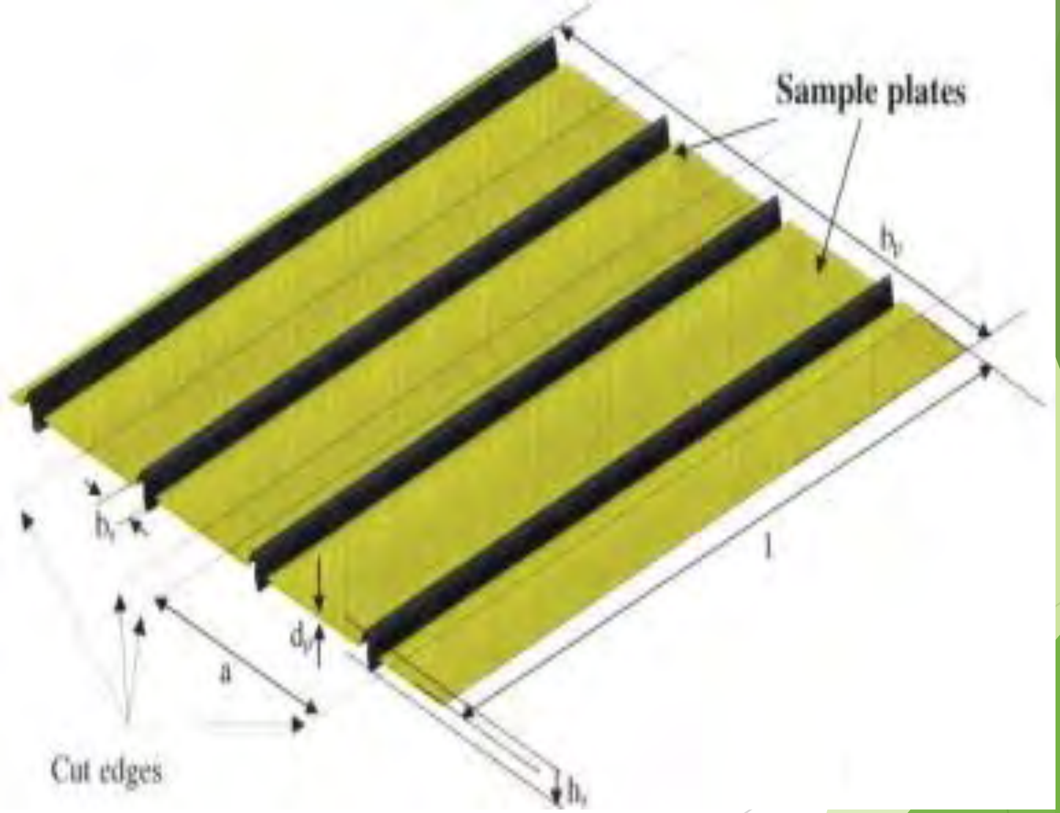


Catalyst Sample Prep: Intro

- All samples require some level of prep and physical measurement prior to testing
  - Cut down to fit 150 x 150 mm cross-section
  - Measure length, width, pitch, etc.
  - Discount any plugging/lost ceramic material
- Three main types:
  - Plates
  - Honeycomb
  - Corrugated (uncommon)

Catalyst Sample Prep: Plates

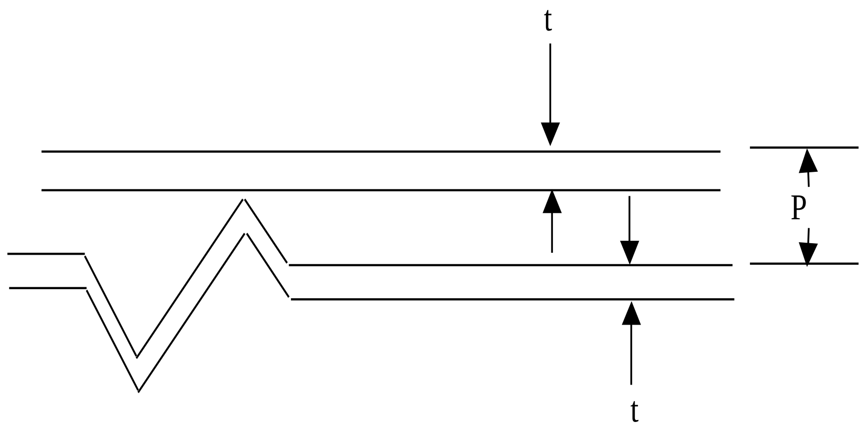
- Typical plate elements are 450 mm wide by full sub-layer length
- Plates are sampled in pairs from various modules of the top sub-layer
  - 14-16 plates for each test element
- Plates cut into coupons with 150 mm width for testing
  - Usually cut two coupons from each plate
  - 22-26 coupons to fill test box (varies according to element pitch)



Catalyst Sample Prep: Plate Calculations

Physical Measurements:

- Coupons
  - Length
  - Width (inlet/outlet)
  - Thickness (inlet/outlet)
  - Ceramic material loss estimate
- Sample Box
  - Width
  - Height

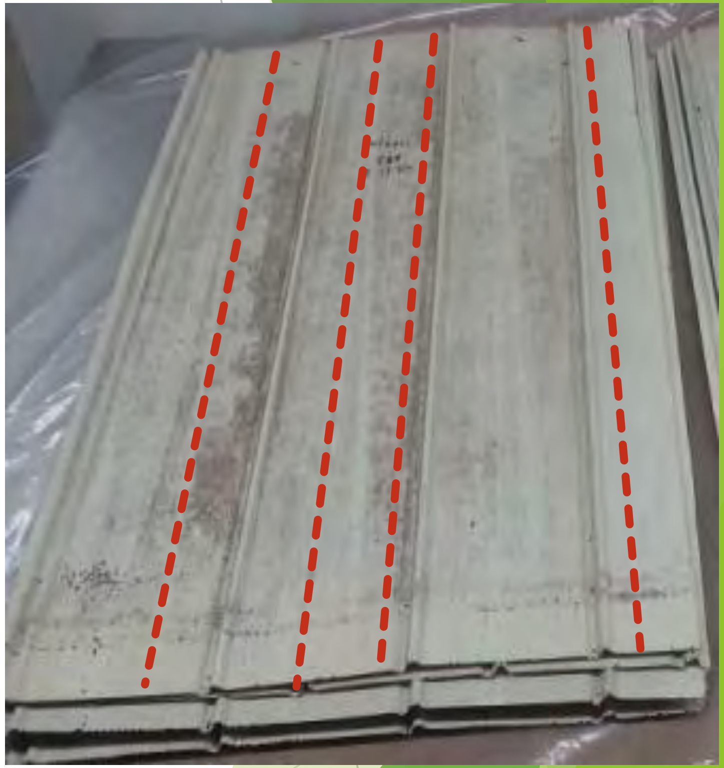


Calculations:

- Sample surface area
- Sample volume
- Face area (total/blocked)
- Target flow

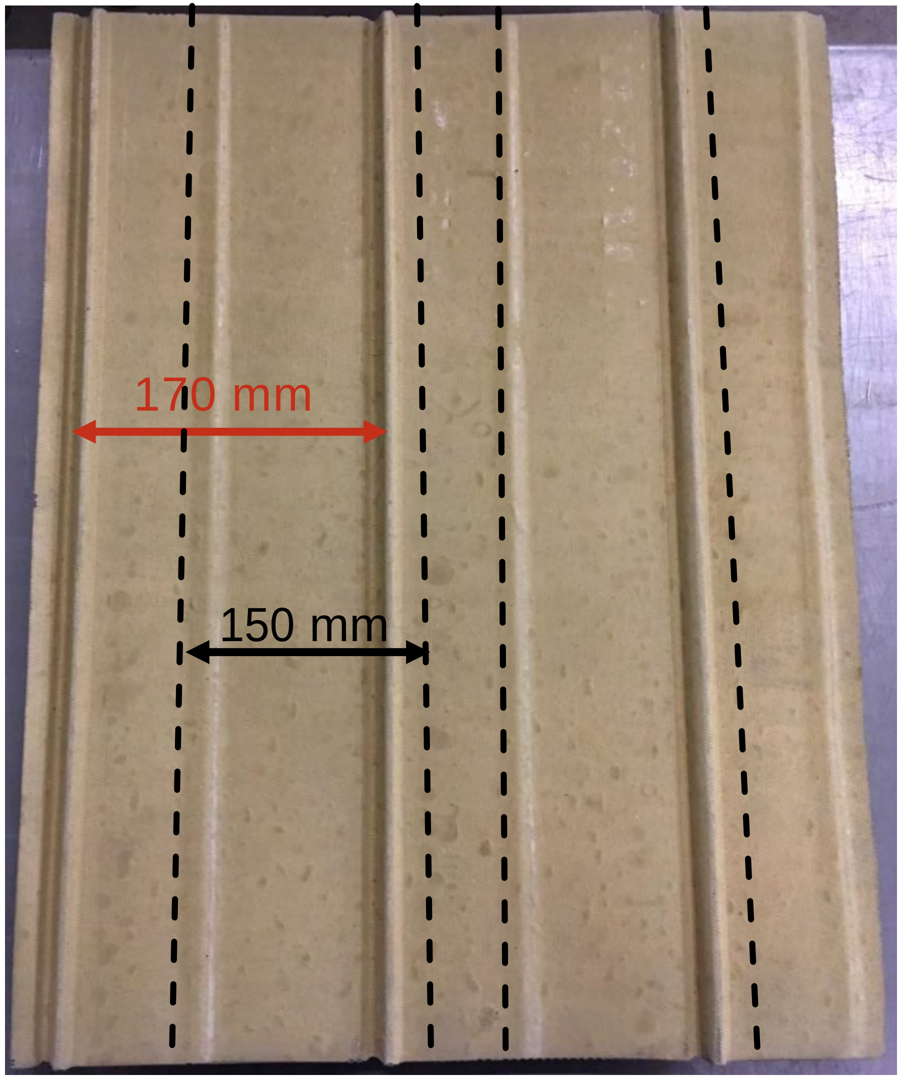
Catalyst Sample Prep: Plates

- 22-26 plate coupons required to fill 150 x 150 mm cross-section
  - 5.7 mm pitch: 26 coupons
  - 7.0 mm pitch: 22 coupons
- Bench-scale test element should closely match full-scale spacing
- Uneven stacking and damaged or bowed plates can impact bench-scale testing results
  - Non-uniform flow distribution through test element
  - Portions of catalyst material not exposed to flue gas

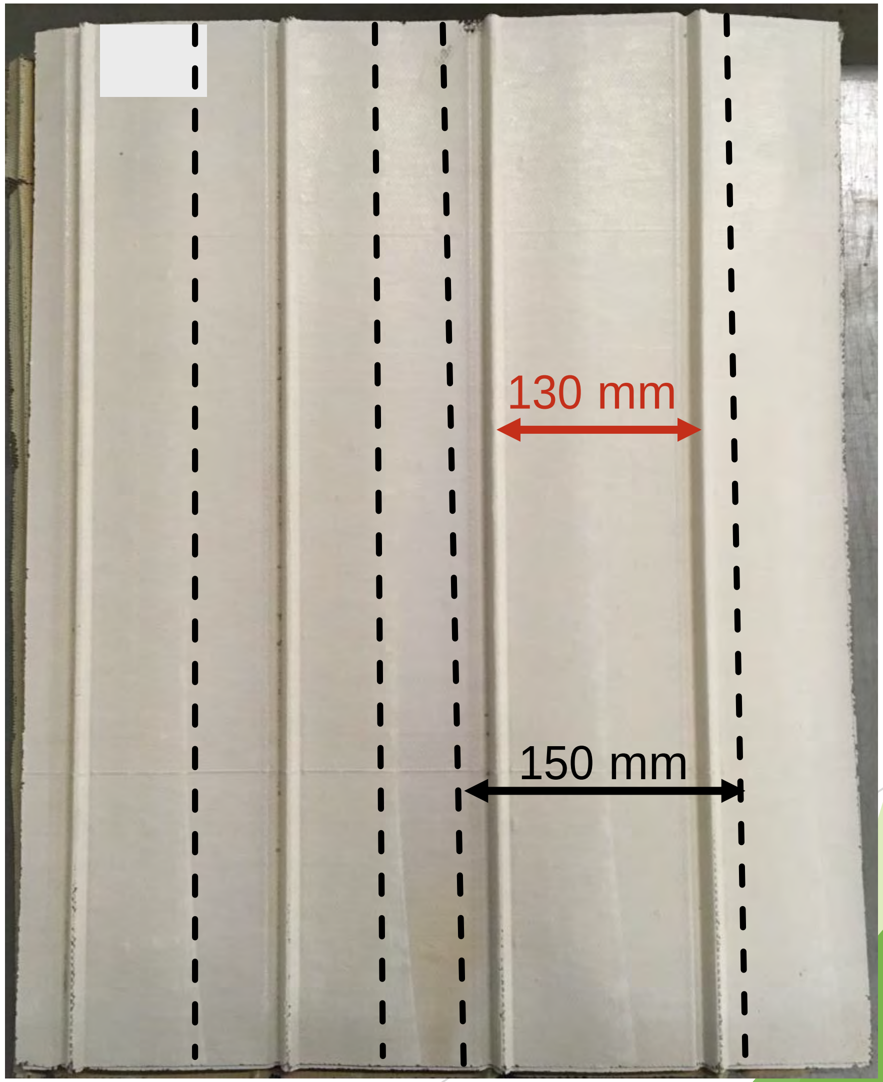


Catalyst Sample Prep: Plates

3-Notch Plate



4-Notch Plate

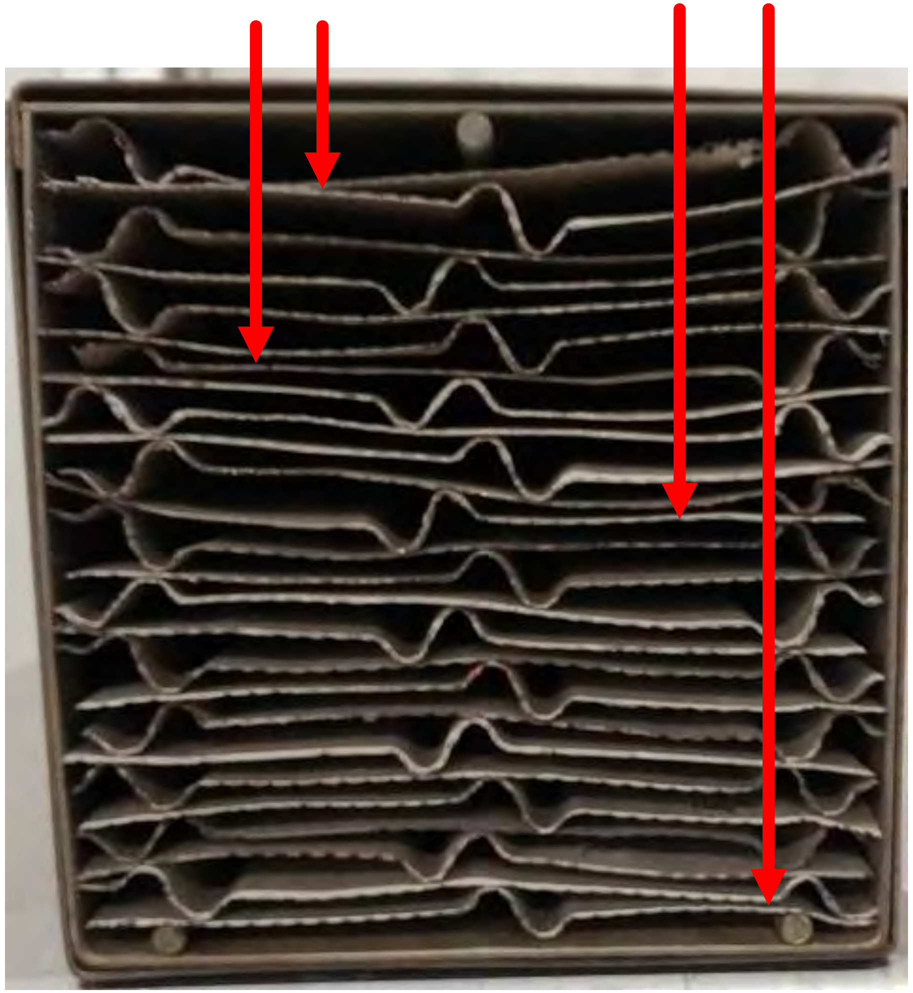


Catalyst Sample Prep: Plates

3-Notch Plate

4-Notch Plate

Catalyst Sample Prep: Plates



Initial Stacking

Restack

Catalyst Sample Prep: Plates

Initial Stacking

Re-Stack

Activity: 38 m/h

Bias: -6.1%

Activity: 40.5 m/h

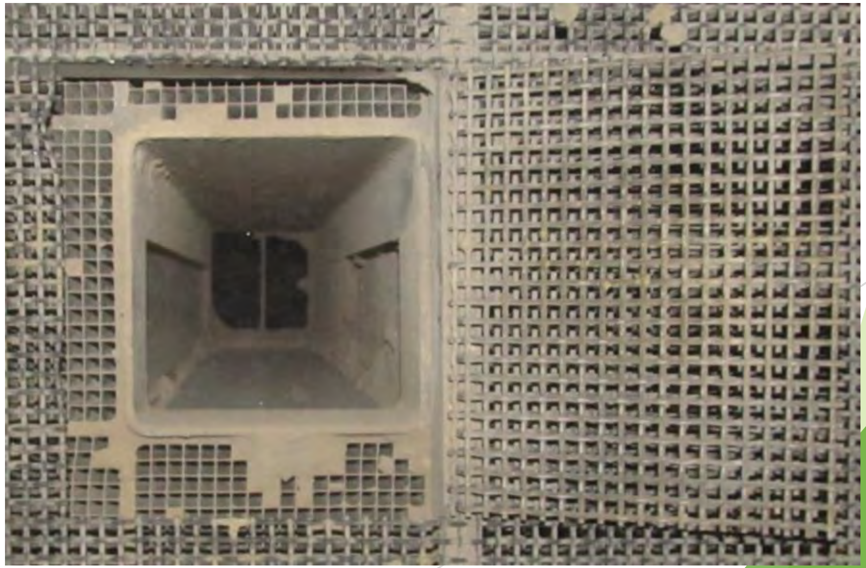
Catalyst Sample Prep: Plates

- Stacking most critical for testing new/regenerated samples
  - Used for guarantee verification
- Typically receive larger quantity of material; no flow direction restriction

	Initial Stacking	Restack
Activity (m/h)	36.2	41.9
Bias	-13.6%	

Catalyst Sample Prep: Honeycombs

- Several test blocks installed in various locations across the layer
- Test elements shaved on two sides to fit in metal casing
- Must be removed from metal casing prior to testing
  - Samples are very brittle
  - Minimize shock to elements
  - Ash caking makes removal difficult at times



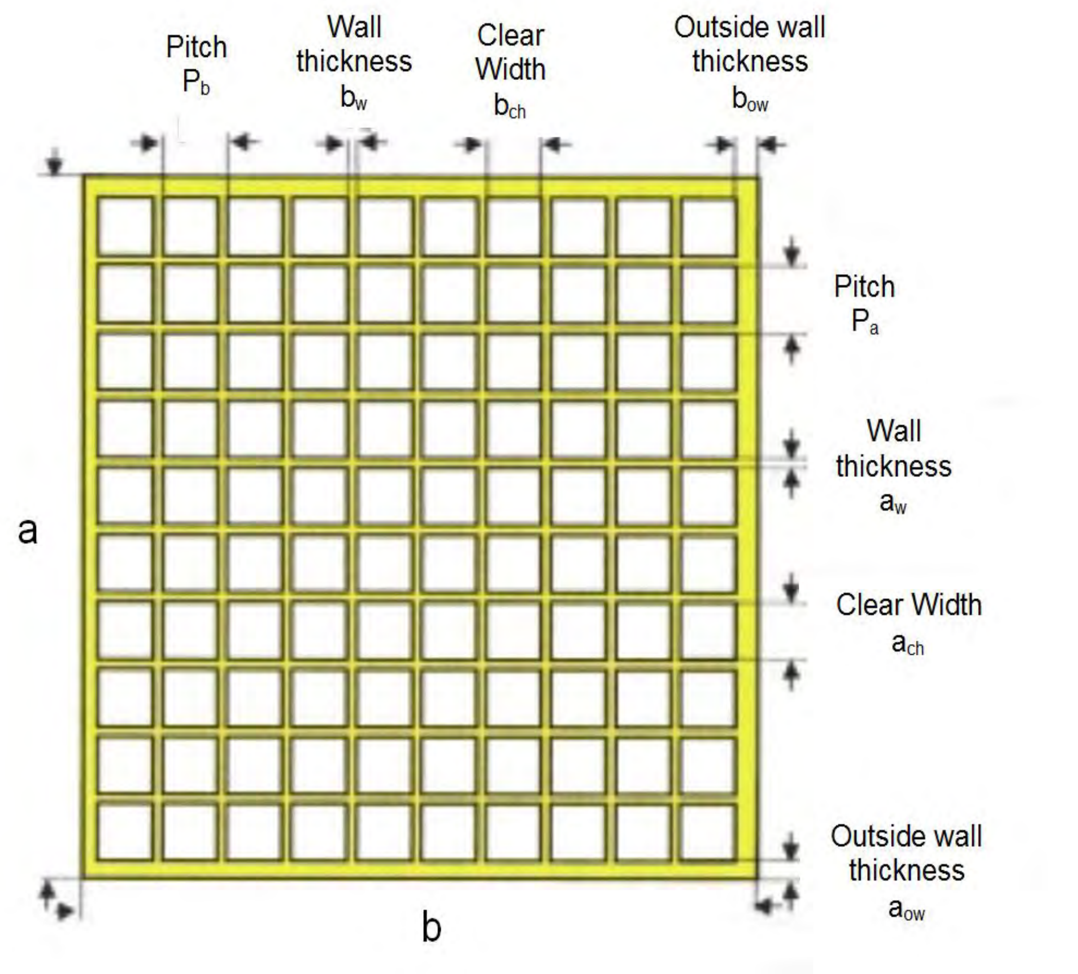
Catalyst Sample Prep: Honeycomb Calculations

Physical Measurements

- Sample length (all four sides)
- Channel opening
- Wall thickness
- Block face height/width
- Plugged channels

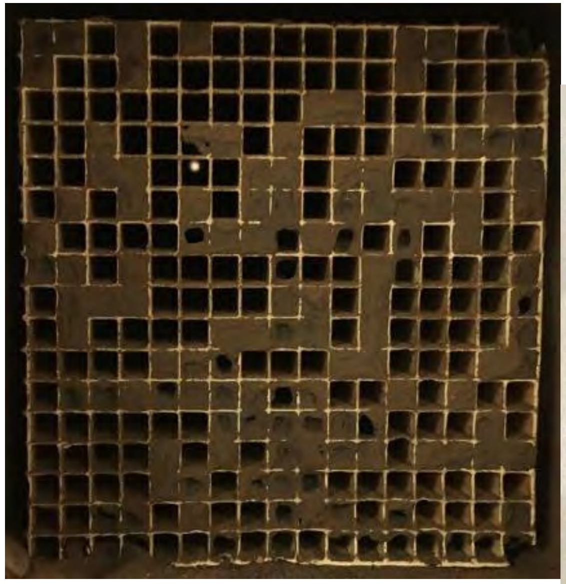
Calculations:

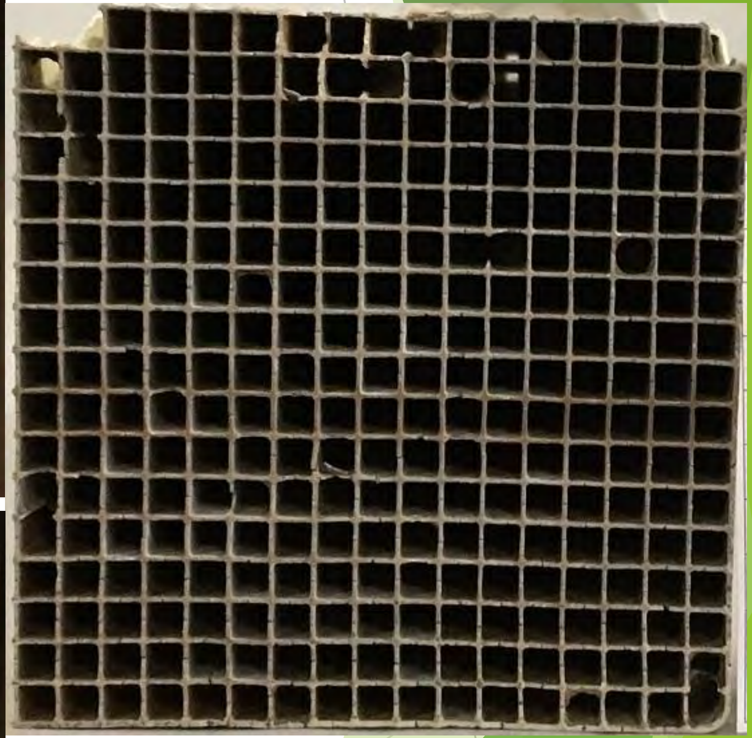
- Sample surface area
- Sample volume
- Face area (total/blocked)
- Target flow



Catalyst Sample Prep: Honeycombs

- Must clean sample to reduce plugging below 20%
  - Minimize number of cells unplugged to maintain representative sample quality
  - Necessary to achieve desired channel velocity during testing
  - Plugging not just at catalyst face; within channels as well





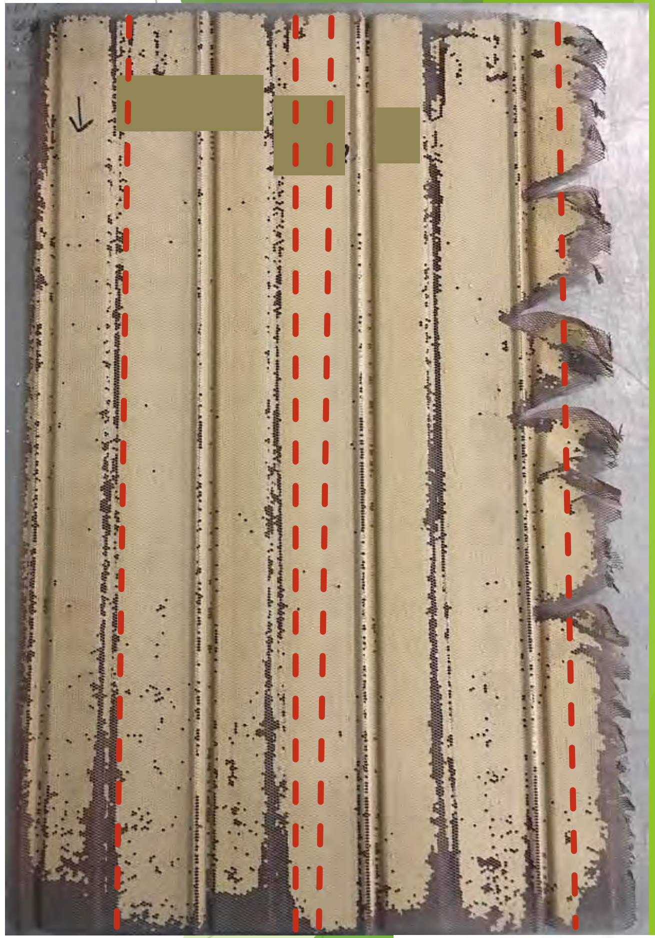
Catalyst Sample Handling/Shipping: Intro

- Catalyst material must be handled with care throughout the sampling/shipping process
- Mishandling samples can impact the quality of results
  - Bent/bowed plates
    - Improper stacking in test boxes
  - Excessively plugged/broken honeycomb elements
    - Can be untestable
  - Contamination/water damage
- Ensure samples are properly labeled
  - Plant, Unit, Reactor, Layer
  - Flow direction

Catalyst Sample Handling/Shipping: Plate Catalyst

Sampling Notes:

- Minimize damage to leading edges
- Avoid tearing/ripping material, particularly along edges
  - Older/regenerated material tends to be more fragile
  - Discard excessively damaged plates
- Immediately ID each plate including flow direction
- Select plates from randomly distributed modules
- Collect reference material before installation, if possible



Catalyst Sample Handling/Shipping: Plate Catalyst

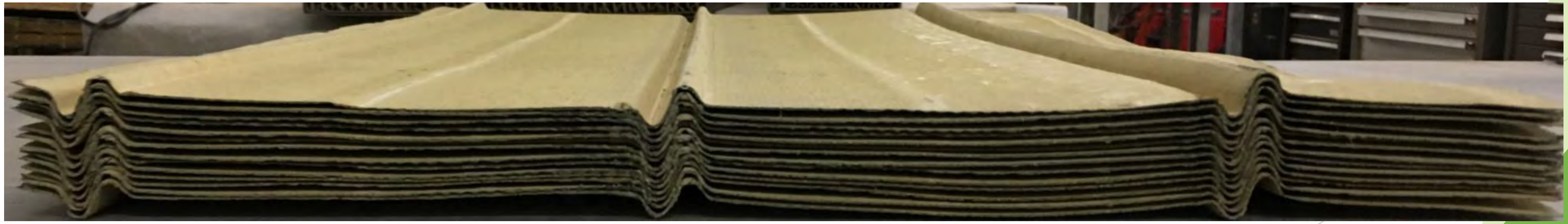
Shipping Notes:

- Packing/carrying samples in garbage bags results in bowing
- Protect samples from damage/contamination during shipping
  - Wrap in plastic/bubble wrap
  - Avoid excessive stacking to prevent crushing notches
  - Line up notches
- Select crate/box large enough for easily remove samples
  - Pack with paper/bubble wrap

Catalyst Sample Handling/Shipping: Plate Catalyst

Notches Not Aligned

Notches Aligned



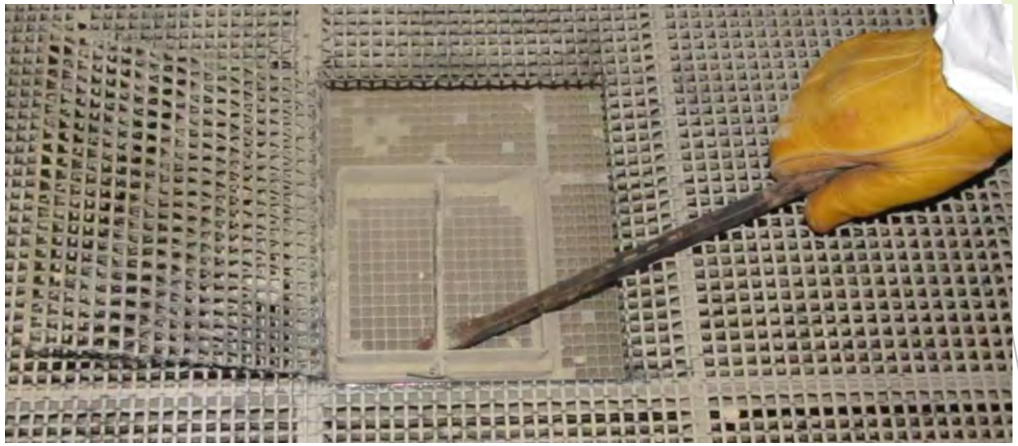
Catalyst Sample Handling/Shipping: Plate Catalyst

INNOVATIVE COMBUSTION TECHNOLOGIES, INC.

Catalyst Sample Handling/Shipping: Honeycomb Catalyst

Sampling Notes:

- Honeycombs are much more fragile than plates
- Avoid physical shock to element block, if possible
  - Fissures within element
  - Protect element face
- Select sample elements with minimal plugging
  - Excessively plugged elements not representative
- Immediately ID element on sheath



Catalyst Sample Handling/Shipping: Honeycomb Catalyst

Shipping Notes:

- Less labor intensive compared to plates
- Wrap elements in plastic to prevent contamination
- Package in crate/box such that elements will not shift/fall during shipment

Catalyst Sample Handling/Shipping: Honeycomb Catalyst

Target Testing Conditions: Intro

Key Flue Gas Parameters:

- Temperature, °F
- Oxygen (O_2), vol%, dry
- Moisture (H_2O), vol%
- Nitric Oxide (NO_x), ppmvd
- NH_3:NO_x Ratio/Alpha (α)
- Sulfur Dioxide (SO_2), ppmvd
  - Sample conditioning
- Sulfur Trioxide (SO_3), ppmvd
  - Not standard at sample inlet
- Flow Rate
  - Face velocity
  - Channel velocity

Parameter	Accuracy	Maximum Drift
Temperature	±4.5°F (±2.5°C)	4.5°F (2.5°C)
Linear Velocity	±5% of target	±2% of value
O_2	±1% of target	±0.5% of value
NO_x	±1% of target	1 ppm
SO_2	±1% of target	±0.5% of value
NH_3:NO_x	±2% of target	±2% of value

Target Testing Conditions: Flow

- Full-scale vs. Bench-scale flow
  - Power plant: >1,000,000 Nm^3/h
  - Bench-scale facility: 90-220 Nm^3/h
- Match linear velocities to design
  - Cell blockage for honeycombs
  - Material loss for plates
- Face and channel velocities are not the same
  - Face velocity (also known as superficial velocity)
    - Velocity at the face of the catalyst
    - Function of volume flow and layer cross sectional area
  - Channel linear velocity
    - Velocity in the channels of the catalyst
    - Function of face velocity and catalyst void fraction (supplied by manufacturer or based on geometric measurements)
    - Takes catalyst pitch into account

Target Testing Conditions: Flow

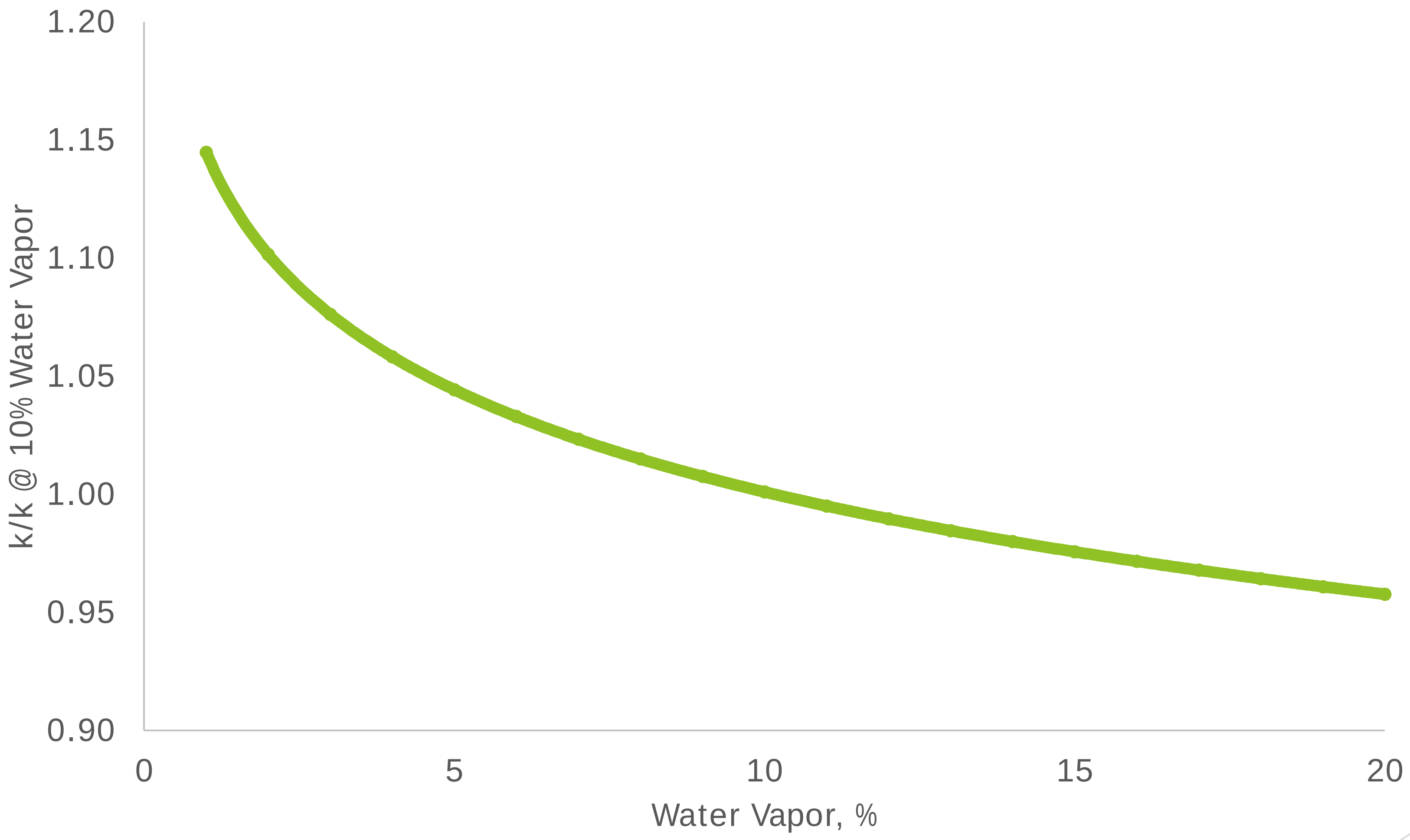
- Periodic pitot testing to prevent excessive flowmeter drift
  - Marginally Low flow
    - Longer residence time
    - High bias in measured k (or k_0)
    - Overestimating deactivation trend
    - Underestimating catalyst lifetime
  - Marginally High flow
    - Shorter residence time
    - Low bias in measured k (or k_0)
    - Underestimating catalyst deactivation
    - Overestimating catalyst lifetime

Target Testing Conditions: Moisture

- Moisture correction curve published by EPRI in SCR Catalyst Testing Protocol: 2nd Edition
- Lab generated moisture content may not match full-scale moisture content
- Tests conducted to validate moisture correction curve (at high and low moisture content)

Full-Scale	Units	Bituminous Target Conditions	PRB Target Conditions
Temperature	°F	694	745
Channel LV	act m/s	5.70	5.55
Inlet NO_x	ppmvd	349	107
Inlet SO_2	ppmvd	1,533	346
O_2	%	5.0	3.5
H_2O	%	7.8	14.1

Target Testing Conditions: Moisture



Target Testing Conditions: Moisture

	Units	Bituminous (Target)	Bituminous (High Moisture)
Moisture	%	7.9	14.6
$k_{measured}$	m/h	34.6	37.2
$k_{corrected}$	m/h	-	35.8
Error	%	-	+3.34

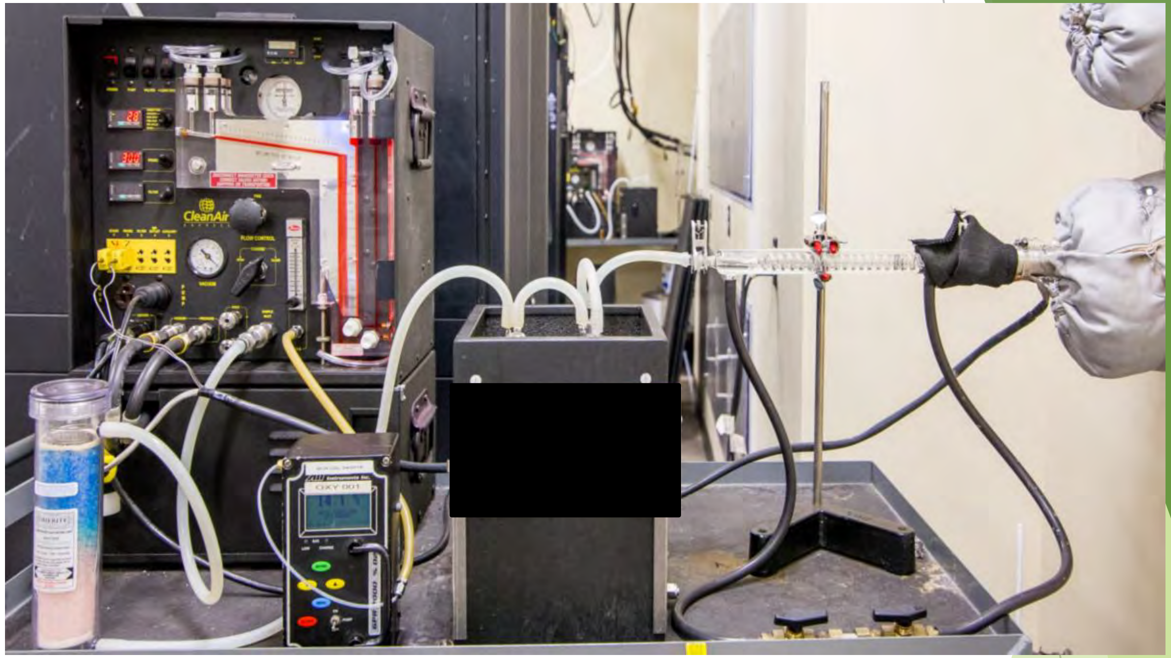
	Units	PRB (Target)	PRB (Low Moisture 1)	PRB (Low Moisture 2)
Moisture	%	14.1	7.1	9.1
$k_{measured}$	m/h	40	38.2	38.4
$k_{corrected}$	m/h	-	39.9	39.5
Error	%	-	-0.23	-1.23

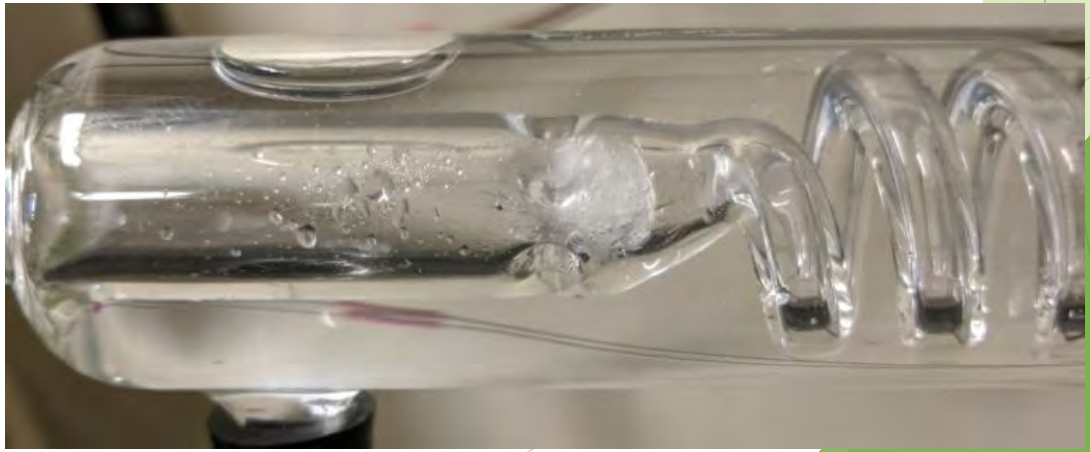
Testing Equipment: Intro

- Flue gas composition analysis
  - FTIR analyzers at inlet and outlet for continuous measurement of most key species
  - Oxygen analyzers (zirconia cell) at furnace exit and sample outlet
  - Heated sample lines to prevent condensation/ABS fouling
- Extractive analysis
  - SO_3 by controlled condensate method (CCM)
  - NH_3 by steam distillation for special applications

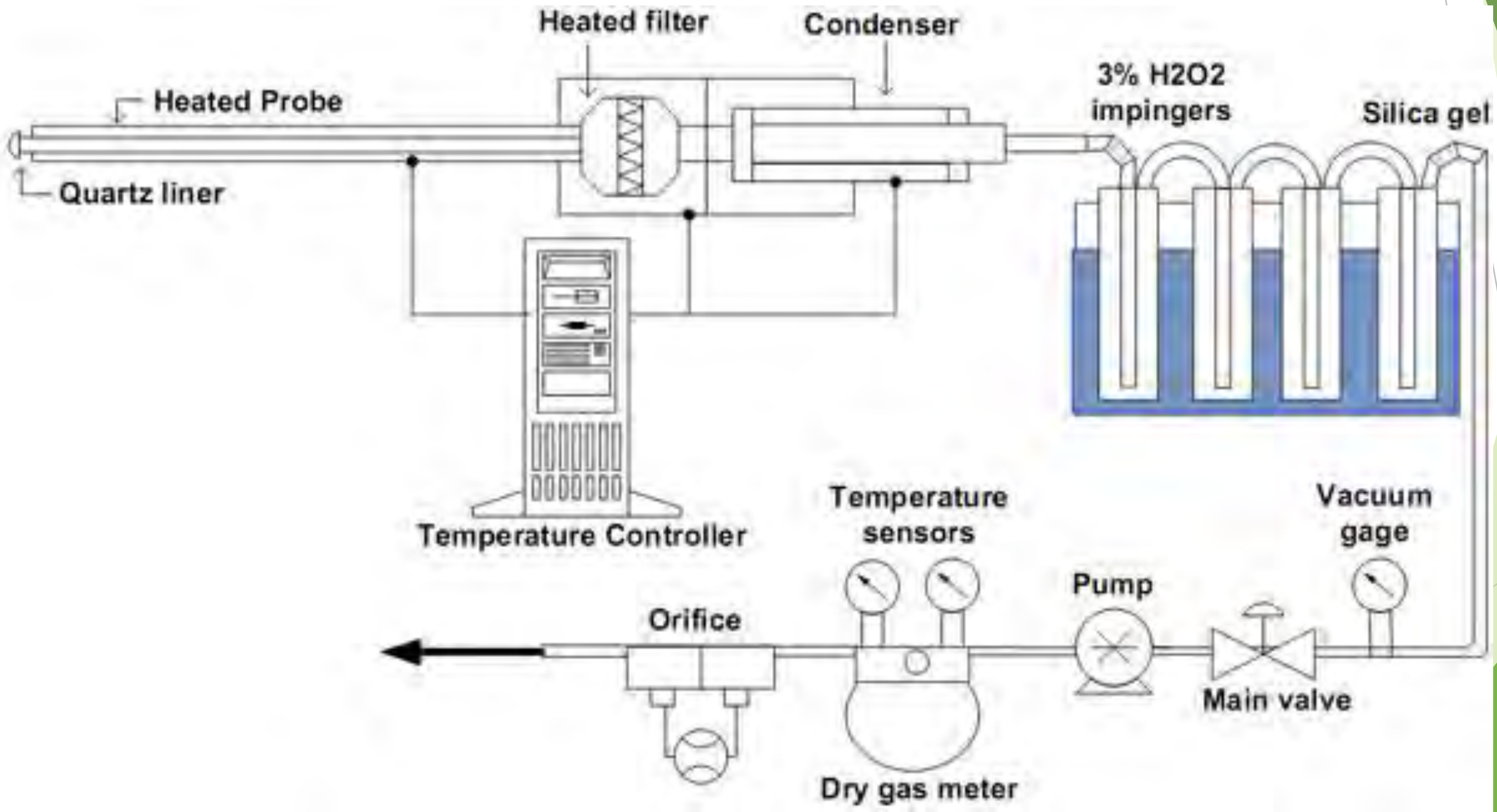
Testing Equipment: SO_3 Sampling

- Temperature, pressure, and O_2 corrected
- Heated quartz-lined probe
- Glass wool prevents SO_3 from passing through condenser
- O_2 leak-checks
  - In-leakage leads to inaccurate SO_3 concentrations
  - Handheld O_2 meters
  - 10-15 in Hg. vacuum
- Important to regularly verify dry gas meter pump calibration
  - Corrosive sample gas can quickly alter calibration factor





Testing Equipment: SO_3 Sampling



Testing Equipment: SO_3 Sampling

- SO_3 measurement method
  - Barium ion titration
  - Same day analysis
    - Ensures no trending in results
  - Highly accurate/precise titrations
  - Low-cost and convenient
- Stability is established when
  - Each subsequent value is not consistently higher/lower than the last
  - Mean slope ≤2%
  - Relative standard deviation ≤10%

INNOVATIVE COMBUSTION TECHNOLOGIES, INC.

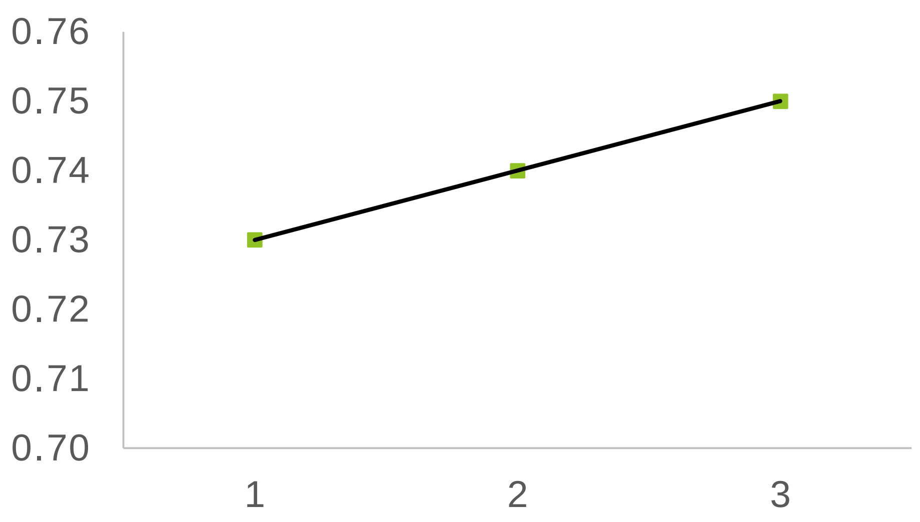
Testing Equipment: SO_3 Sampling

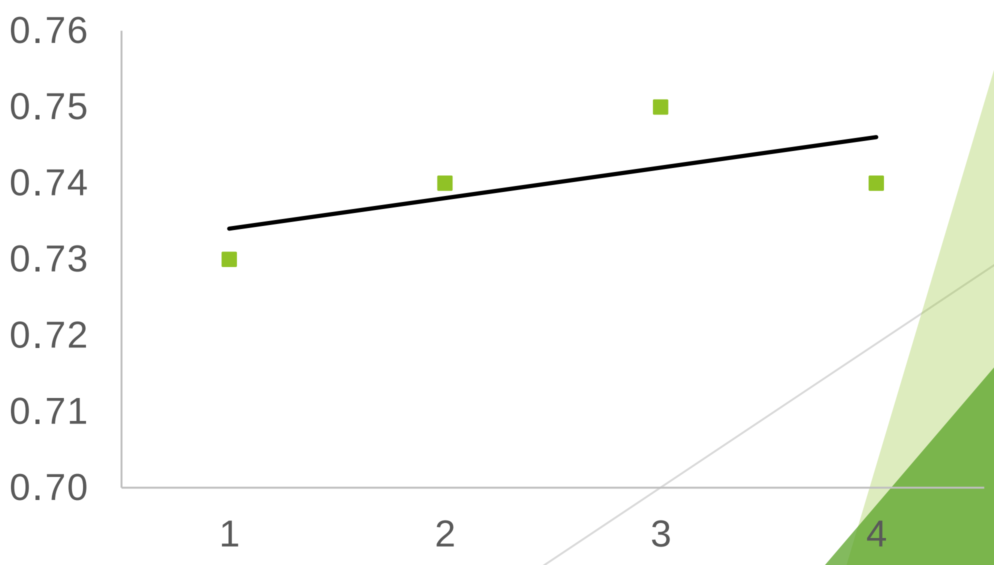
Run	Conversion (mol%)
1	0.73
2	0.74
3	0.75

Slope	Standard Deviation
1.35	1.35

Run	Conversion (mol%)
1	0.73
2	0.74
3	0.75
4	0.74

Slope	Standard Deviation
0.54	1.10





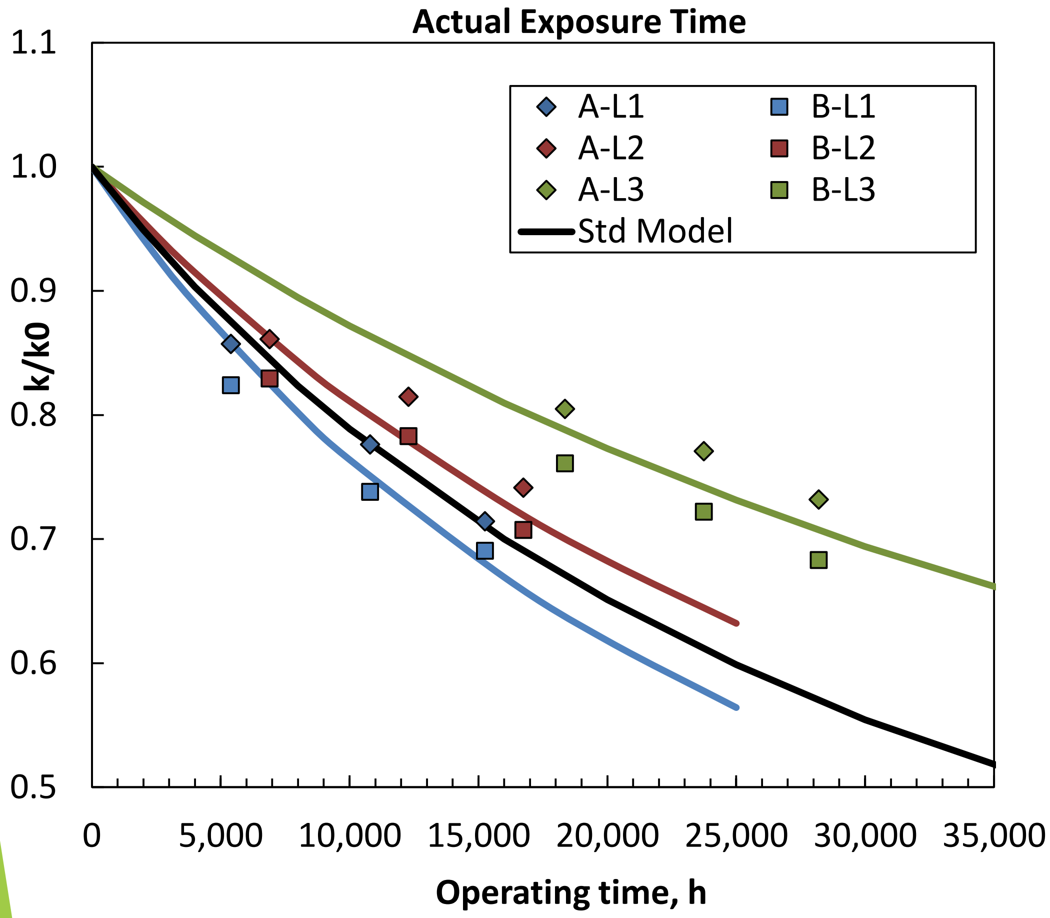
Value of Good Recordkeeping: Intro

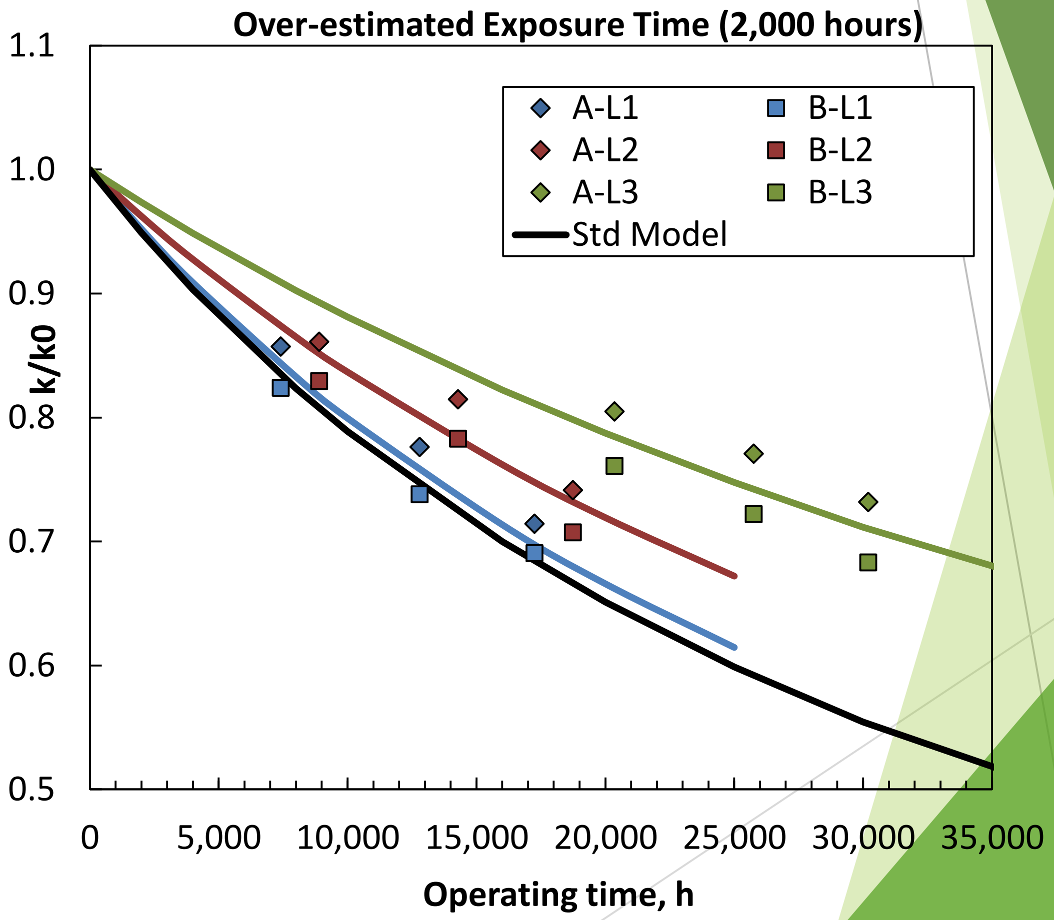
- Catalyst exposure hours
  - From date of install to sampling date
  - Maintain consistent checkpoints each year
- Catalyst design parameters
  - Installed catalyst volume
  - Specific surface area
  - Other reactor specific info
- Reactor design vs. field tests
  - Adjust future catalyst testing parameters as necessary
- Historical catalyst testing results

Value of Good Recordkeeping: Hypothetical Plant Background

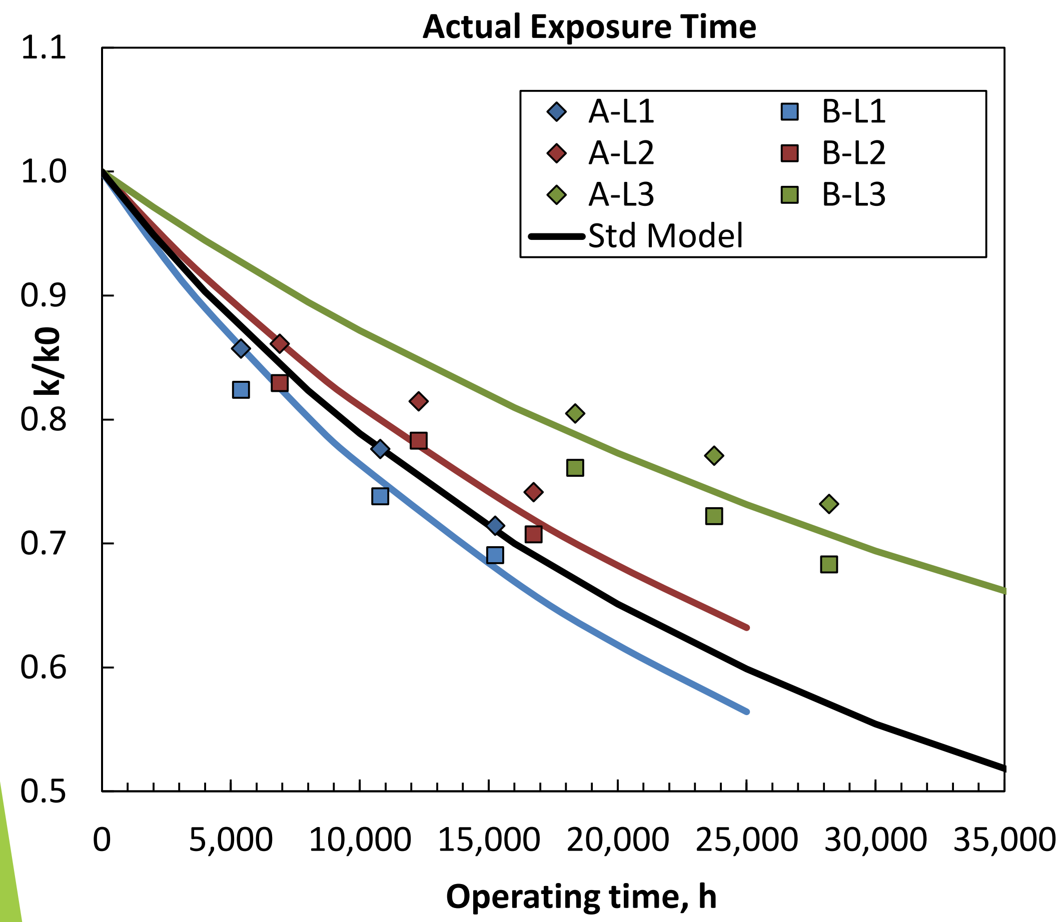
- Bituminous coal-fired boiler with no hot-side ESP
  - Standard catalyst deactivation: $k/k_0 = 0.70$ at 16,000 hours
- Split SCR reactors with three layers available for catalyst modules
- Inlet NO_x: 325 ppmvd
- deNO_x required: 90%
- NH_3 slip limit: 2 ppmvd
- Flow: 1,600,000 Nm^3/h per reactor
- Layer 1 (Honeycomb):
  - Installed volume: 242 m^3/reactor
  - A_{spec}: 539 m^2/m^3
  - Exposure age: 15,249 hours
- Layers 2/3 (Plate):
  - Installed volume: 251 m^3/reactor
  - A_{spec}: 351 m^2/m^3
  - Exposure age: 16,740 (Layer 2)
    28,200 (Layer 3)

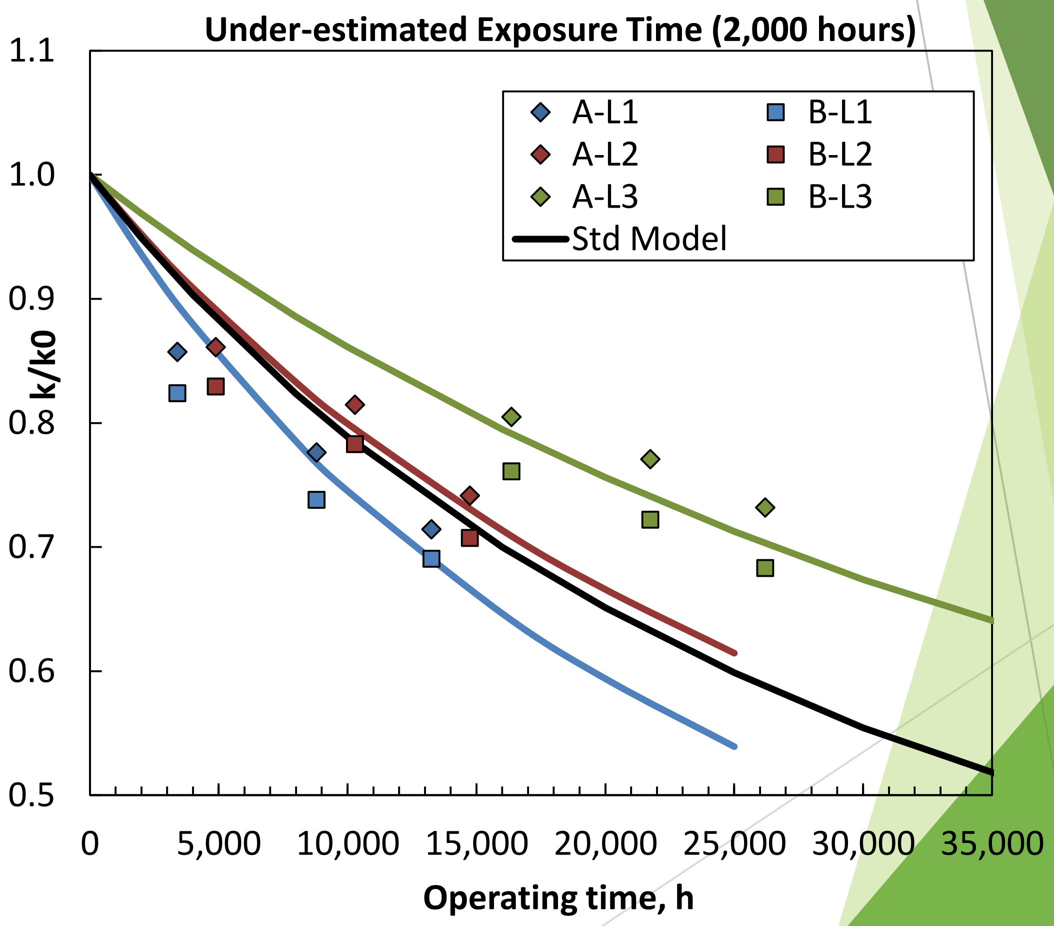
Value of Good Recordkeeping: Activity Trend (Over-estimate Time)



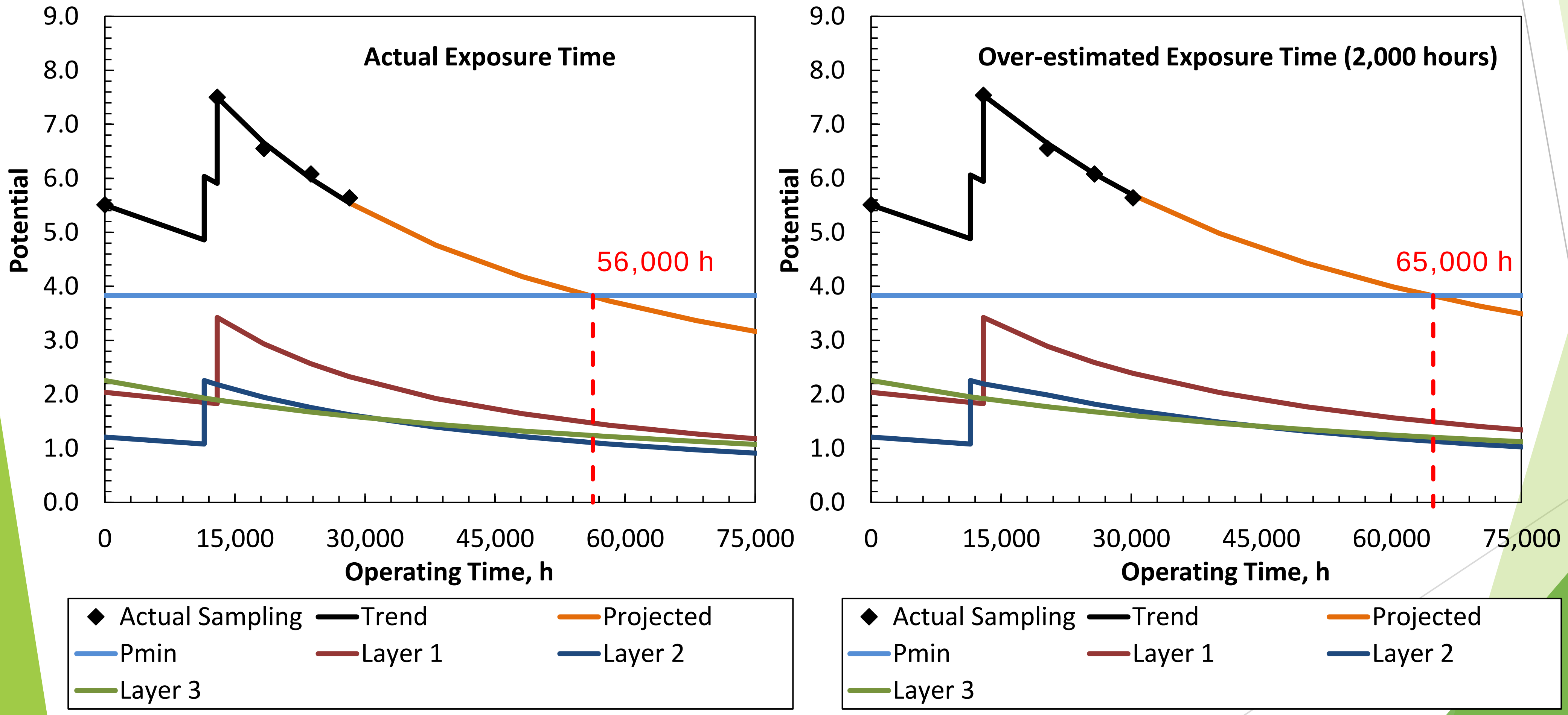


Value of Good Recordkeeping: Activity Trend (Under-estimate Time)

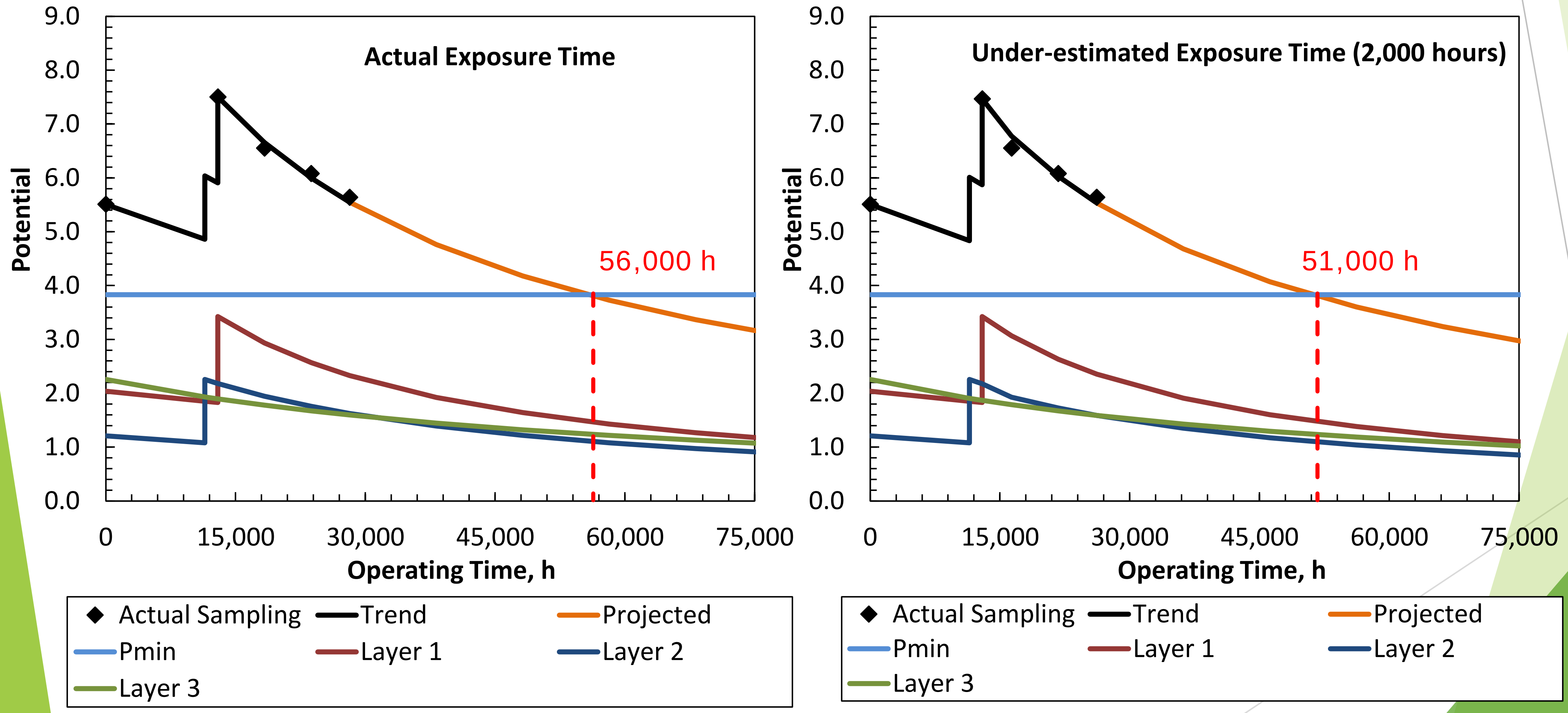




Value of Good Recordkeeping: Reactor Potential Trend (Over-estimate)



Value of Good Recordkeeping: Reactor Potential Trend (Under-estimate)



Importance of Baseline Testing: Intro

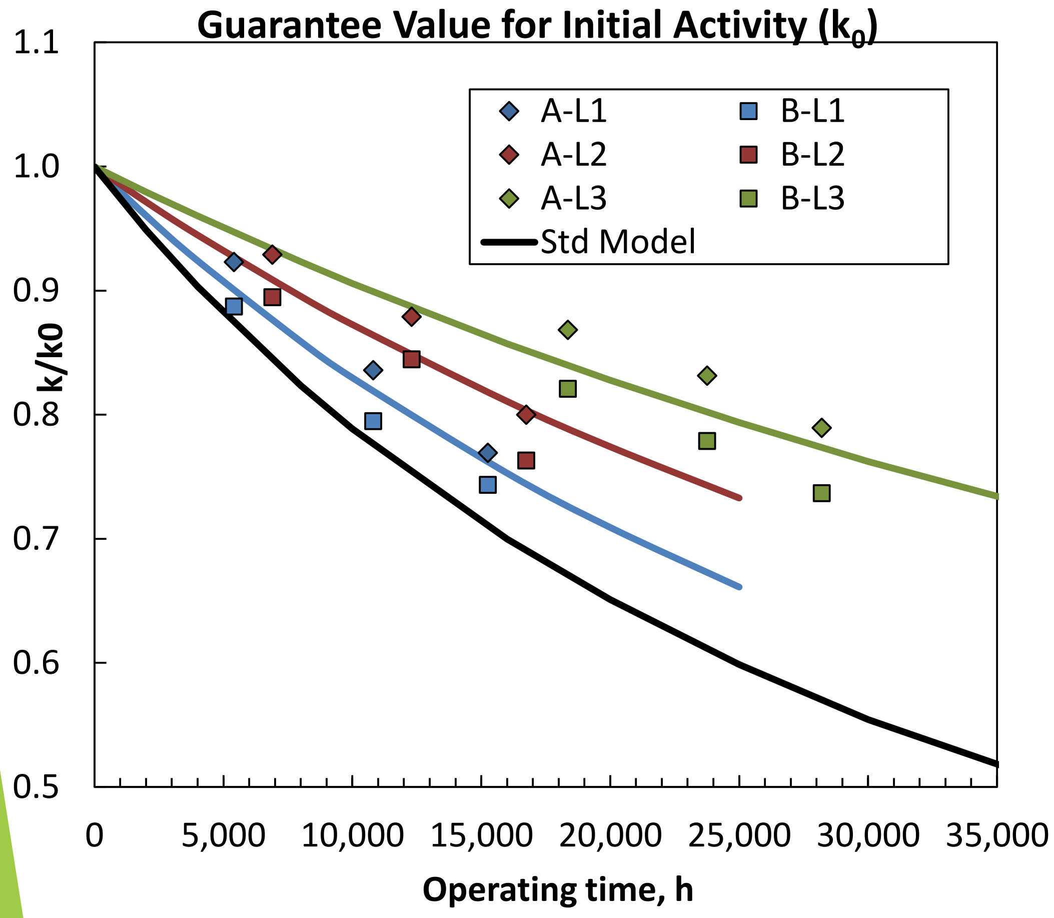
- Vital to test new material for accurate baseline performance
- Manufacturer initial activity guarantees can be lower than actual measured values
- Forgoing baseline testing can lead to:
  - Underestimating catalyst deactivation rates
  - Overestimating remaining catalyst/reactor lifetimes
- Multiple baseline tests provide more representative value

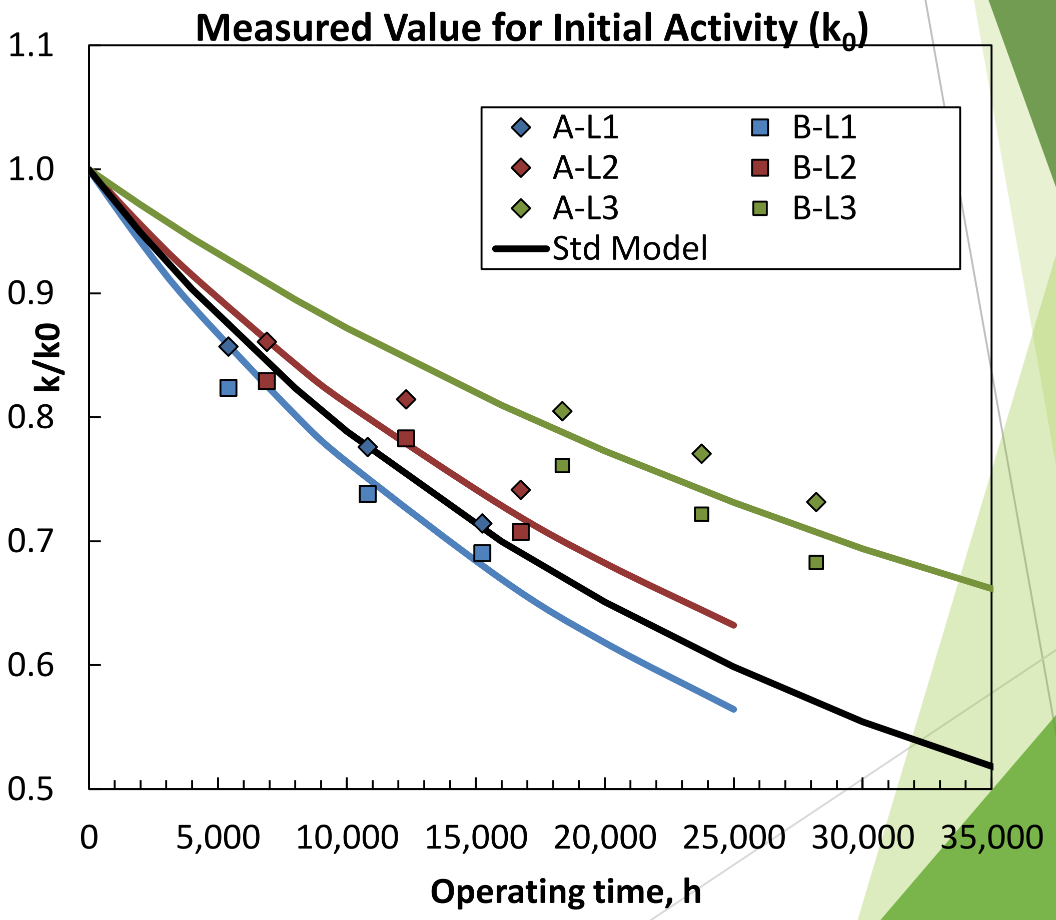
Importance of Baseline Testing: Hypothetical Plant Background

- Bituminous coal-fired boiler with no hot-side ESP
  - Standard catalyst deactivation: $k/k_0 = 0.70$ at 16,000 hours
- Split SCR reactors with three layers available for catalyst modules
- Inlet NO_x: 325 ppmvd
- deNO_x required: 90%
- NH_3 slip limit: 2 ppmvd

- Flow: 1,600,000 Nm^3/h per reactor
- Layer 1 (Honeycomb):
  - Installed volume: 242 m^3/reactor
  - A_{spec}: 539 m^2/m^3
  - Exposure age: 15,249 hours
- Layers 2/3 (Plate):
  - Installed volume: 251 m^3/reactor
  - A_{spec}: 351 m^2/m^3
  - Exposure age: 16,740 (Layer 2)
    28,200 (Layer 3)

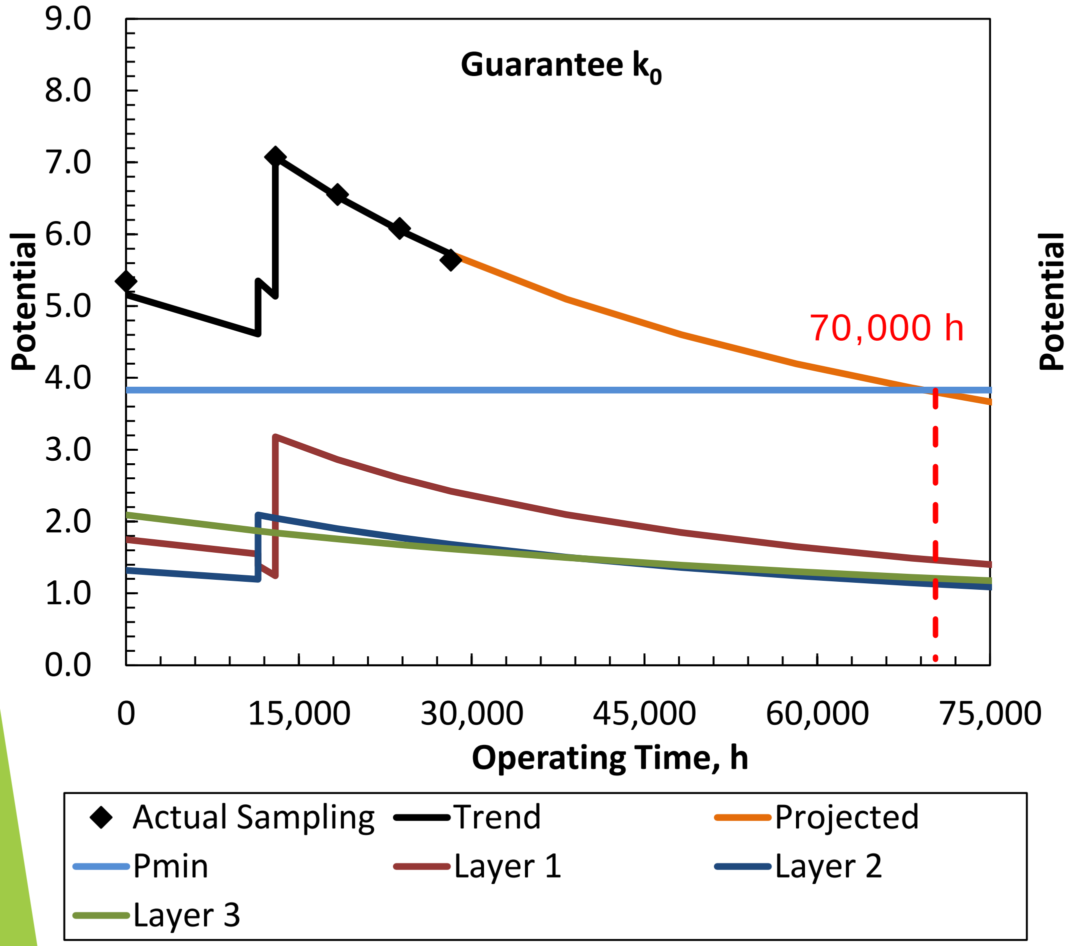
Importance of Baseline Testing: Activity Trend

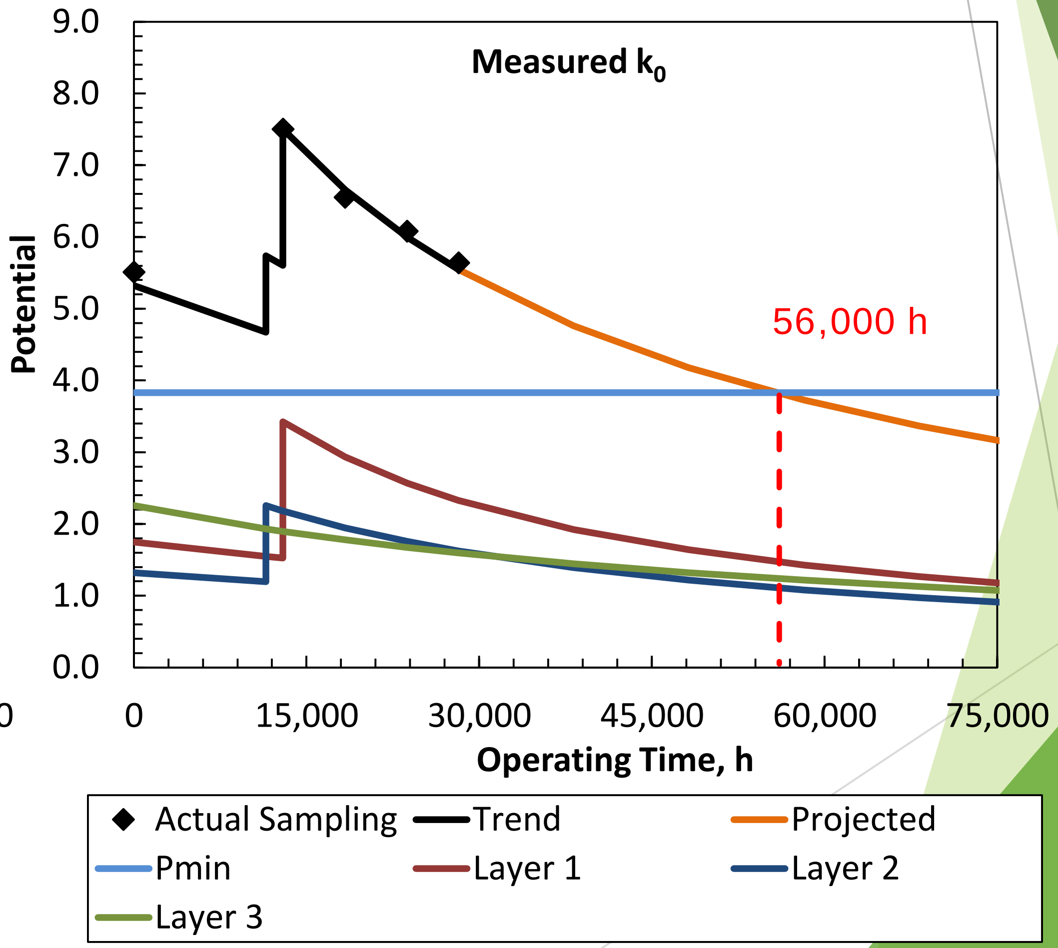




Importance of Baseline Testing: Reactor Potential Trend

Remaining catalyst life over-estimated by 14,000 hours using guarantee k_0 instead of measured k_0





Summary

- Bench-scale testing provides most representative catalyst performance data
  - Can test full-length catalyst elements
  - Test at exact design parameters for SCR system
  - No corrections to results
- Sample handling/prep can have significant impact on results
  - Damage to elements during sampling/shipping can result in improper stacking
  - Contamination can affect sample integrity
- Small inaccuracies impact analytical value of data
  - True initial activity for projections
  - Accurate exposure hours

Innovative Combustion Technologies, Inc.

Marc Harton (mharton@innovativecombustion.com)

Mustafa Syed (msyed@innovativecombustion.com)