

# ***THOR<sup>®</sup> Steam Reforming Pilot Plant Demonstration Program Experience***



# Agenda

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- ***History of Steam Reforming Pilot Plant Experience***
- ***Pilot Plant Demonstration Program Implementation***
  - ***Project Planning***
  - ***Technical Planning***
  - ***Pilot Plant Preparation and Operations***
  - ***Post Test Activities***
- ***Simulants***
- ***Lessons Learned***

# ***History of Pilot Plant Experience***

## ***Steam Reforming***

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### ***Pilot Plant Demonstration Programs:***

- ***Studsvik Processing Facility***
  - ***Waste Feeds: Ion exchange resins, oils, solvents, nitrates – 5 test series***
  - ***Product: Carbonate***
- ***Bechtel-Studsvik: WTP***
  - ***Waste Feeds: Hanford LAW – 4 test series***
  - ***Product: Carbonate and alkali aluminosilicate***
- ***CWI: Idaho Cleanup Project, Integrated Waste Treatment Unit***
  - ***Waste Feed: SBW simulant – 6 test series***
  - ***Product: Carbonate and alkali aluminosilicate***
- ***WSRC: Savannah River Site, Tank 48H Treatment***
  - ***Waste Feed: Tank 48H simulant – 2 test series***
  - ***Product: Carbonate***

# *History of Pilot Plant Experience*

## *Steam Reforming*

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1. **Hazen Studsvik – April 1997**
  1. 6” Fluidized Bed, Hanford 5.2 NaNO<sub>3</sub> surrogate, carbonate product
  2. 15” Fluidized Bed, ion exchange resins, oils, solvents
2. **LMITCO INEEL – 2002 (3” Fluidized Bed, SBW surrogate)**
3. **Hazen Bechtel/Studsvik – Dec. 2001 (6” Fluidized Bed, LAW surrogate, carbonate and NAS products)**
4. **SAIC STAR – Jan. 2003 (6” Fluidized Bed, SBW surrogate, carbonate)**
5. **SAIC STAR – Aug. 2003 (6” Fluidized Bed, SRS Tank 48H surrogate, carbonate)**
6. **SAIC STAR – Nov. 2003 (6” Fluidized Bed, SBW surrogate, carbonate)**
7. **SAIC STAR – Aug. 2004 (6” Fluidized Bed, LAW surrogate, NAS product)**
8. **SAIC STAR – July and Sept. 2004 (6” Fluidized Bed, SBW surrogate, NAS product)**
9. **Hazen Engineering Scale for IWTU SBW Carbonate – Nov. 2005 through May 2006 (15” Fluidized Bed, SBW surrogate, carbonate product)**
10. **Hazen Engineering Scale for SRS Tank 48 Carbonate – Sept. 2006 through Nov. 2006 (15” Fluidized Bed, Tank 48 complex surrogate, carbonate product)**
11. **Hazen Engineering Scale for IWTU SBW Mineralized – Oct. through Dec. 2006 (15” Fluidized Bed, SBW surrogate, NAS mineral product)**

# History of Pilot Plant Experience

## Steam Reforming

### **Studsvik Processing Facility**

- **Flowsheet: THOR<sup>®</sup> steam reforming, dual stage**
- **Duration: ~1.5 month, 1997**
- **Scale-up: 15" to 45" (9x)**
- **Location: Hazen Research**



# History of Pilot Plant Experience

## Steam Reforming

### CWI - IWTU

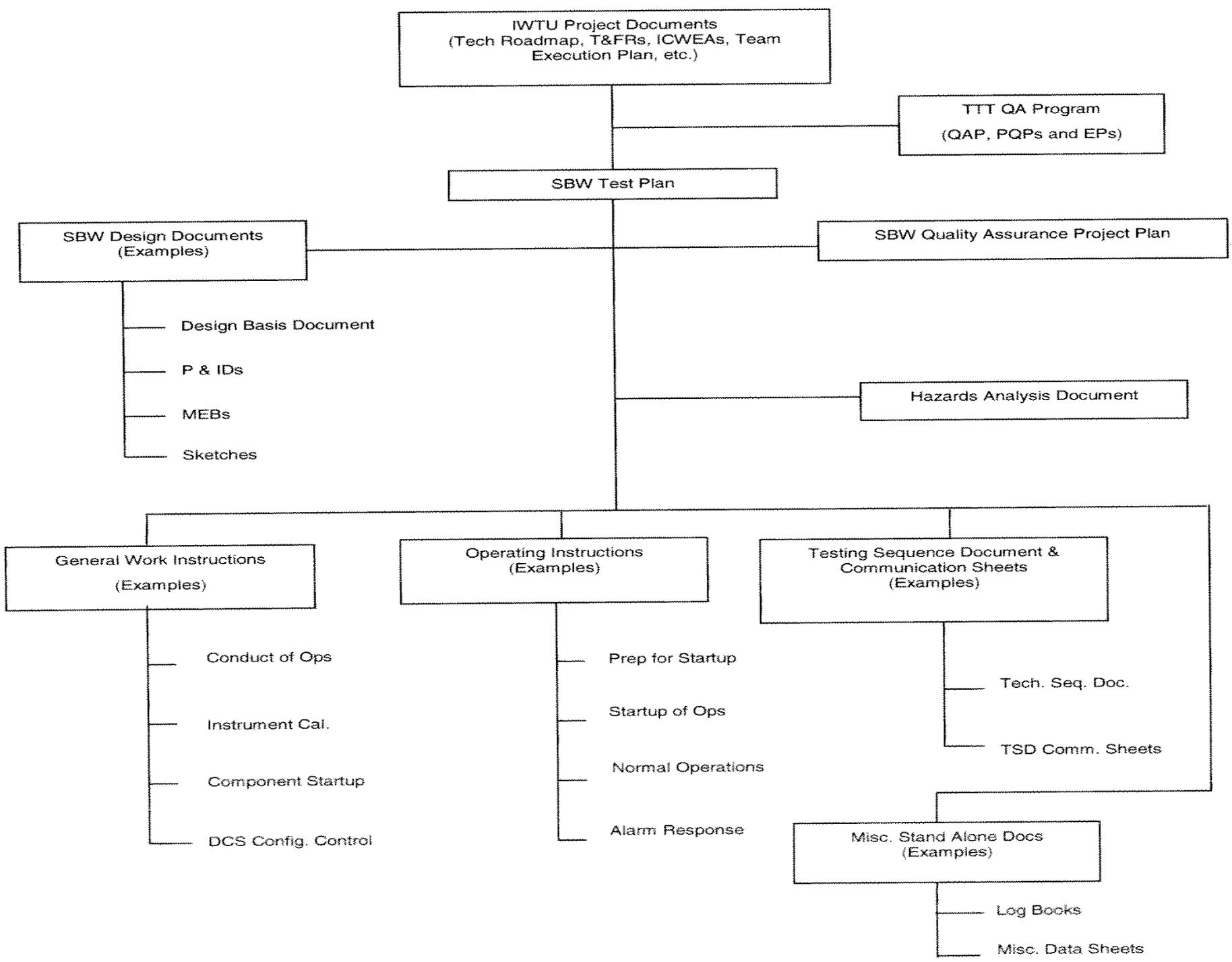
- **Flowsheet:** THOR<sup>®</sup> steam reforming, dual stage
- **Duration:** 4 Series, ~4.0 months, 2002 to 2006
- **Scale-up:** 3" to 6" to 15" to 48" (10x)
- **Location:** STAR Center and Hazen Research



# ***Demonstration Program Implementation Project Planning - IWTU***

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- ***Uses TTT's NQA-1 QA Program***
- ***Core testing documents are:***
  - ***Test Plan (roles, responsibilities, baseline process testing matrices, sample and analytical plan)***
  - ***Quality Assurance Project Plan***
- ***Test plan is supported by:***
  - ***Design documents (P&IDs, design basis document, mass and energy balance, and design sketches)***
  - ***Hazards analysis document***
  - ***Project Execution Plan***
- ***Operating documents are based on core documents:***
  - ***Operating instructions***
  - ***General work instructions***
  - ***Communication sheets and logbooks***



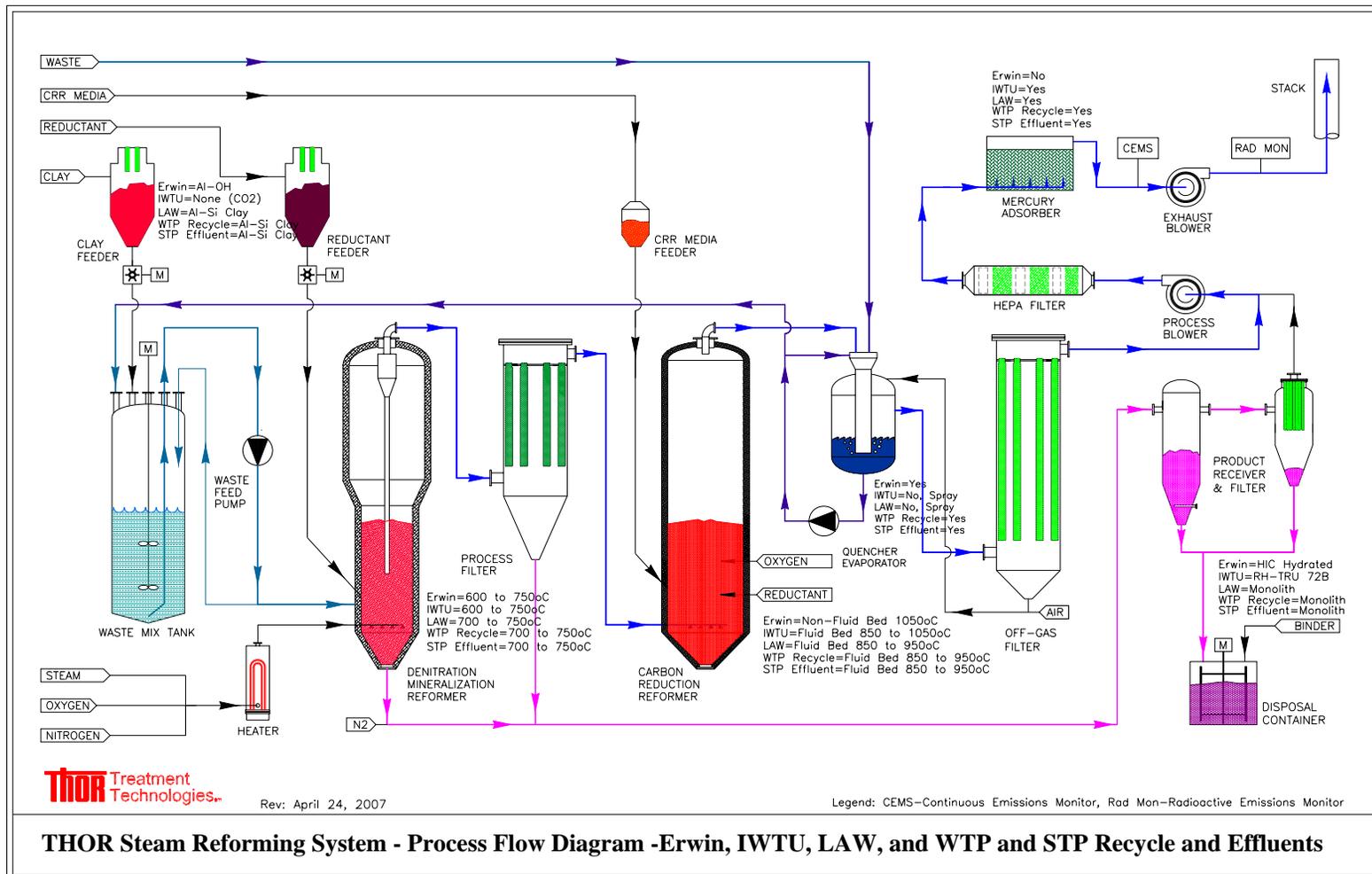
# ***Demonstration Program Implementation Technical Planning - IWTU***

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- ***Identify what needs to be tested by evaluation of:***
  - ***Waste to be treated***
  - ***Final disposition of waste form***
  - ***Data gaps***
  - ***Scale-up factors***
  - ***Remote maintenance features***
  - ***Other***
- ***Identify components for which above data is needed:***
  - ***Unit operations***
  - ***Equipment***
  - ***Integrated systems***

# Demonstration Program Implementation Technical Planning - IWTU

## Example: Process Flowsheet evaluation for IWTU



THOR Steam Reforming System - Process Flow Diagram -Erwin, IWTU, LAW, and WTP and STP Recycle and Effluents

# ***Demonstration Program Implementation Technical Planning - IWTU***

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- ***Establish test objectives:***

- ***Establish scale for test***

- ***Review existing data***
    - ***Determine allowable scale factors***

- ***Prepare flowsheet***

- ***Determine what equipment can be tested separately***
    - ***Determine what equipment must be tested as an integrated system***

- ***Identify variables and establish measurable limits***

- ***Instrumentation and data collection needed and frequency***
    - ***Analytical methods, detection limits, and frequency for samples***

- ***Establish acceptance criteria***

# ***Demonstration Program Implementation Technical Planning - IWTU***

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## ***Example: Major Test Objectives for IWTU***

- ***Confirm expected process chemistry and achieve excellent material balance closure***
- ***Demonstrate safe, predictable, long-term operation of key system components and the integrated process***
- ***Produce a granular solid product that:***
  - ***Contains essentially all radionuclide surrogates and major elemental components of the simulant feed***
- ***Collect necessary data for IWTU design and operation, e.g.***
  - ***Materials of construction***
  - ***Equipment sizing and design***
  - ***Process temperatures, pressures, and flow rates***
  - ***Automatic process control***
  - ***Process operating procedures***

# Demonstration Program Implementation

## Technical Planning - IWTU

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<b>Objective Statement</b>	<b>Quantifiable Objective Target or Criteria</b>	<b>Measurable Parameters and Test Methods</b>
<ul style="list-style-type: none"><li>• <b>Establish target steady-state system operating conditions</b></li></ul>	<ul style="list-style-type: none"><li>• <b>Verify initial carbon selection for DMR (steady, autothermal process operation is maintained)</b></li><li>• <b>Verify adequacy of DMR startup bed media (bed does not agglomerate, and sustainable DMR operation is maintained)</b></li><li>• <b>Demonstrate controlled, steady, carbon and O<sub>2</sub> addition to DMR to maintain stable autothermal process operation within a <math>\pm 20^{\circ}\text{C}</math> operating range.</b></li><li>• <b>Determine sustainable simulant feed rate to the DMR in gallons per minute and accompanying nozzle air ratios (NAR).</b></li></ul>	<ul style="list-style-type: none"><li>• <b>Monitoring of thermocouples in DMR</b></li><li>• <b>Monitoring of carbon types and addition rates</b></li><li>• <b>Monitoring of DMR bed particle appearance and size distribution</b></li><li>• <b>Flow controllers for gas and oxygen injection.</b></li><li>• <b>Monitoring of waste simulant feed rate and NAR</b></li></ul>

# ***Demonstration Program Implementation Technical Planning - IWTU***

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- ***Evaluate safety considerations***

- ***Perform hazards assessment***

- ***Identify hazardous constituents in simulants***

- ***Evaluate substitutions of non-hazardous surrogates***

- ***Identify required detection limits for constituents of interest***

- ***Determine minimum and maximum concentrations for each constituent***

- ***Identify industrial hazards and controls***

- ***Evaluate permitting requirements***

- ***Review existing and new permit requirements***

- ***Prepare permitting plan and strategy***

# ***Demonstration Program Implementation Technical Planning - IWTU***

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- ***Prepare test matrices***

- ***Evaluate and prepare scoping test matrix***

- ***Select main operating parameters***
    - ***Determine acceptable ranges for operating parameters***
    - ***Select major test series conditions for parametric operations***

- ***Evaluate and prepare demonstration test matrices***

- ***Prepare decision criteria for selection of final demonstration test operating parameters***
    - ***Sample and analysis plan matrices***
    - ***Measurable objectives stated***
    - ***Acceptance criteria fixed***

# Demonstration Program Implementation Technical Planning - IWTU

## Example – Scoping Test Matrix for IWTU

Test Number	Approx Duration (hrs)	Feed	Feed Rate (gpm)	DMR Temp (°C)	CRR Temp (°C)	UDS Conc. (g/L)	Alkali Comp <sup>a</sup>	Approximate Clay Conc. <sup>a, b</sup> (g/L)	Heavy Metals	POHC	Test Purpose
Preheat	48	None	0	Heat to 725	Heat to 950	0	NA	0	No	No	System to Temperature.
System Stabilization	1	Water	~0.10	725	950	0	NA	0	No	No	Initiate water feed.
MIN-S-1	32	SBW <sup>c</sup>	Up to 0.20	725	950	0	20%	339	No	No	Baseline DMR temp, high clay stoich.
MIN-S-2	35	SBW <sup>c</sup>	Up to 0.15	~750	950	0	20%	339	No	No	High DMR temp, high clay stoich.
MIN-S-3A	10	SBW <sup>c</sup>	Up to 0.15	~700	950	0	20%	339	No	No	Low DMR temp, high clay stoich. Test S-3B evaluated charcoal as a DMR solid reductant instead of coal.
MIN-S-3B	6										
MIN-S-4	12	SBW <sup>c</sup>	Up to 0.15	~700	950	0	26%	228	No	No	Low DMR temp, low clay mix ratio.
MIN-S-5	12	SBW <sup>c</sup>	Up to 0.15	~750	950	0	26%	228	No	No	High DMR temperature, low clay mix ratio.
MIN-S-6	13	SBW <sup>c</sup>	Up to 0.15	725	950	0	26%	228	No	No	Baseline DMR temperature, low clay mix ratio.
MIN-S-7A	25	SBW <sup>c</sup>	Up to 0.15	725	950	80	26%	228	No	No	Baseline DMR temp, low clay stoich, add UDS, obtain Hg stack emissions blank prior to P-1. DMR start-up bed was alumina for remaining tests.
MIN-S-7B	20										

a. Percentages shown represent total alkalis on an oxide basis. Actual testing ratios utilized may vary based on JTG, feed compositions, and refinement of mineralizing calculations.

b. The clay concentration at a given mix ratio is based on the concentration of alkali metals (Na, K, and Cs) in the SBW feed. (See Appendix D, TP 1.2.)

c. SBW without heavy metals, per Table 4, TP 1.2.

# Demonstration Program Implementation

## Technical Planning - IWTU

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### Example – Offgas Emissions Sample and Analysis Matrix for IWTU

Analyte	Location	Type	Applicable Tests	EPA Method
Hg	Downstream of GAC Stage 2	Extractive	Table 11	Ontario Hydro Method
Hg	Downstream of GAC Stage 3	Extractive	Table 11	Ontario Hydro Method
Hg, Pb, Cr, Ni, Ce, Cs	Stack	Extractive	Table 11	29
Particulate Matter	Stack	Extractive	Table 11	5
HCl, Cl <sub>2</sub>	Stack	Extractive	Table 11	26A
Dioxins/Furans/PCBs	Stack	Extractive	POHC feed	23A
VOCs	Stack	Extractive	POHC feed	0031
SVOCs	Stack	Extractive	POHC feed	0010
Total Organics	Stack	Extractive	POHC feed	0040, 0010
SO <sub>2</sub>	Stack	Instrumental	All	6C
NO <sub>x</sub>	Stack	Instrumental	All	7E
CO	Stack	Instrumental	All	10
CO <sub>2</sub>	Stack	Instrumental	All	3A
O <sub>2</sub>	Stack	Instrumental	All	3A
THC	Stack	Instrumental	All	25A

# Demonstration Program Implementation Technical Planning - IWTU

## Example – Offgas Emissions Sample and Analysis Matrix for IWTU

Test Number	Approx. Duration (hrs)	Hg GAC#1	Hg GAC#2	Metals	PM/ (HCl+Cl <sub>2</sub> )	D/F/PCBs	VOCs	SVOCs	Field GC	TCO/Grav	Total Sample Events**
EPA Method		OHM*	OHM*	29	5/26A	0023A	0031	0010	0040	0010	
MIN-S-7	24	X	X	X							3
MIN-P-1	Up to 48	X	X	X	X	X	X	X	X	X	9
MIN-P-2	Up to 72	X	X	X	X						4
MIN-P-3	Up to 72	X	X	X							3
MIN-P-4	Up to 72	X	X	X	X		X				5
MIN-P-5 (Optional)	Up to 72	X	X	X	X		X	X	X	X	8
MIN-P-6 (Optional)	Up to 24	X	X	X	X	X	X	X	X	X	9
<b>Sampling Events</b>		<b>7</b>	<b>7</b>	<b>7</b>	<b>5</b>	<b>2</b>	<b>4</b>	<b>3</b>	<b>3</b>	<b>3</b>	<b>41</b>

# Demonstration Program Implementation Technical Planning - IWTU

## Example – Solids and Liquid Sample and Analysis Matrix for IWTU

Sample Location	Sample Frequency	Number of Samples per Sampling Event	Analytical Laboratory	Analyses/Data Required
SBW Surrogate Solution/Slurry	Once every makeup batch or as requested	Two	Hazen Lab, INL Lab	Batch volume, concentration of each constituent in Tables 5 & 6, %UDS
DMR Startup Bed Material	At beginning of test program	Two	Hazen Lab	Archive one, analyze one
DMR Carbon Additive	At beginning of production run and after every 75 hours of operation	Two	Hazen Lab, INL Lab	Archive one, analyze one for ash constituents including constituents in Tables 5 & 6 (excluding H <sup>+</sup> )
DMR Bed Sample	As Requested	One	Hazen Lab, INL Lab	Each drain: total mass, particle size distribution (PSD), bulk density, particle density, wt% carbon. As Requested: wt% of each constituent in Tables 5 & 6 (excluding H <sup>+</sup> )
PR Solids Product	Once per day or as requested during production runs	Two	Hazen Lab, SRNL Lab, INL Lab	Archive one, analyze one. Each drain: total mass, particle size distribution (PSD), bulk density, particle density, wt% carbon. As Requested: wt% of each constituent in Tables 5 & 6 (excluding H <sup>+</sup> ), TCLP, PCT, XRD.
Continued				

# Demonstration Program Implementation

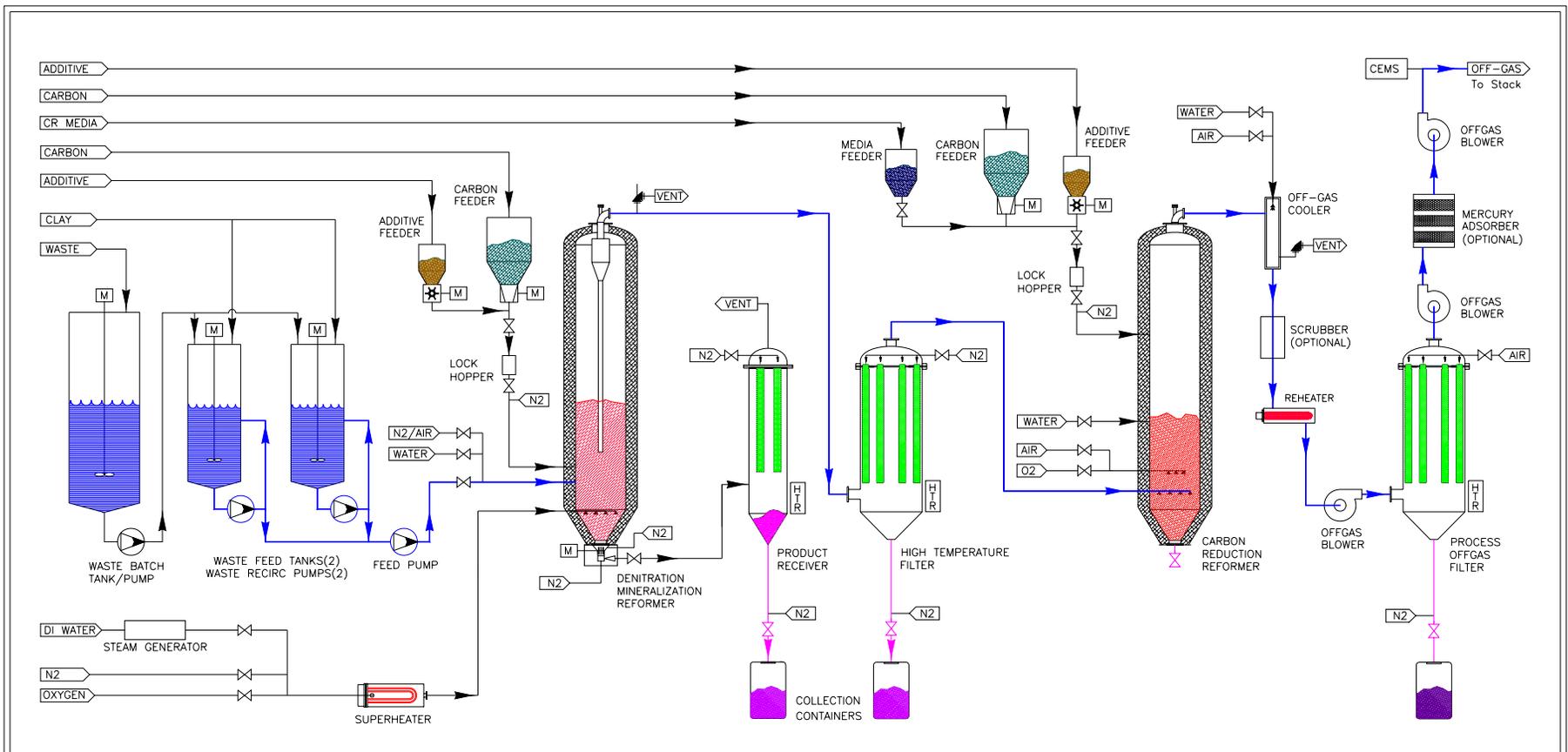
## Technical Planning - IWTU

**Example:**  
**Communication**  
**Sheet for IWTU**  
*(Sign-offs on bottom of sheet)*

		Parameter	Target Value	Expected Range	
DMR Fluidizing Gas	Primary Fluidizing Gas Flowrate			20 – 75 scfm	
	Composition			N <sub>2</sub> or Steam + Q	
	Superficial Velocity at Distributor			0.8 – 1.2 ft/s	
	Steam Boiler Pressure			Max. 50 psig	
	Steam PRV Setpoint			15 – 20 psig	
	Superheater A Outlet Temperature			300 – 400°C	
	Superheater B Outlet Temperature			550 – 650°C	
Bed Parameters	DMR	Bed Temperature		675 – 750°C	
		Fluidized Bed Depth		36 - 42 inches	
		Cyclone Blowback		On, Auto	
	CRR	Bed Temperature		850-1050°C	
Fluidized Bed Depth			25 - 36 inches		
Feed & Additives	DMR	Feedrate (Simulant)		0.1 – 0.4 gpm	
		Feedrate (Feed additive)		100 – 300 grams/hr	
		Carbon Feedrate		10 – 45 lb/hr	
		Atomizing Gas Flowrate		scfm	
	CRR	Carbon Feedrate		10 – 40 lb/hr	
		Mineralizing Additive		Aluminum hydroxide	
		Mineralizing Additive Feedrate		Feed at 2% of carbon feedrate but not less than 0.5 lb/hr	
		ATG Nozzles	Control Parameter		Flowrate or Bed Temperature
			Composition		50 vol% Q / 50% N <sub>2</sub> ; Max. 75%/25%
			O <sub>2</sub> Flowrate		scfm
Process Temperatures	HTF Heaters SP			400 – 500°C	
	PR Heaters SP			120 – 150°C	
	Off-Gas Cooler Outlet SP			150 – 240°C	
	Off-Gas Reheater Outlet SP			150 – 240°C	
	PBF Heaters SP			150 – 180°C	
	GAC Heaters SP			150 – 180°C	
Other					

# Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU

## Finalize Flowsheet



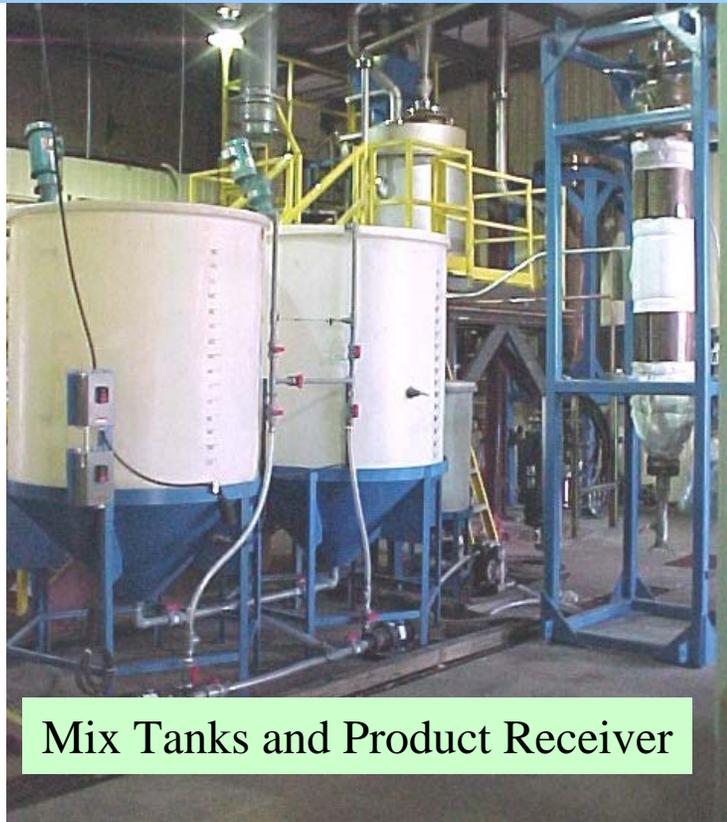
# ***Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU***

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- ***Contract with Test Facility***
- ***Procure hardware***
  - ***Process equipment***
  - ***Support systems***
    - ***Compressed gases***
    - ***Electrical power***
- ***Procure simulant or chemical ingredients***
- ***Assembly process systems***
- ***Program control system***
- ***Checkout electrical, piping, and instruments***
- ***Calibrate instruments***

# *Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU*

Engineering Scale Demonstration Plant  
Hazen, 15" FBSR, Full Integrated System



Mix Tanks and Product Receiver



DMR Reformer and Filter

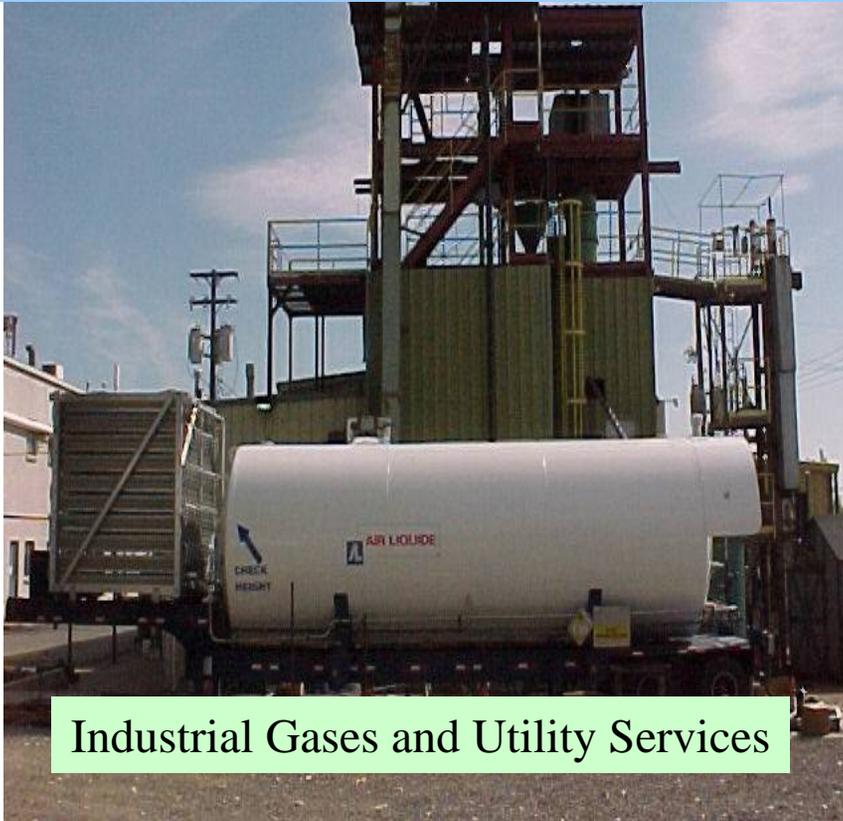
# *Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU*

Engineering Scale Demonstration Plant  
Hazen, 15" FBSR, Full Integrated System



# *Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU*

Engineering Scale Demonstration Plant  
Hazen, 15" FBSR, Full Integrated System



Industrial Gases and Utility Services



Control System

# ***Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU***

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- ***Training***

- ***Process safety***
- ***Industrial safety***
- ***Test Plan and process operating matrix***
- ***Conduct of operations***
- ***Operating procedures***

- ***Conduct of Operations***

- ***Responsibilities of each member of team***
- ***Procedure compliance***
- ***P&ID and procedure changes and control***
- ***Communication sheet for turnovers and process system changes***
- ***Logbooks***

# ***Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU***

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## **•Checkout and Calibration Checks**

- Completion of process and mechanical installation***
- Punchlist development***
- Instrument calibration***
- Control system logic checks***
- Electrical continuity and motor rotation***
- Valve position and actuation***
- Emergency shutdown checks***
- As-Build P&IDs***
- Final P&ID and procedure approval***

# ***Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU***

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- ***Start-up and Operating Procedures***
  - ***Simulant makeup and transfer***
  - ***Process operations***
  - ***Sample collection and handling***
  - ***Analytical methods***
  - ***Alarm responses***
  - ***Notifications***

# ***Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU***

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- ***Gaseous Emissions Data Supports Permitting***
  - ***Gather extensive gaseous effluent data by an independent subcontractor to support permitting (CEMS and grab samples)***
  - ***Determine MACT compliance for applicable pollutants (e.g. dioxins/furans, Hg, Cd, Pb, Cr, particulates, HCl+Cl<sub>2</sub>, CO, THC, and organic DRE for principal organic hazardous constituent (POHC))***
  - ***Measured quantities of heavy metals and POHC to be added to the process during production tests***
  - ***Grab samples to be obtained for the various pollutants per EPA protocols***

# ***Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU***

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## ***• Pilot Plant Operations – Staffing***

### ***– TTT Test Team***

- Test Manager, one***
- Emission controls expert (monitor third party emissions contractor), one***
- Test Engineers, two per 12 hr shift***
- QA Engineer, part-time***
- Process Engineer, one***

### ***– Operations Team (Hazen personnel)***

- Operations Manager***
- Shift Supervisor, one per 12 hr shift***
- Operators, three to five per 12 hr shift***

### ***– Emissions Contractor***

- Engineers, two***

# ***Demonstration Program Implementation Pilot Plant Preparation and Operations - IWTU***

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- ***Management of Changes During Testing***
  - ***Joint Test Group (JTG)***
    - ***Team consisting of:***
      - ***TTT Test Manager***
      - ***TTT Lead Shift Test Engineer***
      - ***TTT Process Engineer (design authority representative)***
      - ***Hazen Operations Manager***
      - ***Other team members and subject matter experts as required***
    - ***Review pilot plant operations daily***
    - ***Approve changes to test Matrices, operating conditions, P&IDs, and procedures on daily basis***
    - ***Evaluate and resolve problems real-time***
    - ***Maintain design and documentation configuration***

# ***Demonstration Program Implementation Post Test Activities - IWTU***

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- ***Shutdown and de-energize systems***
- ***Cleanout systems***
- ***Package and archive samples and products***
- ***Dispose of excess surrogates, consumables, and products***
- ***Complete analyses of samples***
- ***Demobilize operations personnel***
- ***Write final report***

# Demonstration Program Implementation

## Simulant Preparation – IWTU SBW

**Example – Recipe for the Preparation of 100 Liters of SBW Simulant Solution with heavy metals and radionuclide surrogates for IWTU**

Component	Reagent	Target Component Concentration (mol/L)	Adjusted Reagent Concentration (mol/L)	Reagent Amount (kg or liters)
<b>Cations in Solution</b>				
Acid	HNO <sub>3</sub> (See Nitrate)	2.88		
Aluminum	Al(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O (60 wt% sol'n, ρ=1.33)	0.719	0.719	33.788 liters
Boron	H <sub>3</sub> BO <sub>3</sub>	0.0217	0.0217	0.134 kg
Calcium	Ca(NO <sub>3</sub> ) <sub>2</sub> ·4H <sub>2</sub> O (79 wt% Ca(NO <sub>3</sub> ) <sub>2</sub> )	0.0731	0.0731	1.518 kg
Cerium	Ce(NO <sub>3</sub> ) <sub>3</sub> ·6H <sub>2</sub> O	0.00483	0.00483	0.210 kg
Cesium	CsNO <sub>3</sub>	0.00353	0.00353	0.0688 kg
Chromium	Cr(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O	0.00569	0.00569	0.228 kg
Iron	Fe(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O	0.0217	0.0217	0.876 kg
Lead	Pb(NO <sub>3</sub> ) <sub>2</sub>	0.00134	0.00134	0.0447 kg
Magnesium	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (67 wt% sol'n, ρ=1.35)	0.0257	0.0257	0.728 liters
Manganese	Mn(NO <sub>3</sub> ) <sub>2</sub> (50 wt% sol'n, ρ=1.53)	0.0152	0.0152	0.355 liters
Mercury	Hg(NO <sub>3</sub> ) <sub>2</sub> ·2H <sub>2</sub> O	0.00713	0.00713	0.257 kg
Nickel	Ni(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	0.00255	0.00255	0.0742 kg
Potassium	KNO <sub>3</sub>	0.225	0.225	2.272 kg
Sodium	NaNO <sub>3</sub>	2.20	1.94	16.490 kg
Zinc	Zn(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	0.00800	0.00800	0.238 kg
<b>Anions in Solution</b>				
Chloride	NaCl	0.0334	0.0334	0.195 kg
Fluoride	HF (28.9 M sol'n)	0.0506	0.0506	0.175 liters
Nitrate	HNO <sub>3</sub> (67 wt% sol'n, ρ=1.40)	7.53	2.88	19.34 liters
Phosphate	Na <sub>3</sub> PO <sub>4</sub> ·12H <sub>2</sub> O	0.00500	0.00500	0.190 kg
Sulfate	Na <sub>2</sub> SO <sub>4</sub>	0.107	0.107	1.519 kg

# Demonstration Program Implementation

## Simulant Preparation – IWTU SBW

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**Example – Recipe for the preparation of UDS to be added to the SBW supernatant solution for IWTU**

Component	Reagent (Anhydrous)	Component Concentration (wt%)	Reagent Conc. (wt%)
Aluminum	$\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ (kaolin)	2.2	8.9
Iron	$\text{Fe}_2\text{SiO}_4$	2.6	4.7
Silicon	Ground Quartz	24.3*	45.8
Zirconium Phosphate	$\text{Zr}(\text{HPO}_4)_2$	13.1 27.2	40.6
<b>Total Batch</b>			<b>100.0%</b>

# ***Demonstration Program Implementation Simulant Preparation – IWTU SBW***

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- ***IWTU SBW Simulant Preparation - Recipe***
  - ***Recipe prepared by subject matter experts from CWI and TTT***
  - ***Major constituents***
    - ***Concentrations selected to represent most concentrated INTEC tank***
  - ***Minor constituents***
    - ***Concentrations based on highest concentration in three tanks, except***
    - ***Potential catalytic metals (Cr, Ni, Fe) concentrations based on lowest concentration***
  - ***Undissolved Solids (UDS)***
    - ***Inert solids***
    - ***Particle sizes and compositions based on sample analysis from tanks***
    - ***Concentration varied during test series***

# ***Demonstration Program Implementation Simulant Preparation – IWTU SBW***

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- ***IWTU SBW Simulant Preparation - Procedure***
  - ***Individual chemical ingredients procured by Hazen***
  - ***Hazen operations personnel handle, mix, and transfer ingredients and batched simulants***
  - ***Add and mix batch of soluble chemical ingredients into mix tank to make baseline SBW solution***
  - ***Transfer batched baseline SBW solution to one of two waste feed tanks***
  - ***Add heavy metals and UDS solids to waste feed tanks as required by Test Matrix, concentrations may vary***
  - ***Meter combined baseline SBW solution with and without heavy metals and UDS into first reformer***
  - ***Draw samples and analyze composition***

# ***Demonstration Program Implementation Simulant Preparation – IWTU SBW***

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- ***IWTU SBW Simulant Preparation – Lessons Learned***
  - ***Large pieces of chemical ingredients dissolve slowly, must maintain solution well mixed, provide time for dissolution***
  - ***Verify particle size of insoluble solids to minimize solids settling and prevent plugging when transferred***
  - ***Analyze incoming chemical ingredients and verify chemical formulation, especially hydrates***
  - ***Analyze each simulant feed batch***
  - ***Maintain full cover over top of mix tanks to avoid splashing***
  - ***Verify metallurgy of pumps, handling equipment, and seal materials of construction for compatibility with simulant***

# **THOR<sup>®</sup> Steam Reforming**

## **Pilot Plant Test Programs - Lessons Learned**

- **Programmatic - Lessons Learned**
  - **Provide means to make changes during test operations**
  - **Provide authorized Joint Test Group who perform functions:**
    - **Control day-to-day operations**
    - **Provide real-time approval of changes to optimize process and resolve problems**
    - **Maintain configuration control on test documents and systems**
    - **Ensure all work is done safely**
    - **Issue formal daily report**
  - **Daily communications to stakeholders controlled to official daily project status report**
  - **Involve DOE and environmental regulators in operations and plans**

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## **Pilot Plant Test Programs - Lessons Learned**

- **Programmatic - Lessons Learned**
  - **Perform work at commercial facilities where possible**
    - **Utilizes trained technical staff**
    - **Uses existing hardware that can be modified as required**
    - **Cost effective**
    - **Merge operations and safety cultures**
  - **Retain continuity in staffing for positions with high training requirements and knowledge base**
    - **Control system programmers**
    - **Operations supervisors**
    - **Subject matter experts**
    - **Interdisciplinary staff to facilitate timely response to changes**
  - **Use communication sheets with signed approvals and acceptances to document changes - between test managers and operations personnel**

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## **Pilot Plant Test Programs - Lessons Learned**

- **Programmatic - Lessons Learned**
  - **Provide Schedule Contingency**
    - **Design, Construction, and Operation of pilot plants is inherently an iterative process**
    - **Allow time in schedule for hiccups – they will occur**
  - **No Activity should be deemed a failure**
    - **Finding the edge of an envelope is better in a pilot facility than a production plant**
  - **Data is the product – Collection of data is paramount**
    - **Estimate the resources to install, program, and validate instruments, control systems, and software – Double it**
    - **Do not skimp on quantity or quality of measurements**