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Hydrogen Generation Rate (HGR) Measurements at Savannah River National Laboratory

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Tank Closure Forum

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Savannah River NUCLEAR SOLUTIONS℠

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Outline

- **Introduction**
  - Contributions to Hydrogen Generation at SRS
  - Sources of Organic Materials at SRS
  - Overview of the SRNL Programmatic Approach to HGR
  - SRNL Approach to HGR Measurement
  - Experimental Design
    - Apparatus
    - Test Procedure
    - Calculation of HGR
- **Results**
  - Organic Thermolysis Screening Experiments
    - Chemical Degradation
    - Test Plan
    - HGR Measurement
    - Reactivity Assessment
  - Glycolate Model Development Experiments
    - Interim Model at 100 °C
    - Interim Model at Variable Temperatures
  - Prominent Organic Model Development Experiments
    - Test Plan
    - Use of Data for Model Development
    - Xiameter AFE-1010 Tests
    - Reillex HPQ Tests
    - IONAC A-641 Tests
- **Conclusions**
- **Path Forward**

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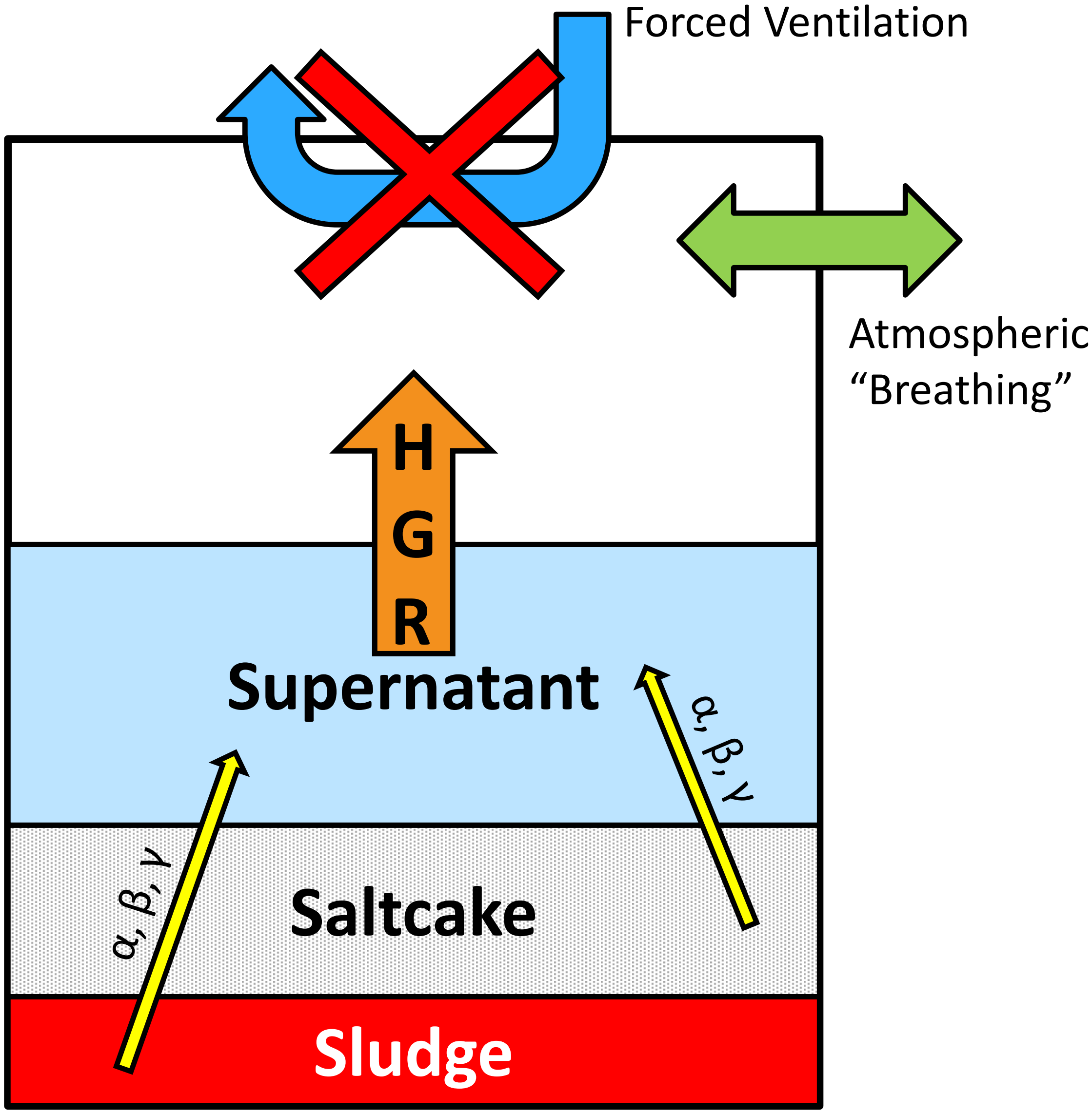
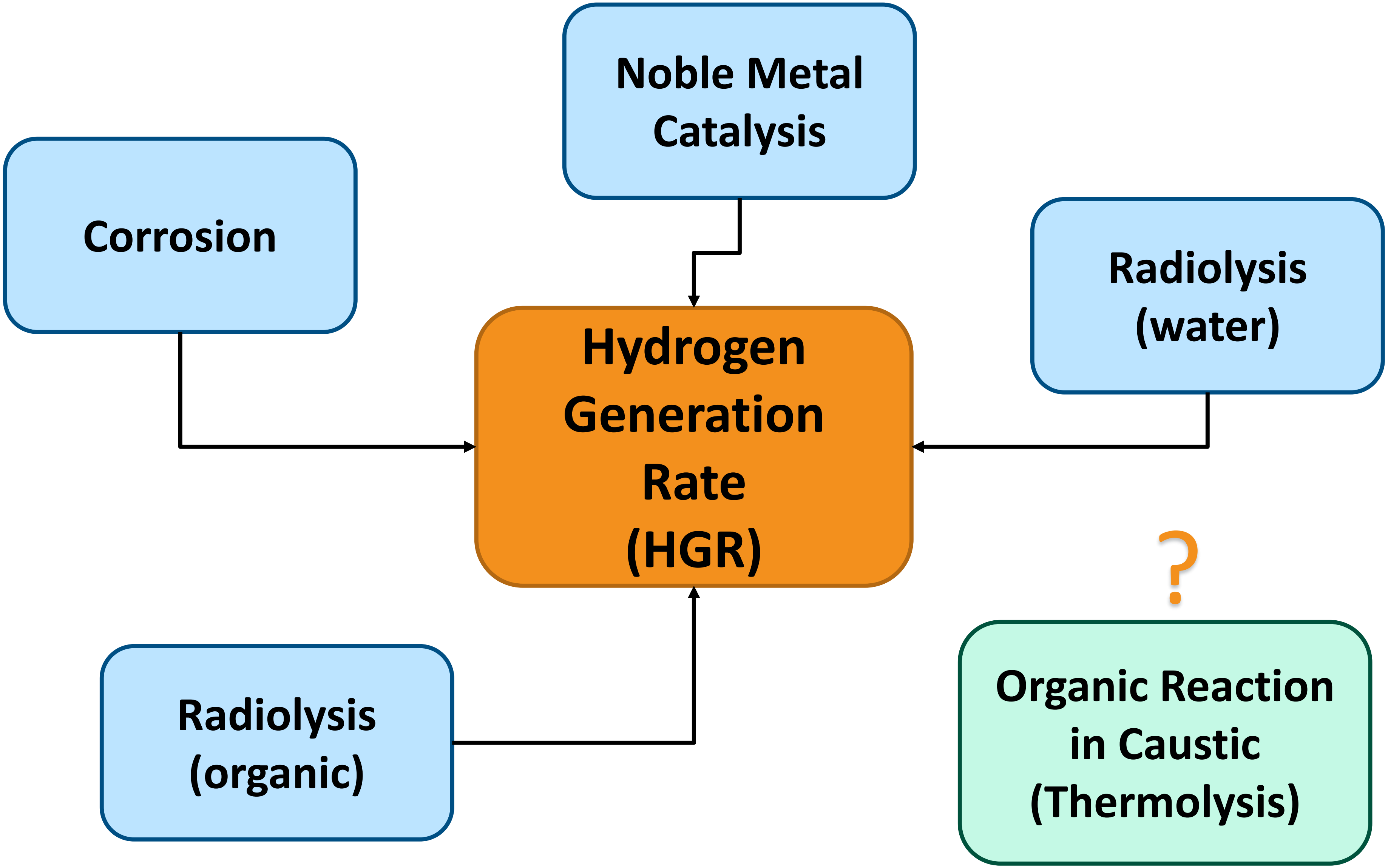
A "New" SRS Safety Challenge – Contributions to Hydrogen Generation Rates (HGRs)

- **SRS waste mostly falls into 3 categories:**
  - Sludge (insolubles)
  - Saltcake (saturated solubles)
  - Supernatant (aqueous solution)
- **Several mechanisms exist to cause H_2 generation in waste tanks**
  - e.g., radiolysis of water by radiation from waste materials
- **H_2 build-up is prevented by ventilation**
  - *Our challenge: what if we lose the ability to ventilate our tanks?*



A “New” SRS Safety Challenge – Contributions to HGRs (cont.)



Sources of Organic Compounds at SRS

H- and F-Area Canyons

Tributylphosphate (PUREX Solvent)
Resin-Related Materials

Isopar (CSSX Solvent)
Calixarenes
Solvent Modifiers

MCU/SWPF

Other Sources:
Oxalate, Lab Returns, Misc.

H- and F-Area Tank Farms

Antifoam (H-10)

HLW Evaporators

DWPF

Formate
Antifoam 747
Glycolate (Future)

Overview of the SRNL HGR Programmatic Approach

- **GOAL: To develop an expression for thermolytic production of hydrogen from organic molecules in Tank Farm waste.**

- **ASSUMPTION #1: Multiple organic molecules are capable of producing hydrogen by chemical reaction (thermolysis).**

$$HGR_{Total}^{Therm} =$$

- **ASSUMPTION #2: Each organic (e.g., compound "A") may have multiple reaction pathways, but exhibits a dominant reaction pathway in caustic tank waste.**

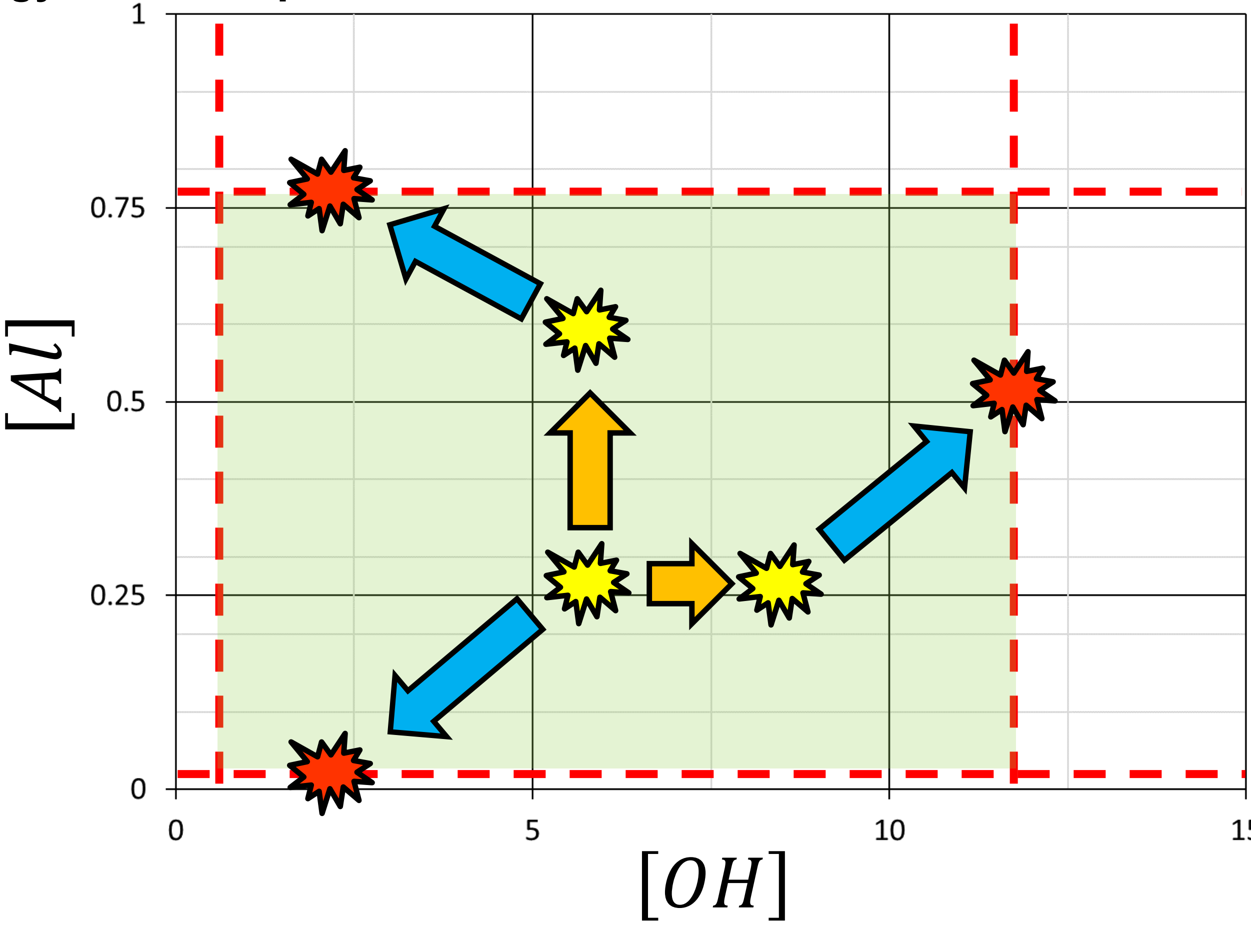
$$HGR_A = HGR_{A,1} + HGR_{A,2}$$

- **ASSUMPTION #3: The dominant reaction pathway for each molecule can be described by an Arrhenius-type kinetic expression.**

$$\boldsymbol{HGR_{A,3}} = f([A],[Na],[OH],etc.) \times e^{-\frac{E_A}{RT}}$$

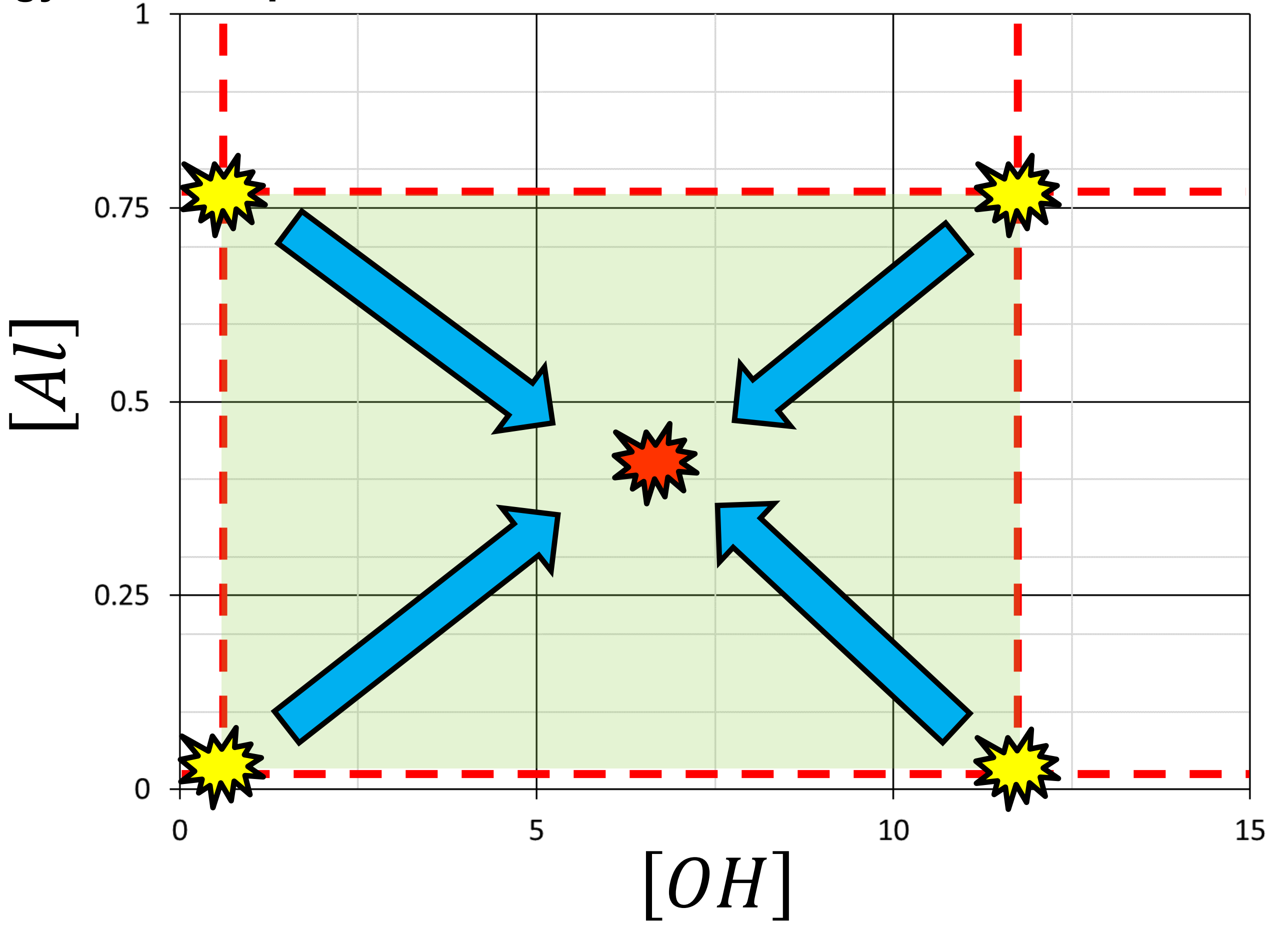
How do we evaluate the reaction expression for each organic?

- **Methodology #1 – Extrapolation from Varied Centroids**

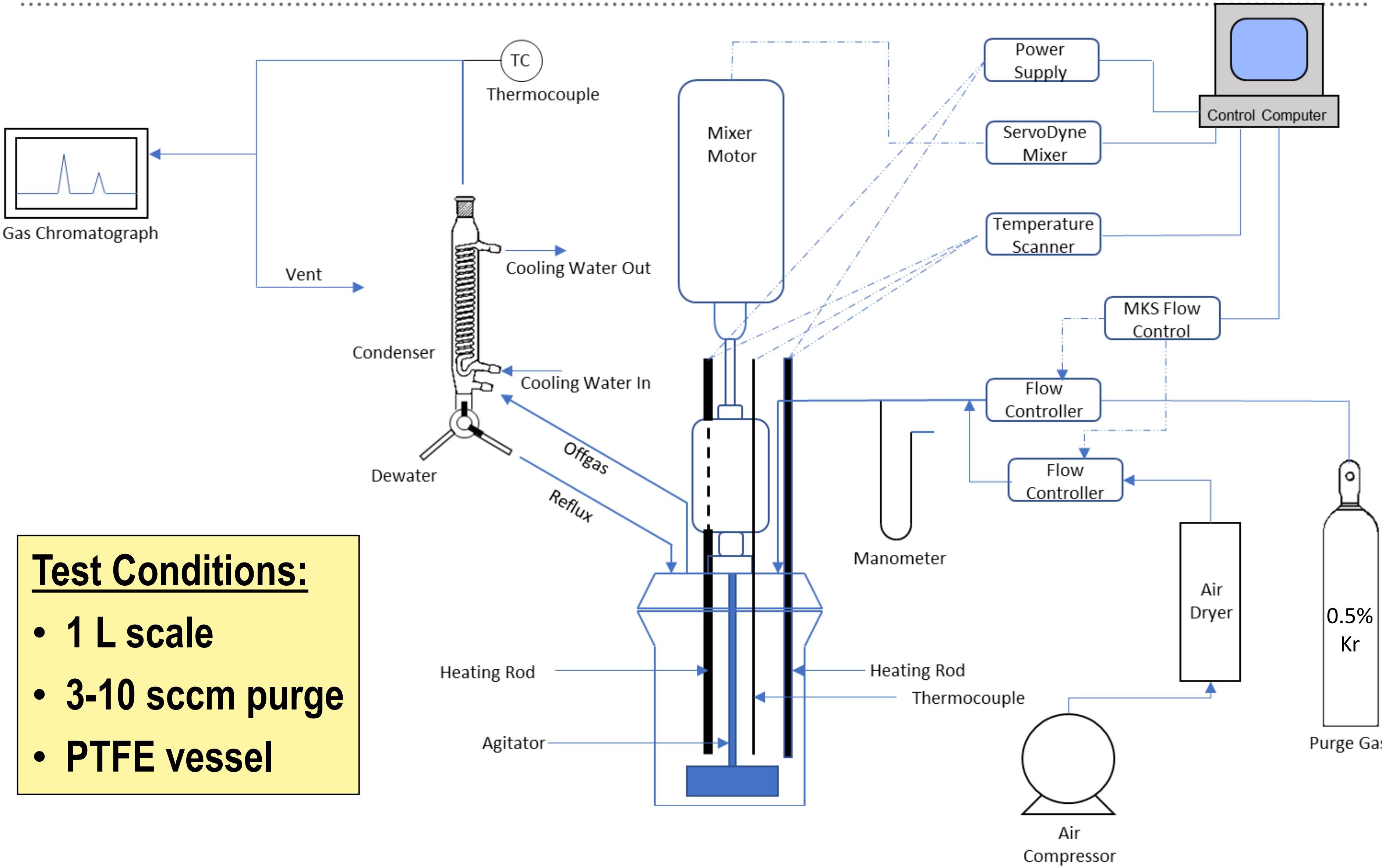


How do we evaluate the reaction expression for each organic?

- **Methodology #2 – Interpolation from Measured Extremes**



Experimental – Apparatus for Hydrogen Generation Rate Measurements

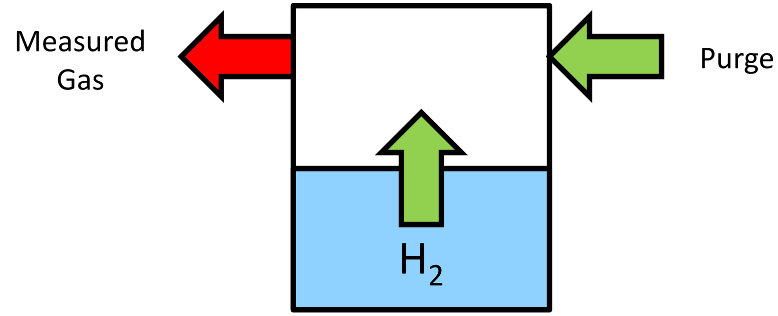


Test Conditions:

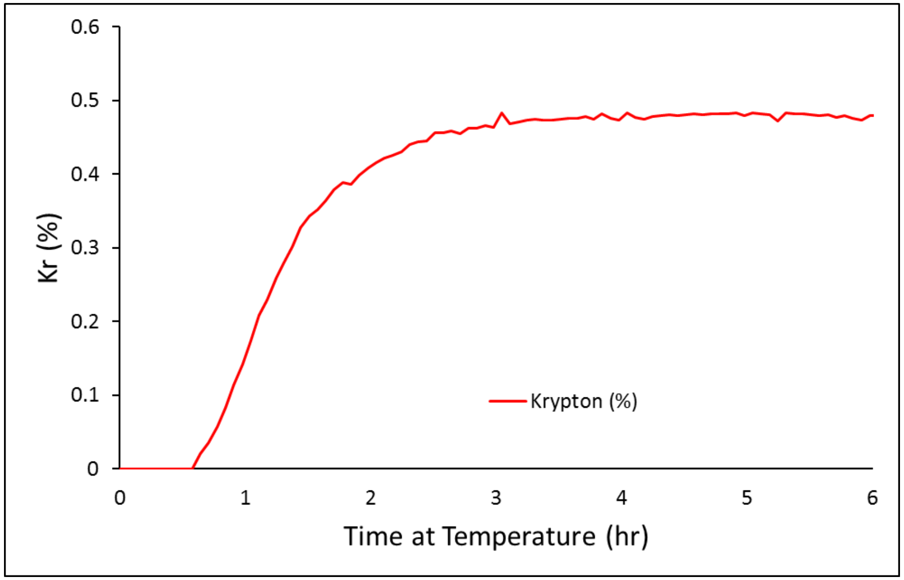
- **1 L scale**
- **3-10 sccm purge**
- **PTFE vessel**

HGR Measurement Experiments

- **Procedure**
  - 1 L of simulant added to kettle.
  - Specified amount of organic material added to kettle.
  - Vessel sealed and purged with air while mixing.
  - Vessel heated to desired temperature.
  - Once at temperature, change purge gas to 0.5% Kr.
  - Allow measurement to proceed ≥4 hours.



$$y_{H_2}(t) = y_{H_2}^{SS}\left(1 - e^{-\frac{t}{\tau_{res}}}\right)$$



How is HGR Calculated?

- **At SRS, HGR is defined as "Volumetric Rate of Hydrogen Gas generation per Unit Volume of Producing Material".**

$$HGR = \frac{\upsilon_{H_2}}{V_{material}} [=] \frac{ft^3\ H_2}{hr \cdot gal}$$

- **Production Rate of H_2 is calculated from GC measurements, response factors, and controlled purge gas flow rates.**

$$\nu_{H_2} = \frac{A_{H_2}^{GC}(a.u.) \times R_{H_2}\left(\frac{ppm\ H_2}{a.u.}\right)}{1{,}000{,}000} \times F_{purge}\left(\frac{mL}{min}\right) \times \frac{60\ min}{hr} \times \frac{ft^3}{28{,}316.8\ mL} [=] \frac{ft^3\ H_2}{hr}$$

- **Volume of material is calculated from solution mass and measured density.**

$$V_{material} = \frac{m_{sol'n}(g)}{\rho_{sol'n}\left({}^{g}/_{mL}\right)} \times \frac{gal}{3{,}785.41\ mL} [=] gal$$

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Organic Thermolysis Screening Experiments

- **Goal of Organic Screening Tests: Identify which organic species require evaluation beyond established correlations**
  - Empirical expression for organic thermolysis generated by Hu in 2004

$$HGR = a_{thm} \cdot r_f \cdot [TOC] \cdot [Al]^{0.4} \cdot L_f \cdot e^{-E_{thm}/RT} \; [=] \; \frac{mol\ H_2}{kg \cdot day}$$

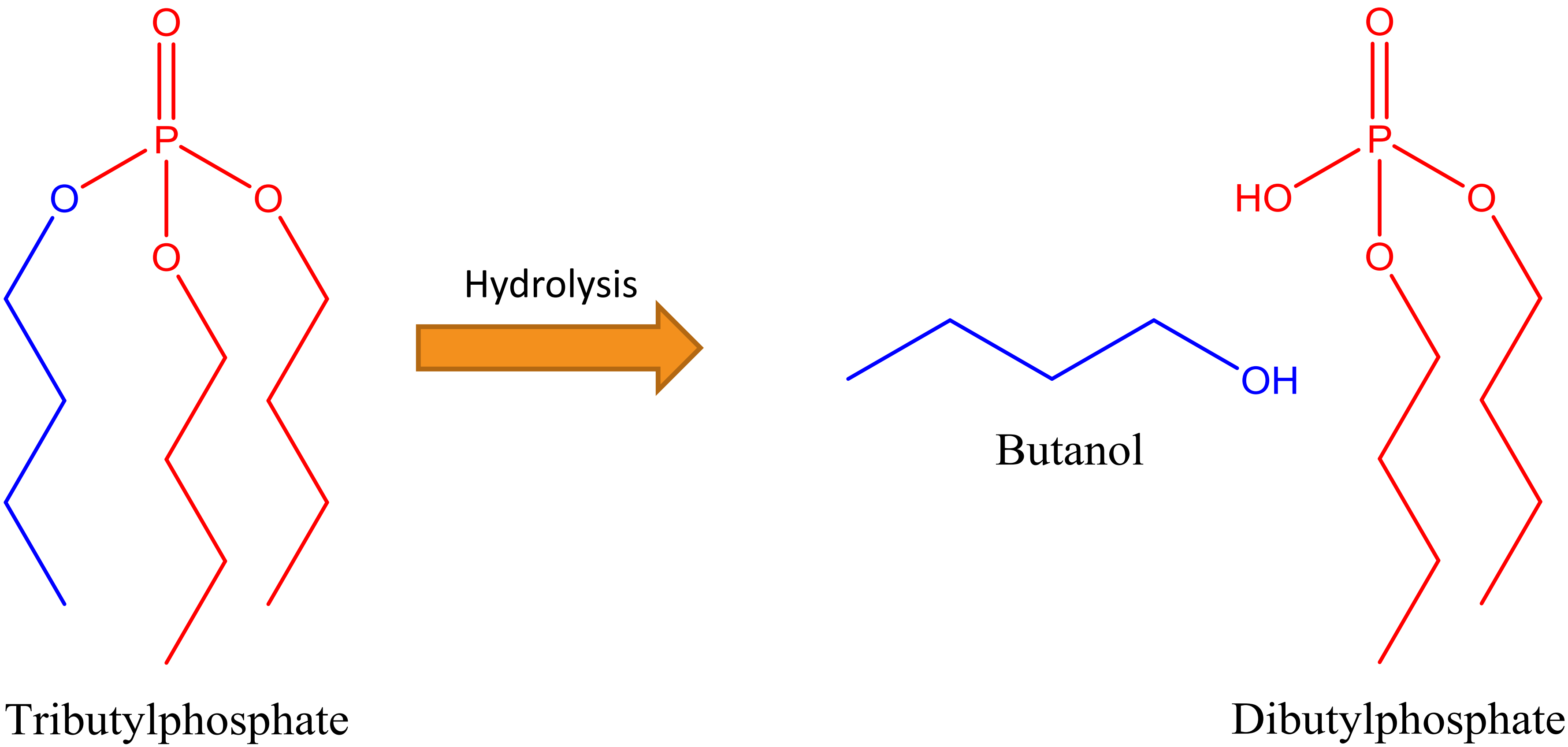
- **Strategy: Perform screening tests for each organic species of interest in a single, well-understood simulant (Tank 38)**

Species	Conc. (M)
$Al(OH)_4^-$	9.34E-02
NO_2^-	2.31E+00
NO_3^-	1.25E+00
OH^-	2.86E+00
SO_4^{2-}	6.13E-02
CO_3^{2-}	6.54E-01
Na^+	7.94E+00

- **Challenge: What organics should be examined?**
  - Solution: Use process knowledge to determine organic state

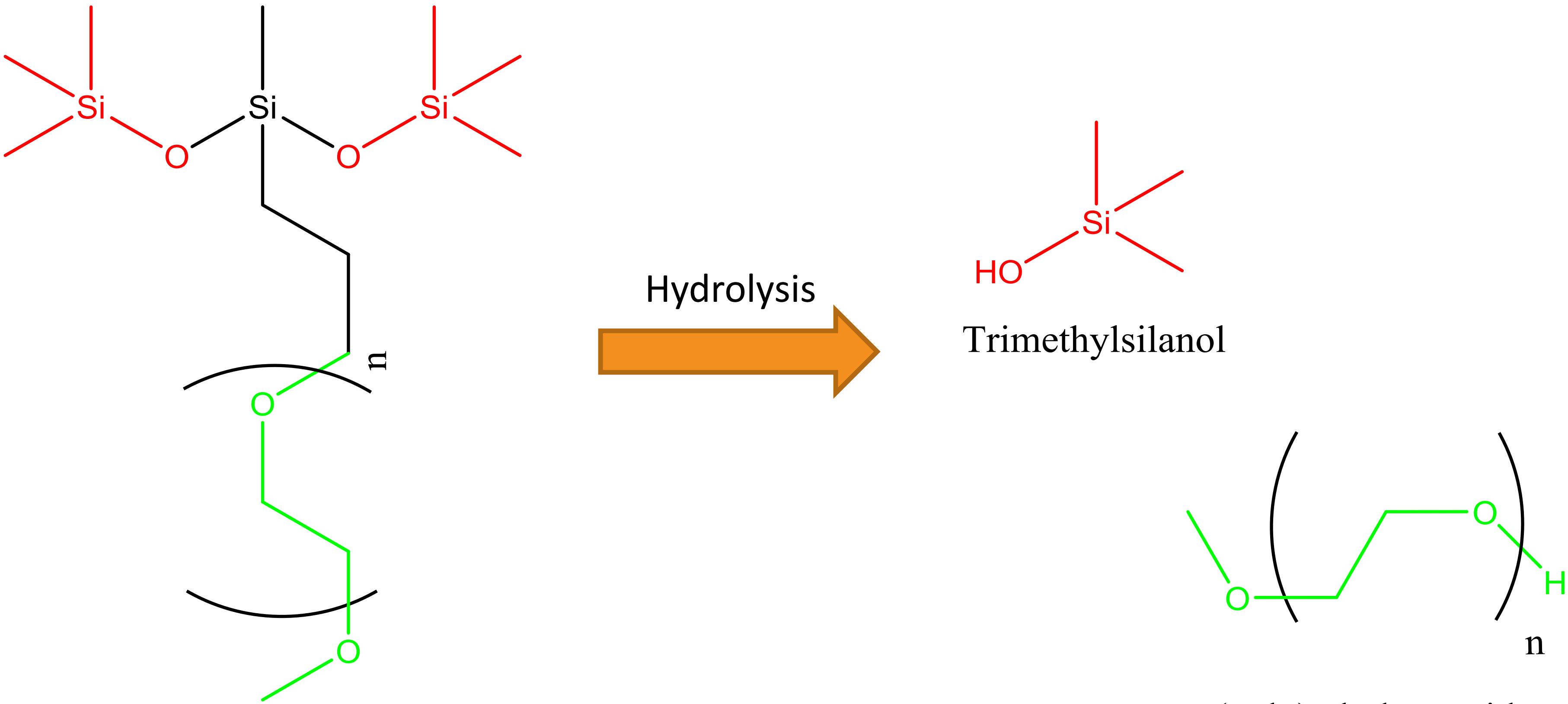
Chemical Degradation - Tributylphosphate

- **Case #1: Hydrolysis of tributylphosphate to dibutylphosphate and butanol**
  - Known to occur rapidly in radioactive waste. Suggests that all tributyl phosphate has been converted to dibutylphosphate and butanol.



Chemical Degradation – Antifoam 747

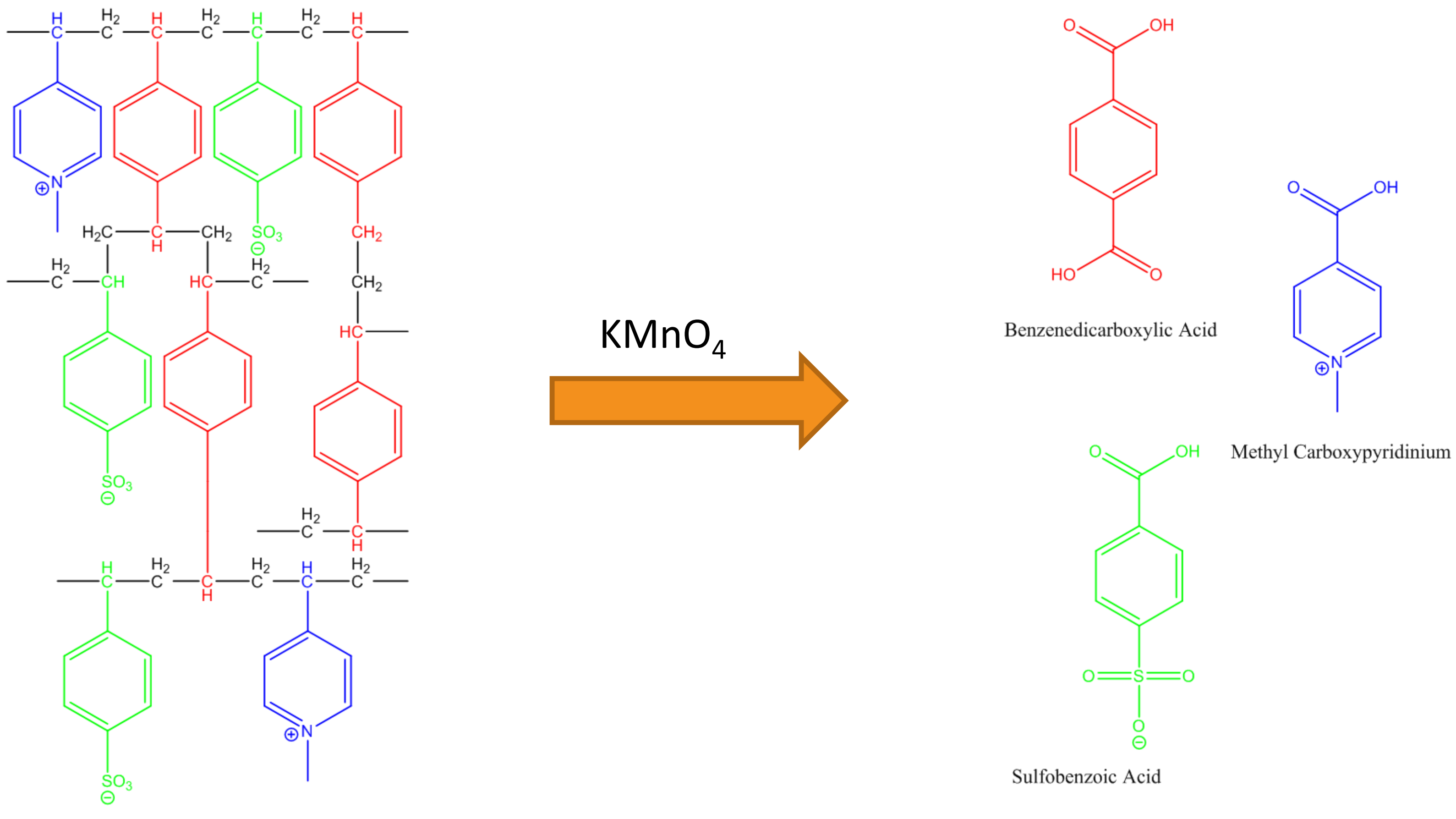
- **Case #2: Hydrolysis of Silwet L-77 (main component of Antifoam 747) to Trimethylsilanol (TMS) and polyethyleneoxide/polyethyleneglycol (PEO/PEG)**
  - Has been demonstrated historically in simulant sludge batch flowsheet experiments.



L-77

(poly)ethyleneoxide

- **Case #3: Permanganate Digestion of Ion Exchange Resins**
  - Resin typically digested before transfer to Tank Farm. Chemical literature suggests destruction of non-aromatic functional groups.



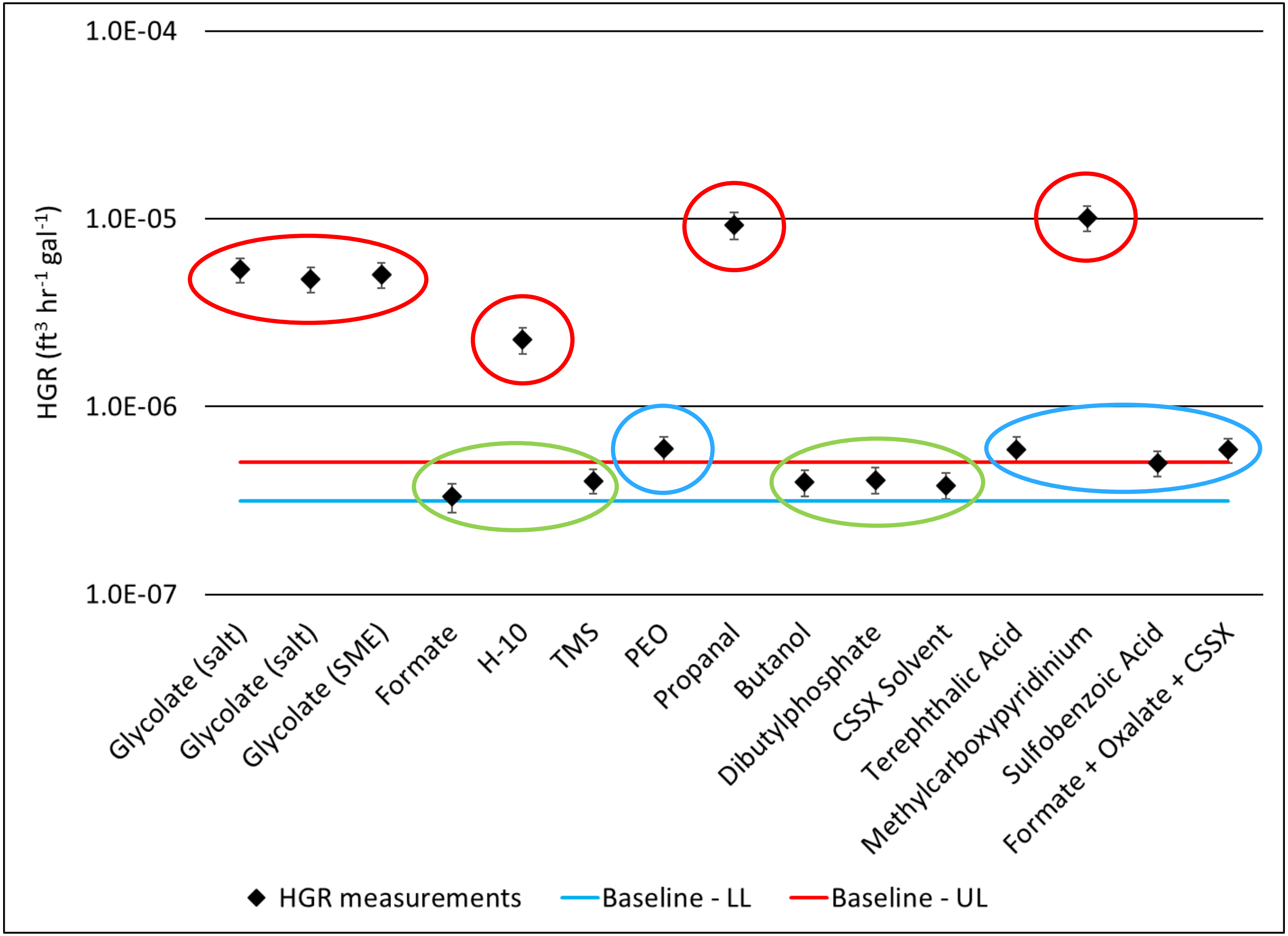
Resin Structure

Organic Screening Test Summary

ID	Organic Compound	TOC Conc. (mg C / L)	
1	None (Steel Vessel)	100*	Vessel Material Tests
2	None (PTFE Vessel)	100*	
3	Sodium Glycolate	320	Glycolate Source Tests
4	SME Glycolate	>320	
5	Sodium Formate	800	Formate Test
6	Sodium Oxalate	30	Oxalate Test
7	Dow Corning H-10	<1000	Antifoam/ADP Tests
8	Trimethylsilanol	100	
9	Polyethylene Glycol	350	
10	Propanal	60	
11	Butanol	130	Tributylphosphate Tests
12	Dibutylphosphate	370	
13	CSSX Solvent	62	CSSX Solvent Test
14	Benzenedicarboxylic Acid	290	Ion Exchange Surrogate Tests
15	Methylcarboxypyridinium	300	
16	Sulfobenzoic Acid	210	

*100 mg C/L as trace TOC impurity in simulant

Organic Screening Results – Absolute HGR



Evaluation of Reactivity

- **Reactivity can be evaluated by normalizing HGR:**

$$HGR_{obs} = HGR_{meas} - HGR_{baseline}$$

$$\lambda_{org} = \frac{HGR_{obs}}{[TOC]_{added}} [=] \frac{ft^3 H_2 \cdot L}{hr \cdot gal \cdot mol}$$

- **Reactivity can be defined by r_f in the Hu equation:**

$$HGR = a_{thm} \cdot r_f \cdot [TOC] \cdot [Al]^{0.4} \cdot L_f \cdot e^{-E_{thm}/RT} [=] \frac{mol\ H_2}{kg \cdot day}$$

$$r_f = \frac{HGR_{obs}}{a_{thm} \cdot [TOC]_{added} \cdot [Al]^{0.4} \cdot L_f \cdot e^{-E_{thm}/RT}} = \beta \frac{HGR_{obs}}{[TOC]_{added}}$$

Evaluation of Reactivity (Cont.)

Compound	TOC mg/L	λ_{org} (2σ) (× 10^{-5})	r_f (2σ)
Glycolate (salt)	330	13.1 - 20.9	1.43 - 2.28
Glycolate (SME)	410	11.4 - 16.0	1.24 - 1.74
Formate	820	0 - 0.05	0 - 0.01
CSSX Solvent	60	0 - 1.62	0 - 0.18
Formate + CSSX + Oxalate	910	0.06 – 0.41	0.01 - 0.04
DBP	380	0 - 0.37	0 - 0.04
Butanol	130	0 - 0.92	0 - 0.10

Compound	TOC mg/L	λ_{org} (2σ) (× 10^{-5})	r_f (2σ)
TMS	100	0-1.24	0 – 0.13
PEO	360	0.17 - 1.05	0.02 – 0.11
Propanal	60	140 - 198	15.2 – 21.6
Xiameter	< 1030	> 1.74	> 0.19
Sulfobenzoic Acid	220	0 - 1.21	0 – 0.13
Terephthalic Acid	370	0.15 - 1.03	0.02 – 0.11
Methylcarboxy-Pyridinium	190	52.3 - 72.3	5.70 – 7.88

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Glycolate Model Development Experiments

- **Goal of Glycolate Model Development Tests: Determine which conditions most impact thermolytic HGR from glycolate**
  - Glycolate is currently not present in SRS Tank Waste, but will be added under the upcoming Alternate Reductant Flowsheet

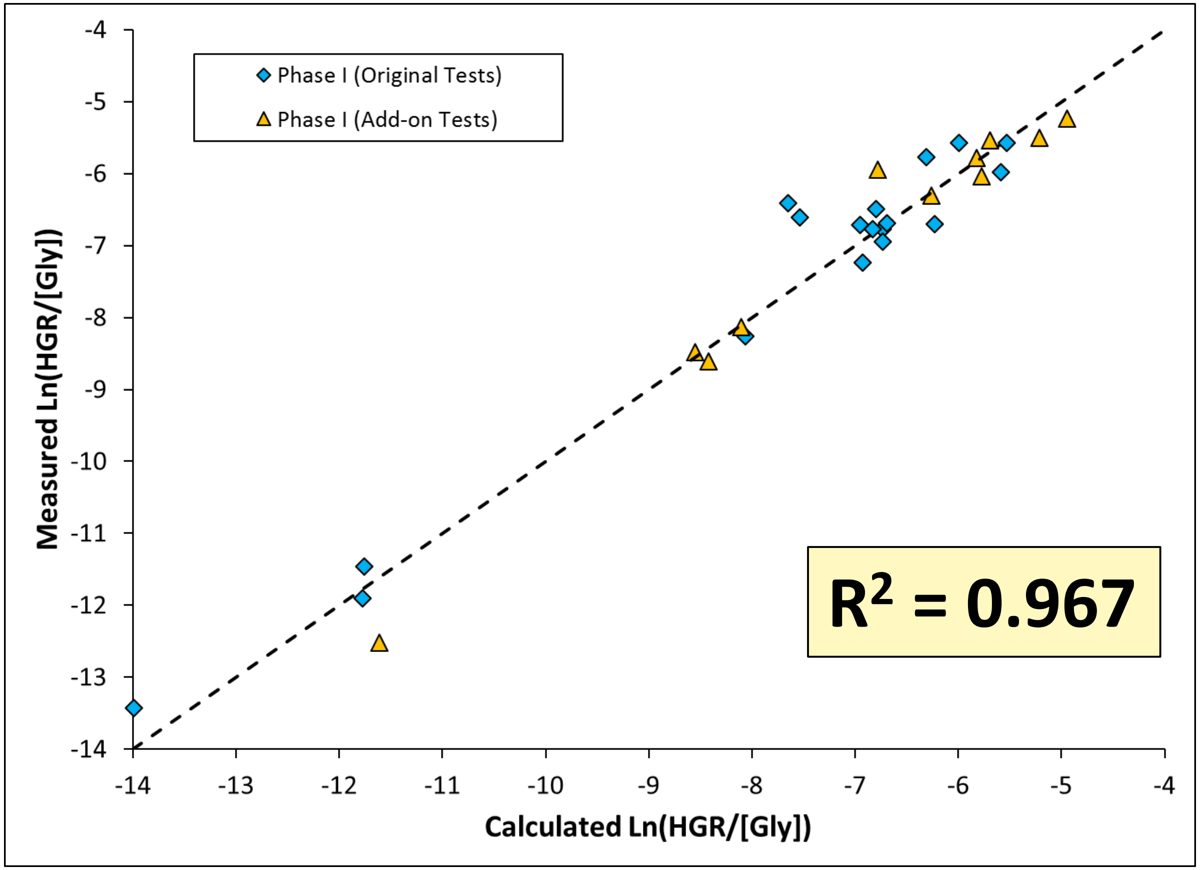
- **Strategy: Perform measurements of thermolytic HGR from glycolate in conditions with sufficient salt concentration variability to:**
  - 1) determine impact of salt concentration on reaction rates
  - 2) confidently describe thermolytic HGR from glycolate at all possible conditions

- **Solution: Generate a statistically-driven experimental matrix of tests**
  - D-Optimal criterion used to determine the most "ideal" test conditions to examine

Interim Glycolate Model at 100 °C

Using all available simulant data (>30 tests) to generate an interim model for Glycolate HGR at 100 °C:

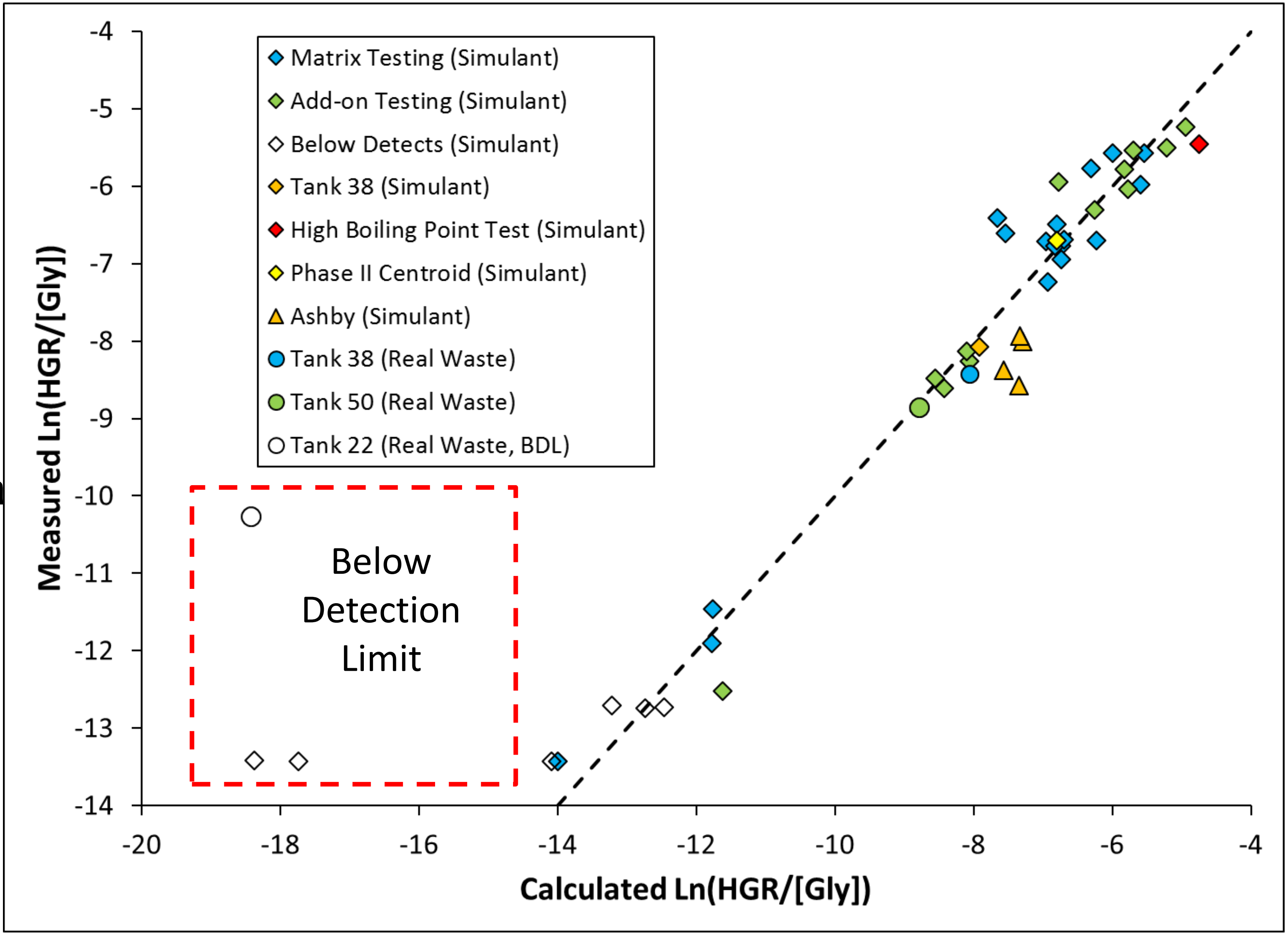
$$HGR\left(\frac{ft^3}{hr \cdot gal}\right) = 8.502 \times 10^{-7}\,\frac{[Al]^{0.239}[OH]^{1.076}[Na]^{2.756}[Gly]}{[NO_2]^{0.430}}$$



Evaluation of Interim Glycolate Model Against All Measurements

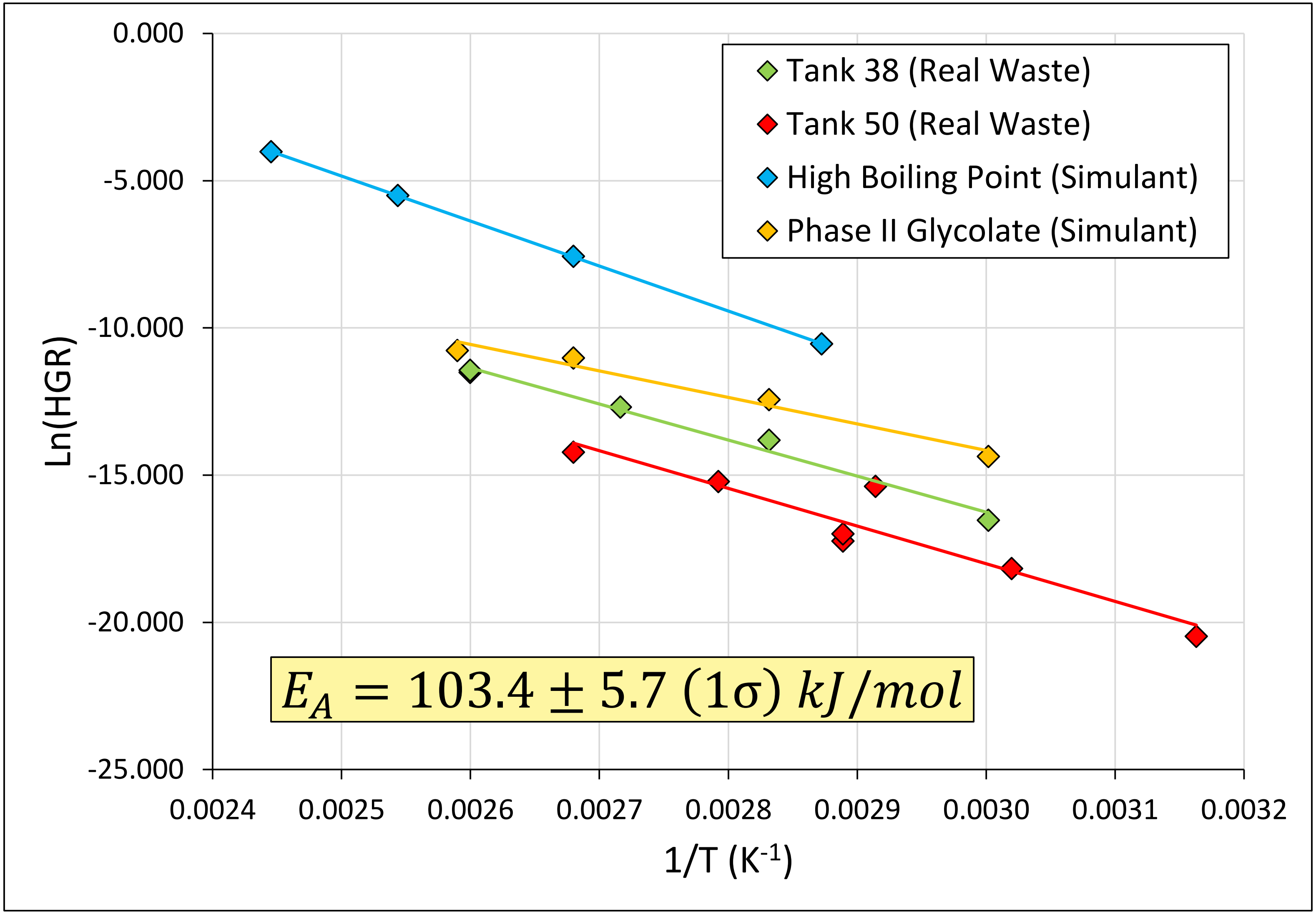
Model exhibits good agreement with all 100 °C data tested to date:

What a



Arrhenius Plots of Glycolate HGR Measurements

Four sets of data across multiple temperatures.



Previous Results with Glycolate

Data from all phases of testing used to generate a temperature-dependent interim model for glycolate thermolysis.

$$HGR_{GLY}\left(\frac{ft^3}{hr \cdot gal}\right) = 8.502 \times 10^{-7}\,\frac{[Al]^{0.239}[OH]^{1.076}[Na]^{2.756}[Gly]}{[NO_2]^{0.430}}$$

$$HGR_i = k_i \times f_i([x]) \times e^{-E_i/RT}$$

$$8.502 \times 10^{-7} = k_i \times e^{-103{,}400/373.15R}$$

$$k_i = 1.268 \times 10^{8}$$

$$HGR_{GLY} = 1.268 \times 10^{8}\,\frac{[Al]^{0.239}[OH]^{1.076}[Na]^{2.756}[C_{Gly}]}{[NO_2]^{0.430}}\,e^{-103{,}400/RT}$$

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Prominent Organic Model Development Experiments

- **Goal of Prominent Organic Model Development Tests: Determine which conditions most impact the thermolytic HGR from other Tank Farm Organics**

- **Strategy: Perform measurements of thermolytic HGR from prominent Tank Farm Organics in Tank 38 Variant conditions**
  - Equivalent to traditional reaction kinetics experiments
  - Selection of Tank 38 allows for analysis of HGR at evaporator-like conditions

- **Modification: When possible, real organic sources were used (rather than chemical substitutes)**
  - IONAC A-641 and Reillex HPQ Ion Exchange Resins were digested as employed as a product slurry for all of testing

Testing Conditions – Experiment Design

- **Model development testing performed in Tank 38 simulant variants at 85, 100, and 110 °C.**
  - Several tests performed for each organic species
  - Temperature varied between 85 °C and 110 °C (boiling)
  - Salt components varied independently to determine the impact of each species on thermolysis

Test Condition	$Al(OH)_4^-$ (M)	NO_2^- (M)	NO_3^- (M)	OH^- (M)	SO_4^{2-} (M)	CO_3^{2-} (M)	Temp (°C)
Tank 38	9.34E-02	2.31E+00	1.25E+00	2.86E+00	6.13E-02	6.54E-01	100
Higher Al	2.80E-01	2.31E+00	1.25E+00	2.86E+00	6.13E-02	6.54E-01	100
Lower NO_2	9.34E-02	1.34E+00	1.25E+00	2.86E+00	6.13E-02	6.54E-01	100
Lower NO_3	9.34E-02	2.31E+00	2.80E-01	2.86E+00	6.13E-02	6.54E-01	100
Lower OH	9.34E-02	2.31E+00	1.25E+00	1.89E+00	6.13E-02	6.54E-01	100
Lower SO_4	9.34E-02	2.31E+00	1.25E+00	2.86E+00	2.04E-02	6.54E-01	100
Lower CO_3	9.34E-02	2.31E+00	1.25E+00	2.86E+00	6.13E-02	2.18E-01	100
Lower Temp	9.34E-02	2.31E+00	1.25E+00	2.86E+00	6.13E-02	6.54E-01	85
Higher Temp	9.34E-02	2.31E+00	1.25E+00	2.86E+00	6.13E-02	6.54E-01	110

Use of HGR Measurement Data to Generate Interim Models for Organic Thermolysis

- **For empirical expression, assume simple rate behavior:**

$$HGR_i = k_i[Al]^{\alpha_i}[NO_2]^{\beta_i}[NO_3]^{\gamma_i}[OH]^{\delta_i}[SO_4]^{\varepsilon_i}[CO_3]^{\theta_i}[C_i]e^{-E/_{RT}}$$

- **This expression can be linearized in log space:**

$$\ln\frac{HGR_i}{[C_i]} = \ln k_i + \sum_{j=\alpha}^{\theta} j_i \ln[j] - \frac{E}{RT}$$

Use of HGR Measurement Data to Generate Interim Models for Organic Thermolysis

- For a given organic species, n different experiments yields n equations to calculate the "best" values of k_i, E, and α through θ

$$\vec{y} = \underline{A} \cdot \vec{x}$$

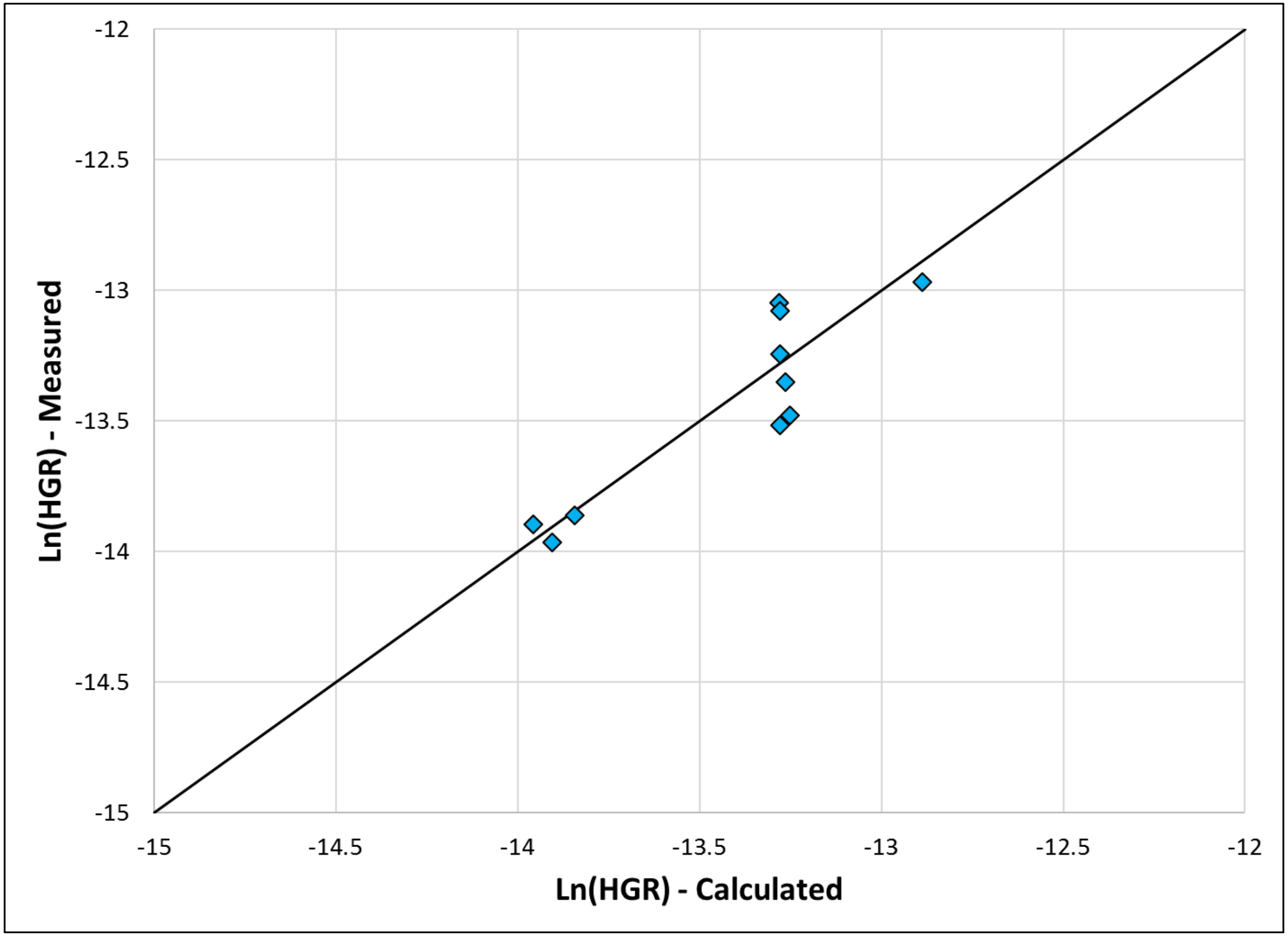
$$\vec{y} = \begin{bmatrix} \ln \frac{HGR_1}{[C_1]} \\ \vdots \\ \ln \frac{HGR_n}{[C_n]} \end{bmatrix} = \underbrace{\begin{bmatrix} 1 & \ln[Al]_1 & \ln[NO_2]_1 & \ln[NO_3]_1 & \ln[OH]_1 & \ln[SO_4]_1 & \ln[CO_3]_1 & -{}^{1}/_{T_1} \\ \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots & \vdots \\ 1 & \ln[Al]_n & \ln[NO_2]_n & \ln[NO_3]_n & \ln[OH]_n & \ln[SO_4]_n & \ln[CO_3]_n & -{}^{1}/_{T_n} \end{bmatrix}}_{\underline{A}} \times \underbrace{\begin{bmatrix} k \\ \alpha \\ \beta \\ \gamma \\ \delta \\ \varepsilon \\ \theta \\ {}^{E}/_{R} \end{bmatrix}}_{\vec{x}}$$

- To solve for x, use linear algebra

$$\vec{x} = (\underline{A}^T \times \underline{A})^{-1} \times (\underline{A}^T \times \vec{y})$$

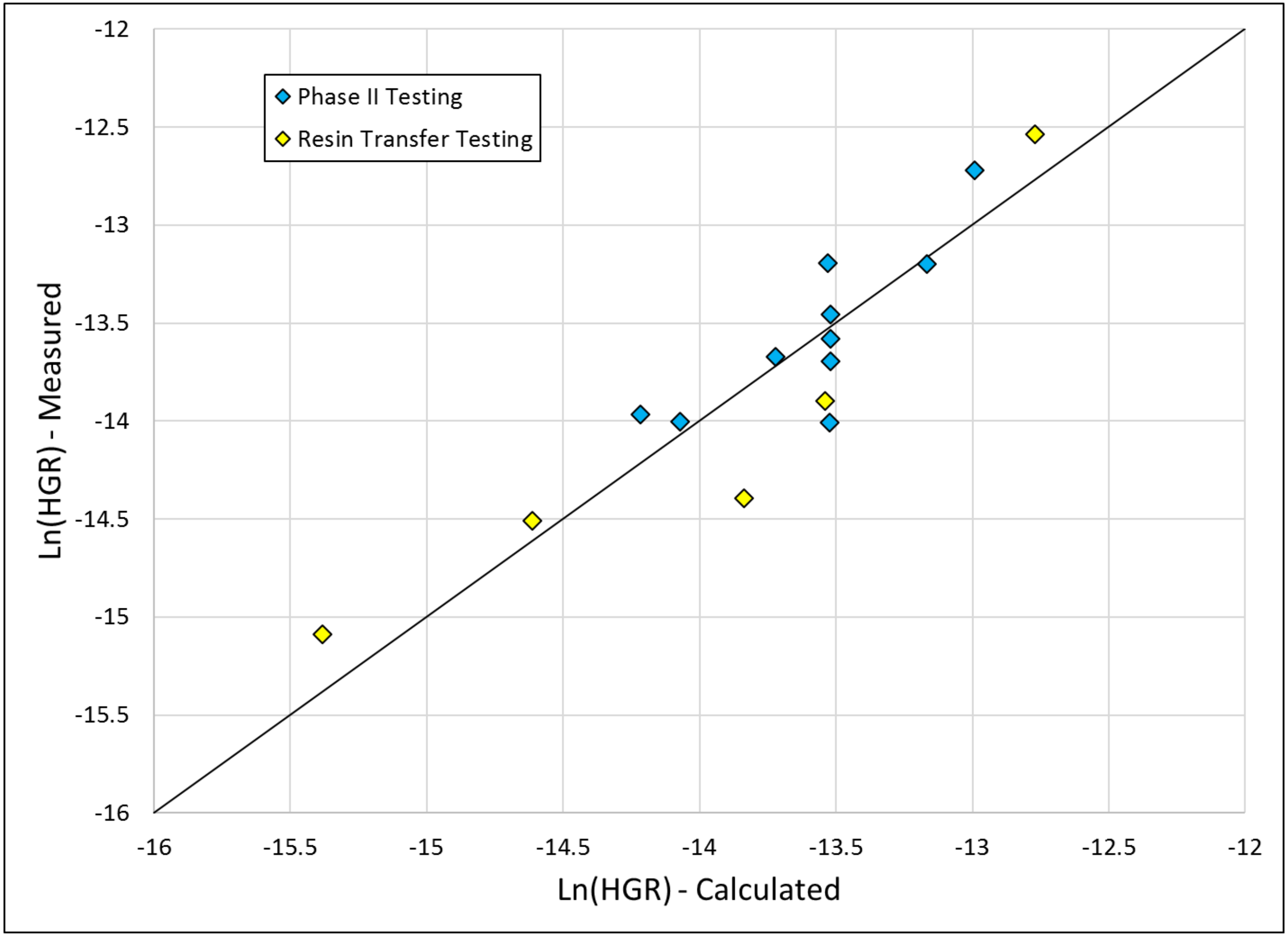
Results from Xiameter AFE-1010 Antifoam Testing – Interim Model

$$HGR_{XIA}^{Thm} = 1.085 \times 10^{3} \left[OH\right]^{1.389} \left[C_{XIA}\right] e^{-45{,}400/RT}$$



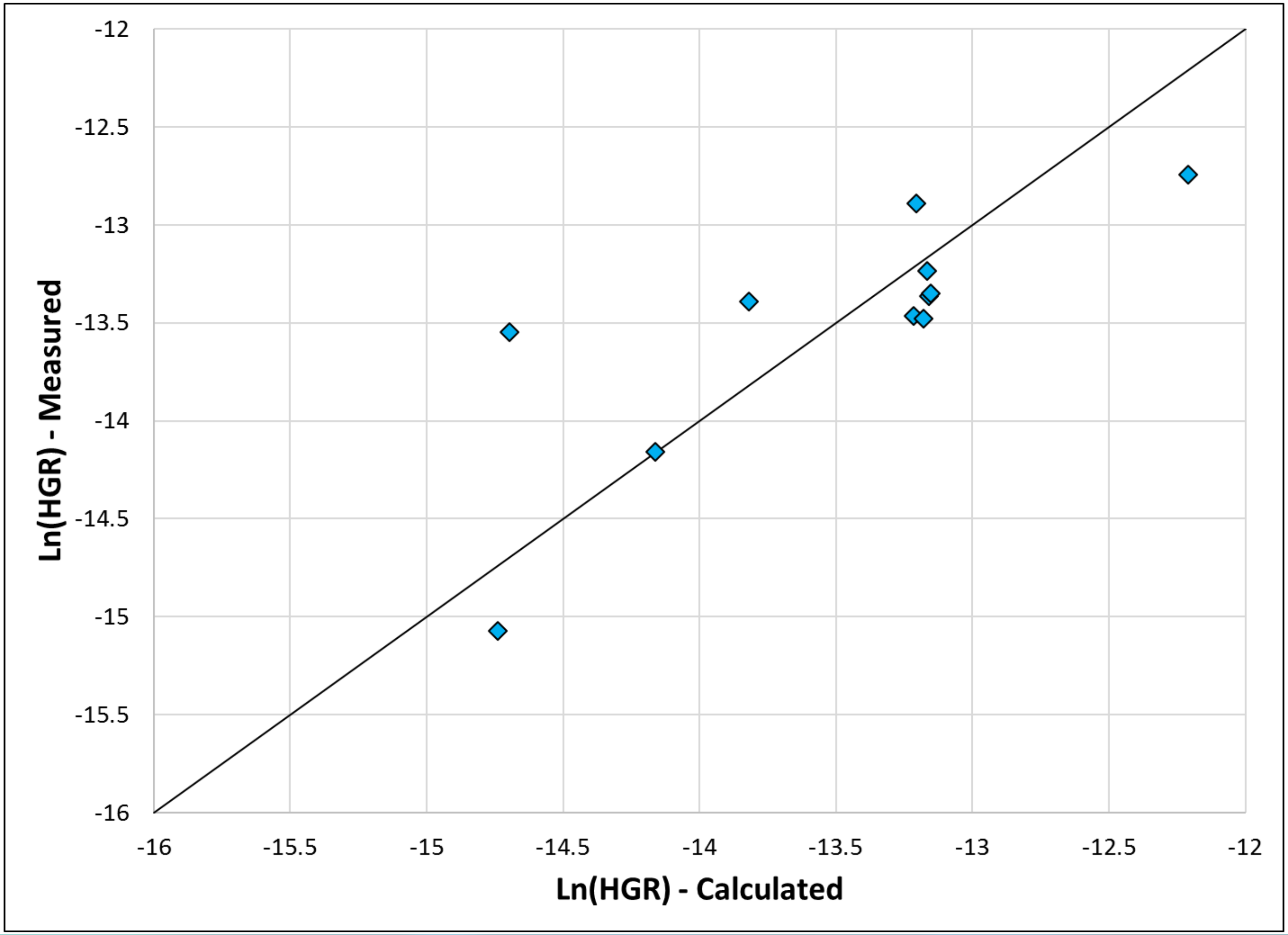
Results from Reillex HPQ Resin Digestion Product Testing – Interim Model

$$HGR_{RLX}^{Thm} = 3.181 \times 10^{3} \left[Al \right]^{0.491} \left[OH \right]^{0.513} \left[C_{RLX} \right] e^{-42{,}200 / RT}$$



Results from IONAC A-641 Resin Digestion Product Testing – Interim Model

$$HGR_{IAC}^{Thm} = 8.453\times 10^{10}\left[OH\right]^{2.430}\left[C_{IAC}\right]e^{-116,700/RT}$$



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Conclusions

- **Several organic compounds have been shown to be inert in caustic environments (e.g., formate, oxalate)**
- **Most of the organic compounds tested are sufficiently unreactive to be safely described or bounded by existing thermolytic correlations (e.g., dibutylphosphate, Isopar)**
- **Some compounds have been shown to exhibit high HGRs in tank farm conditions (e.g., propanal, antifoam agents)**
- **Testing with radioactive waste has demonstrated measurable HGRs with apparent dependence on temperature (consistent with thermolytic H_2 production)**

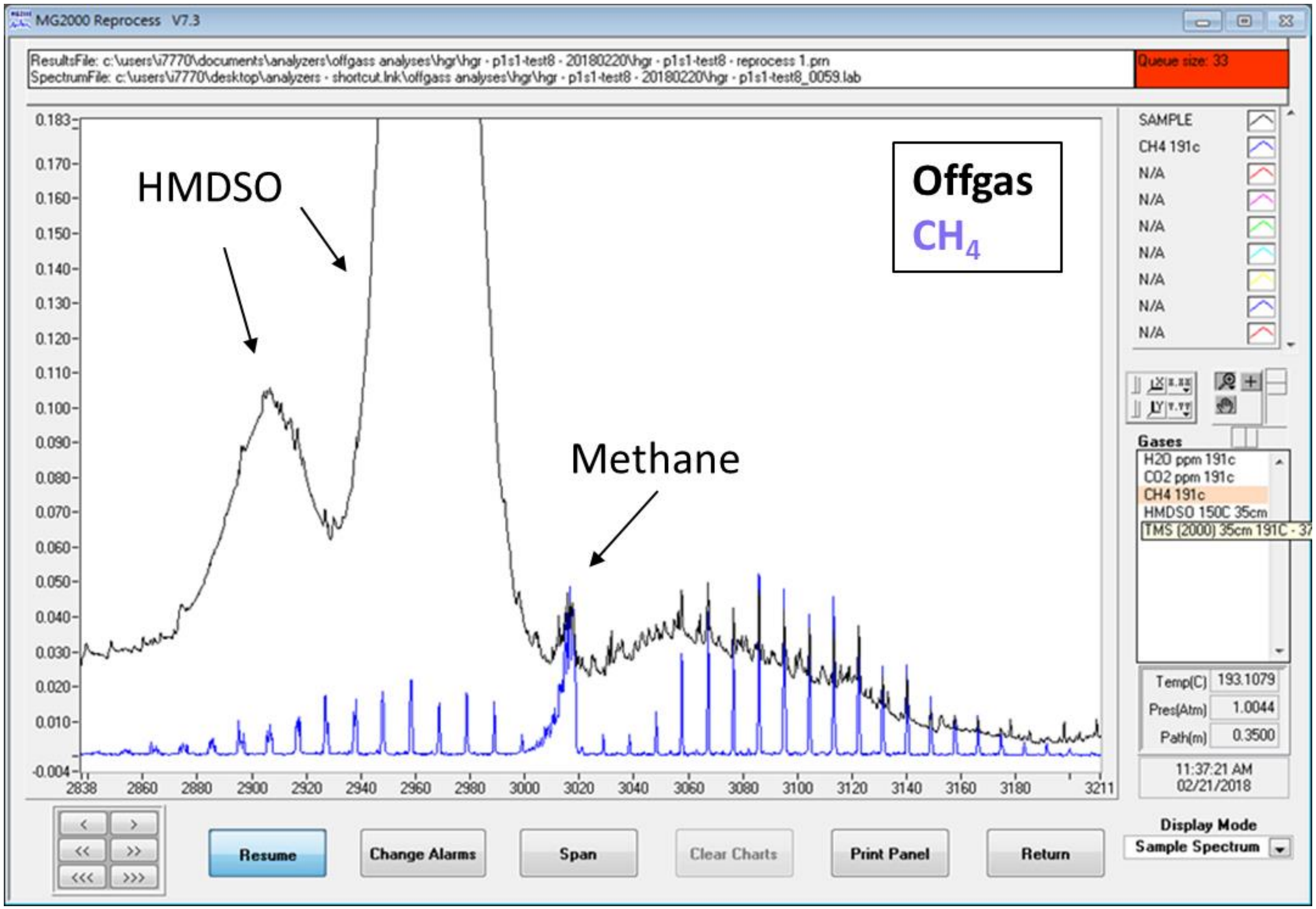
Conclusions (Cont.)

- **HGR expressions have been derived for the most reactive compounds:**
  - Xiameter AFE-1010 exhibits an apparent dependence on $[OH^-]$ and temperature
  - IONAC A-641 resin digestion materials exhibit an apparent dependence on $[OH^-]$ and temperature
  - Reillex HPQ resin digestion materials exhibit an apparent dependence on $[OH^-]$ and temperature, with a possible influence from [Al]
  - Glycolate (not yet incorporated into SRS tank waste) exhibits an apparent dependence on [Al], $[NO_2^-]$, $[OH^-]$, [Na], and temperature

- **SRS organic thermolytic reactions appear to be dependent on caustic media. The dependence of $[OH^-]$ has not been previously quantified or correlated.**

Path Forward – Generation of Other Flammable Gases

- **Select organometallic species (e.g., trimethylsilanol) yield measurable quantities of methane via FT-IR and GC. These generation rates and their impact on vapor-phase flammability should be determined.**

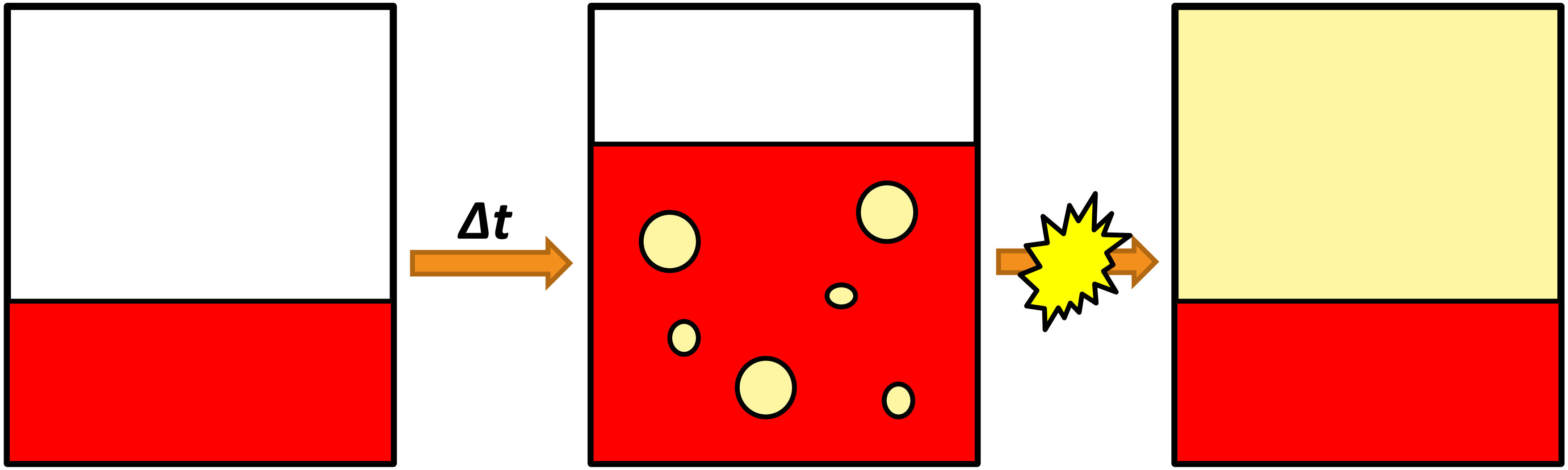


Path Forward – Prediction of Trapped Gas Composition

- **Solid phases provide obstacles to release of generated gas, creating pockets of produced vapors**

- **Sudden, unexpected releases lead to temporarily high headspace concentrations of generated gases (e.g., H_2)**

- **HGR rate data can be leveraged to better predict the generation rates and compositions of trapped gas bubbles and pockets.**



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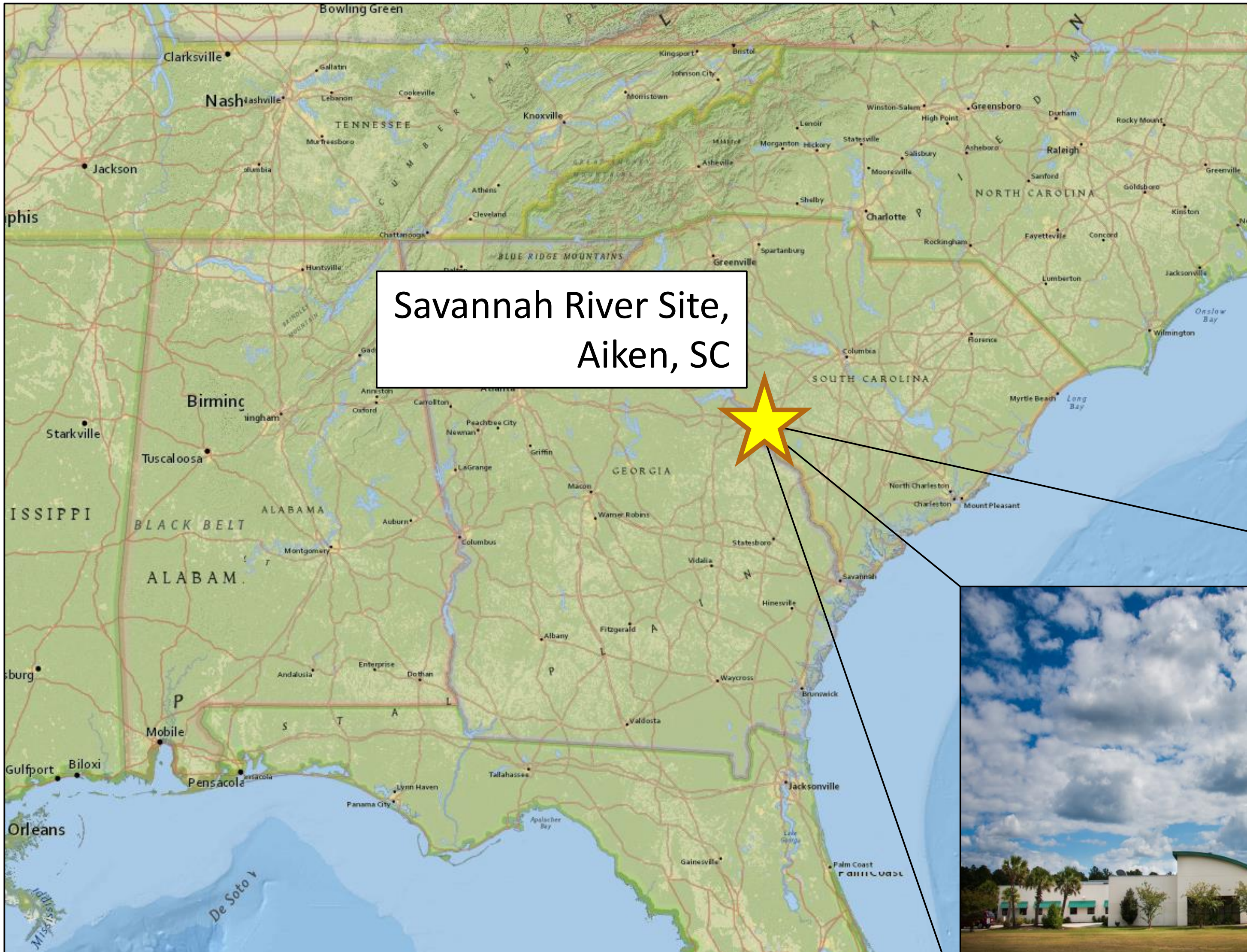
Discussion

Where Are We Located?



Home of the Savannah River National Laboratory (SRNL)

The Savannah River Site (SRS) Mission and Vision

- **Our Mission:**

*…to **safely** and efficiently operate SRS to **protect** the public **health** and the **environment** while supporting the nation's nuclear deterrent and the transformation of the Site for future use.*

- **Our Vision**

*…a long-term national asset in the areas of **environmental stewardship**, innovative technology, national security, and energy independence which acts with an inspired workforce and mature, efficient management processes, while **sustaining public confidence** in our people and*

- **Our Values**
  - **Safe** and Effective Operations
  - Efficient Operations
  - Good Relations with Stakeholders
  - Integrity