



U.S. Department of Energy
Energy Efficiency and Renewable Energy

biomass program

Process Monitoring Tools

**DOE OBP Thermochemical Platform Review Meeting
June 7-8, 2005**

**Vann Bush
Gas Technology Institute**



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- **Project Background and Objectives**
- **Pathways and Milestones**
- **Technical Feasibility and Risks**
- **Competitive Advantage**
- **Project Overview**
- **Accomplishments**
- **Plan/Schedule**
- **Critical Issues**
- **Progression to Next Stage**
- **Summary**



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- FY02 and FY03 Appropriated Funding
 - DE-FC36-02GO12024, DE-FC36-03GO13175
- Focus on Syngas R&D Platform
 - Systems Integration Issues
 - Syngas Cleanup and Syngas Analysis
- Crosscutting Technology for Multiple Pathways
 - Stage Gate Research Track
 - Development Stage
- Project Scheduled Completion 1st Quarter FY06



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- Produce and validate an innovative process diagnostic and monitoring system to quantify syngas composition in gasification and gas cleanup process streams.
- Evaluate gas cleanup and treatment technologies that are required, available, and proven for various syngas end uses.



Pathways and Milestones – C-level and Project Milestones

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Aq Residues

Perennial Crops

Pulp and Paper

Forest Products

Validate Gasification Performance

M 4.11.2
M 4.12.2

M 5.11.2
M 5.12.2

M 6.3.3

M 7.1.3

Validate Gas Cleanup Performance

M 4.11.3
M 4.12.3

M 5.11.3
M 5.12.2

M 6.3.4

M 7.1.4

Validate Integrated Gasification and Gas Cleanup

M 4.11.5
M 4.12.5

M 5.11.5
M 5.12.5

M 6.2.3
M 6.2.4

M 7.1.5
M 7.3.1

Project Milestones	Type	Performance Expectations	Due Date
Select and assemble analytical suite	D	Provide comprehensive analysis of syngas components from raw gas to ultra-clean gas purity	Feb. 2005
Complete analytical suite validation tests	D	Conduct syngas measurements at two test sites with multiple stages of syngas contaminant removal	Sept. 2005



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The project will be successful because:

- Novel **process interface strategy** and technology meet prerequisite for accurate syngas sampling
- Selection and deployment of **state-of-the-art commercial analyzers** lowers technical risks
- Some **redundancy** available in suite to insure coverage of syngas components
- Standard **QA/QC** methods (reference batch methods, spike and recovery ,etc) can be used in assessing analytical accuracy
- **Multiple test sites and multiple feedstocks** permit validation of analytical system and methods



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Advantages of Accurate On-Line Analytical Systems

- Key enabler for cost-effective technology development and systems integration work
- Required technology for process optimization (needed to meet economic performance goals)
- Essential capability for on-line production monitoring to meet syngas end-user specifications



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gti[®]

Subcontractors



Technical Advisory Panel



Institute of Paper Science and Technology
EDUCATION • RESEARCH • TECHNOLOGY TRANSFER • INFORMATION





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Comprehensive, continuous, sensitive and accurate measurements at key points in the process are essential to:

- Compare gasification conditions
- Quantify fuel-specific syngas components
- Characterize syngas quality for end uses
- Monitor gas conditioning and cleanup effectiveness in short-term tests



IMACC FT-IR



SRS
Mass Spec

Varian
GC/PFPD



Agilent GC/MSD/FID/SCD



Contaminant Tolerance Limits for Several Biomass-Derived Fuel Gas Utilization Applications

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Contaminant		Turbine	SOFC	Chemicals
Sulfur	ppmv	20	1 - 0.05	0.2 - 0.01
Halides	ppmv	1	1 - 0.01	0.1 - 0.01
Ammonia	ppmv		5000	10 - 0.02
Sodium + Potassium	ppmw	0.08		0.01
Particles (total)	ppmw	3.0	?	0
Particles (5 -10 μm)	ppmw	0.15		
Particles > 10 μm	ppmw	0.03 - 0		
C ₂ -C ₆	ppmv		2000	? (low)



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Optimize extraction and delivery of syngas samples

- use GTI interface technology to prevent losses and contamination of the syngas sample streams
- operate each analyzer at its upper temperature limit
- condition syngas streams only as much as required for each analyzer

Adapt state-of-the-art analytical instruments and methods to fully characterize syngas composition



Process Interface Strategy

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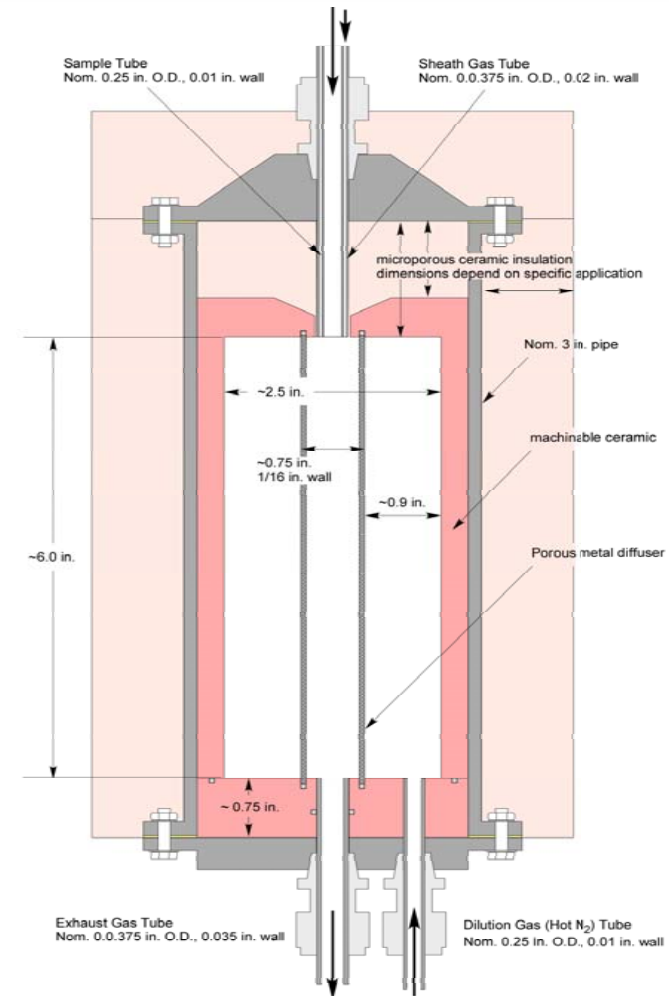
- Goal:** Introduce sample streams of syngas into a suite of analyzers without condensing vapor-phase hydrocarbon species
- Approach:** Lower the partial pressures of the vapor-phase hydrocarbon species:
- depressurize the syngas sample streams to 1 – 2 bar
 - operate each analyzer at its upper temperature limit
 - dilute syngas stream only as much as required for each analyzer
- Tool:** Novel, cylindrical cooling chamber with nitrogen-wall dilution
- Operation:** Minimize dilution by actively comparing measured concentrations (partial pressures) of vapor-phase species with their condensation thresholds
- Advantage:** Near-continuous measurement of unaltered syngas species



Dilution Cooler Sampling Interface

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- Provides syngas interface to analytical instruments
- Delivers syngas to analyzers at precise temperatures
- Preserves syngas components as vapors to enable on-line, near real-time analyses of composition





Analytical Suite for Syngas

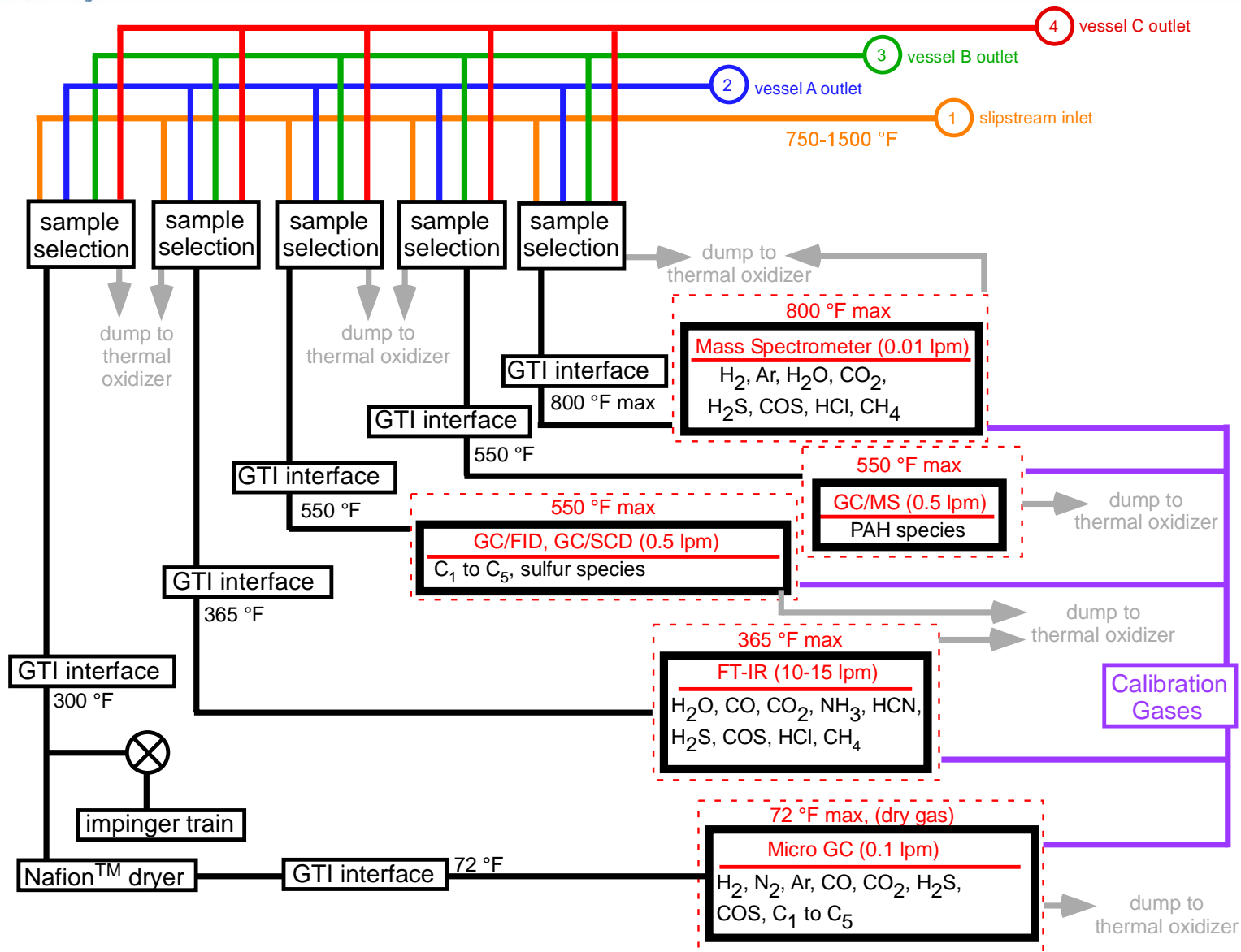
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COMPONENT	INSTRUMENT / METHOD
H ₂ , N ₂ , Ar, CO, CO ₂ , H ₂ S, COS, C ₁ to C ₅	Micro Gas Chromatograph
H ₂ O, CO, CO ₂ , NH ₃ , HCN, H ₂ S, COS, HCl, CH ₄	Fourier Transform-Infrared Spectroscopy
H ₂ , Ar, H ₂ O, CO ₂ , H ₂ S, COS, HCl, CH ₄	Mass Spectrometer
sulfur species	Gas Chromatograph / Sulfur Chemiluminescence Detector / Flame Photometric Detector / Pulsed Flame Photometric Detector
C ₁ – C ₅ hydrocarbons	Gas Chromatograph / Flame Ionization Detector
tar and oil species, aromatics	Gas Chromatograph / Mass Spectrometer



Sampling Manifold Concept

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Analyzer Sampling Locations

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Gasification

Power

Fuels / Chemicals

Sampling Point	Raw Gas	Clean Gas	Ultra-Clean Gas
Probable Samples for Specific Gas Analyzers			
GC/MS/FID/SCD	☒	☒	☒
GC/FPD/TCD	☒		
FT-IR	☒	☒	☒
GC/PFPD		☒	☒
MS	☒		
μGC	☒	☒	☒

- ☒ = primary location
- ☒ = secondary location
- ☒ = other location(s)



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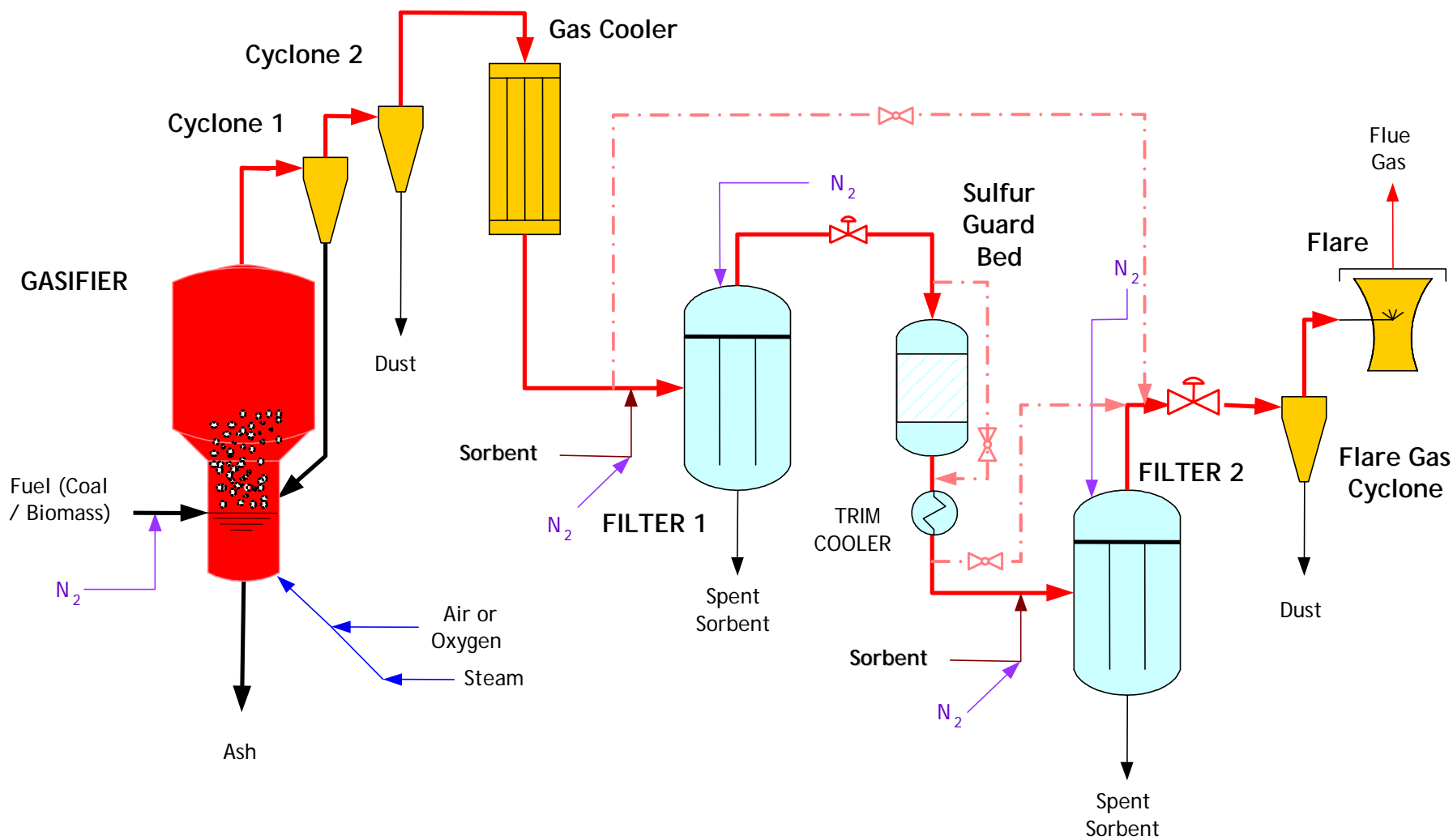
- Coal – 10 tpd w/air; 17 tpd w/oxygen
- Biomass – 24 tpd w/air; 40 tpd w/oxygen
- Opportunity Fuels & Dual Fuel Operation
- Downstream Cleanup Systems
- Hydrogen Production
- CO₂ Removal Technologies
- Gas to Liquids
- Advanced Power Conversion Systems Tests (Fuel Cells, Small Turbines, Engines)
- Advanced Oxygen Production Systems





GTI Flex-Fuel Test Facility with SWPC Ultra-Clean Process Flow Diagram

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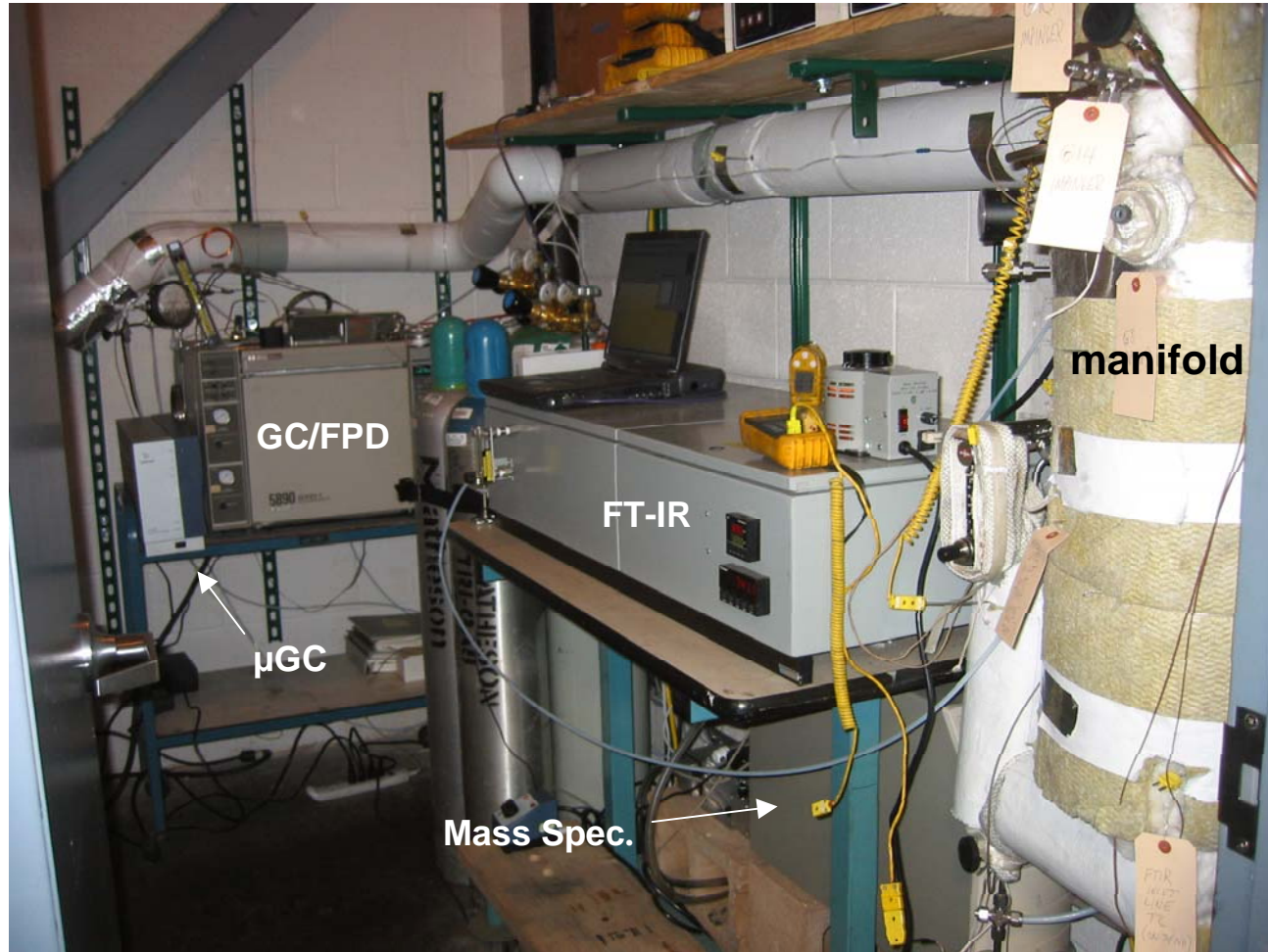




Analytical Suite – Coordinated Sampling for Comprehensive Measurements

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Multiple
analyzers
connected
through a
sampling
manifold.

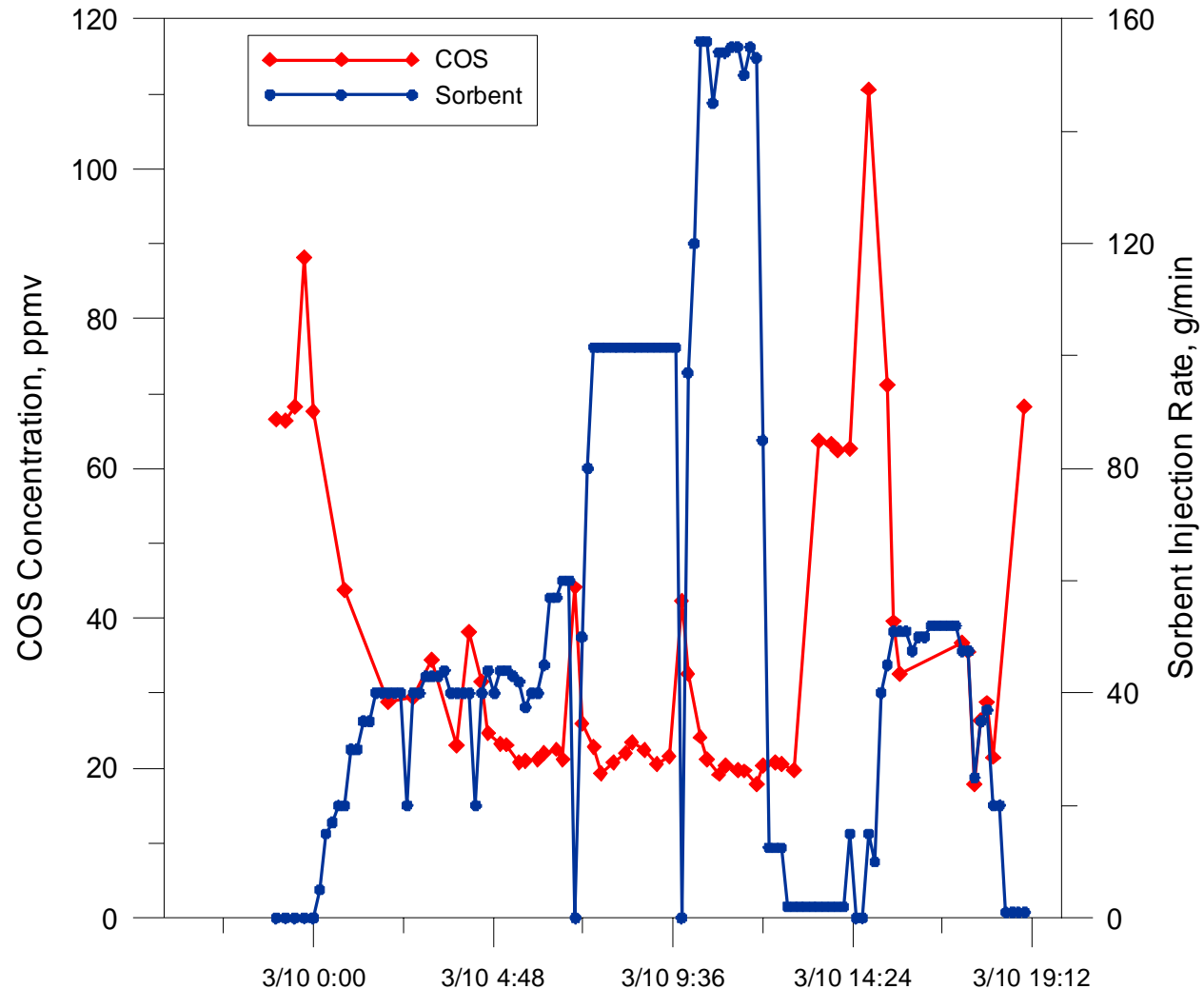




Analytical Results – Sorbent Effect on COS Concentration

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GC/FPD (Agilent 5890) was used to monitor sulfur species including COS downstream of filter-reactor with halide control sorbent.

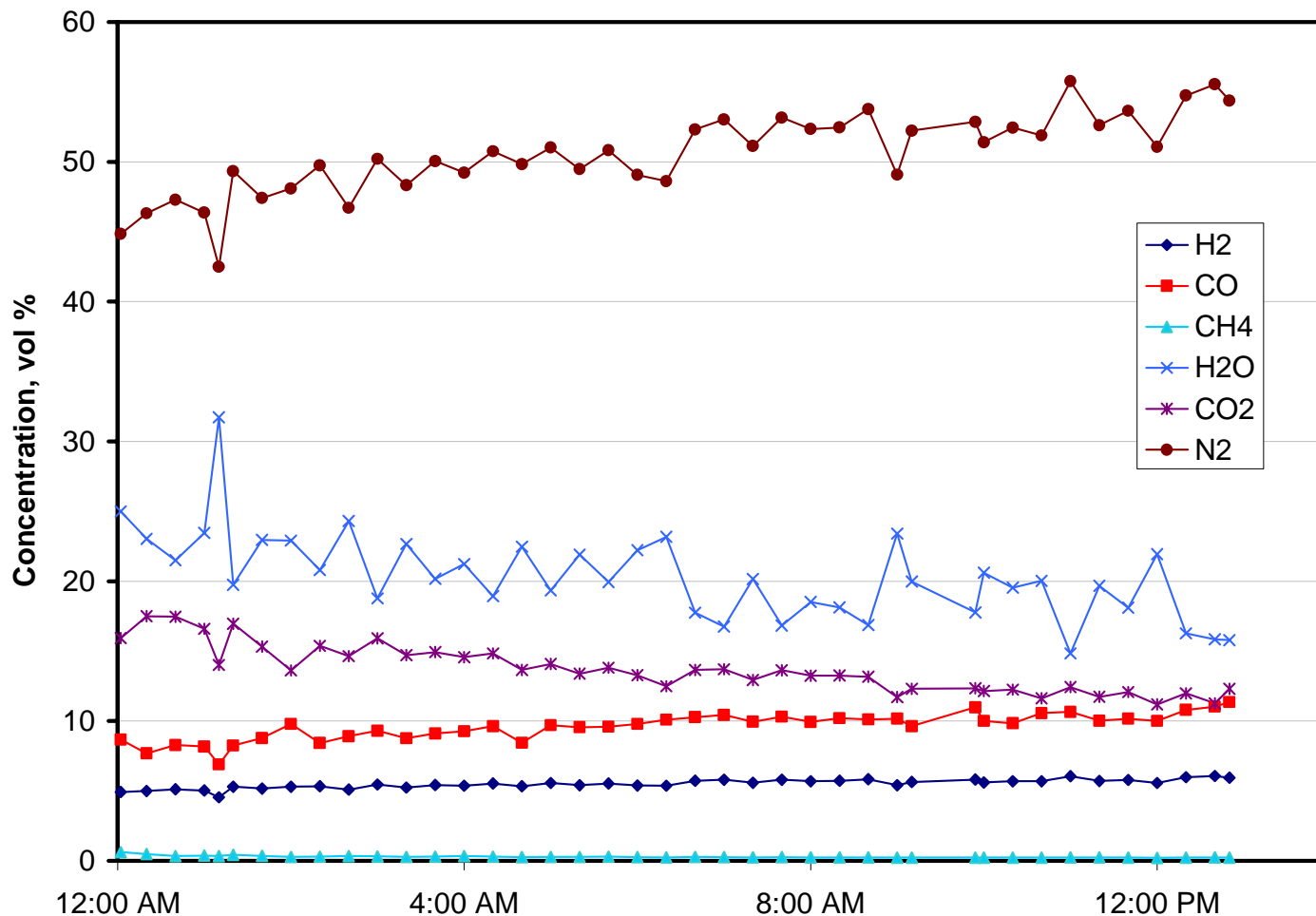




Analytical Results – Major Species Monitoring

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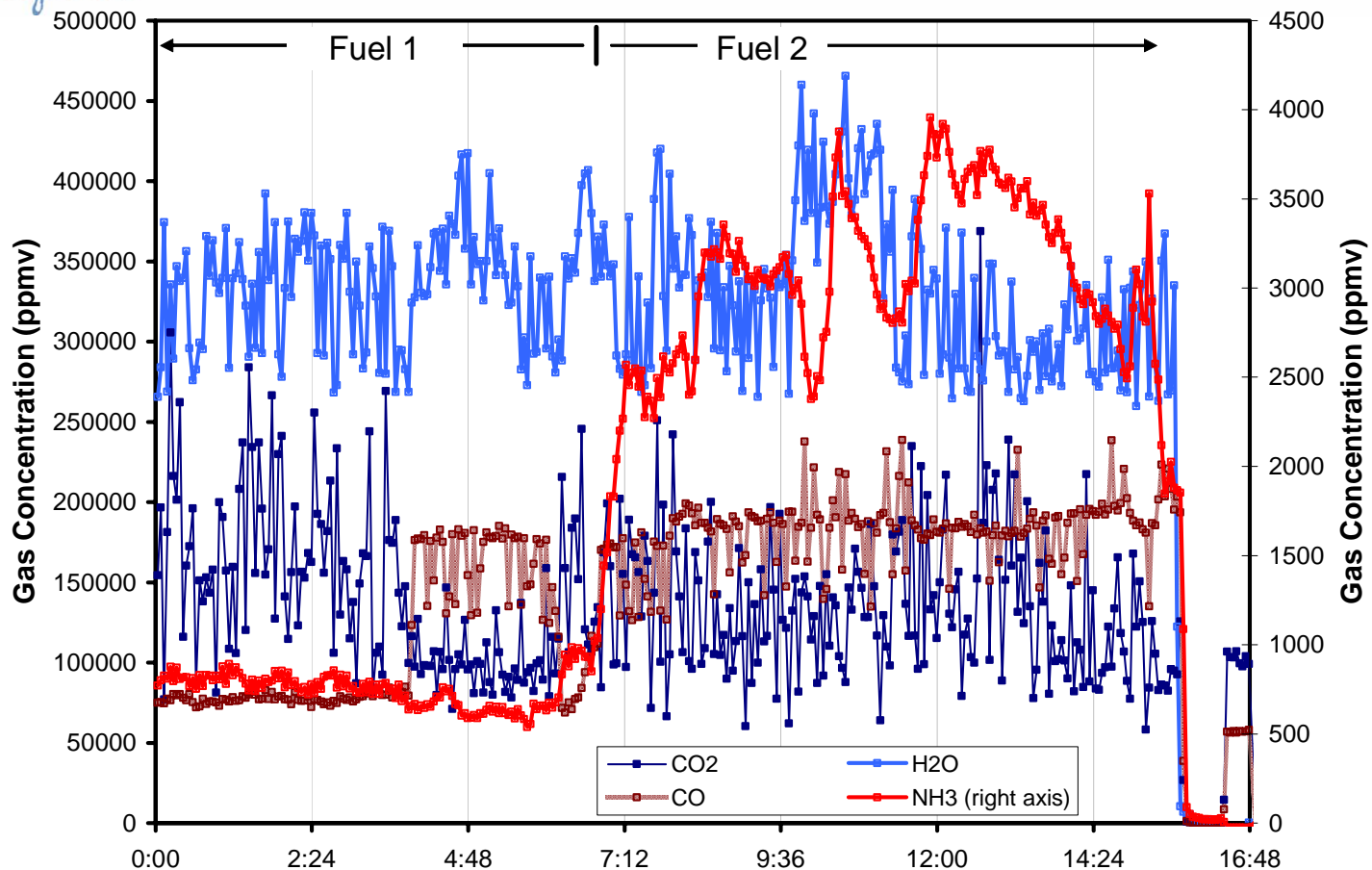
Mass spectrometer (SRS QMS300) monitoring major gas components.





Analytical Results – FTIR Rapid Process Monitoring

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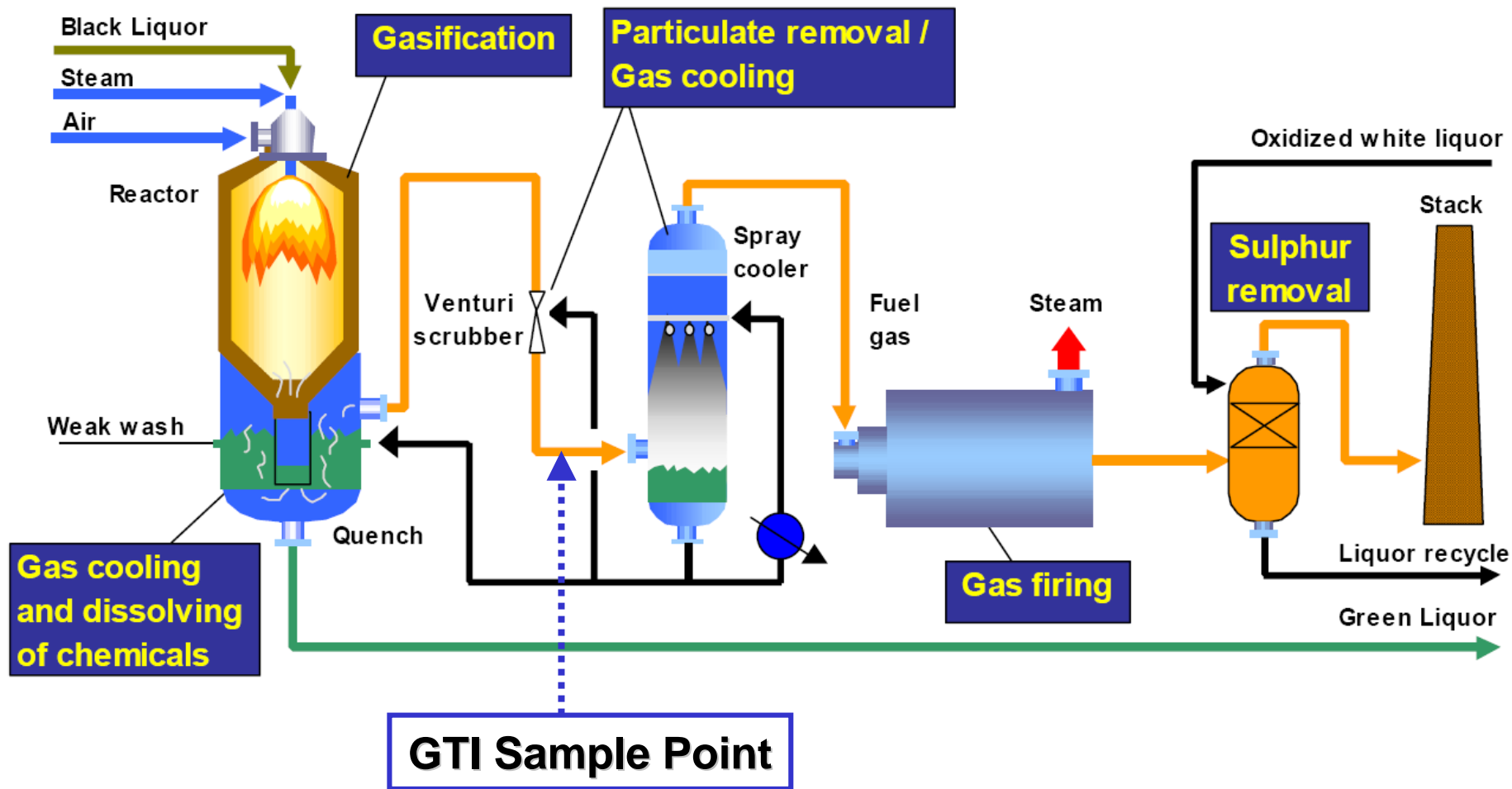


FTIR (IMAAC, 10-m cell) quantifies multiple species with very fast response time useful to identify fuel or process changes.



Syngas Analysis: Chemrec Booster Gasifier at Weyerhaeuser's New Bern Mill

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Testing at Weyerhaeuser New Bern Black Liquor Gasifier

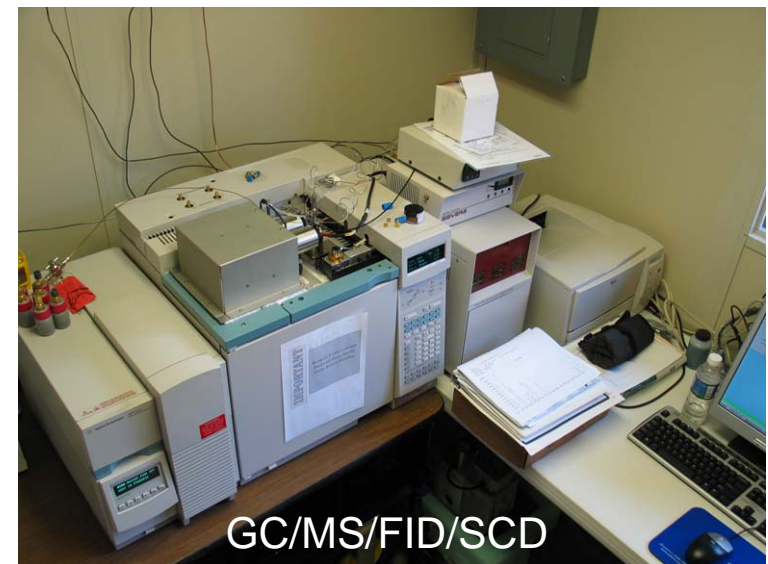
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GTI testing focus

- major components
- sulfur species
- hydrocarbon species

Instruments

- FT-IR
- Mass Spec
- μ GC
- GC/MS/FID/SCD





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- Novel sampling interface developed
- Analytical instruments selected
- Sampling manifold designed and implemented
- Syngas measurements made in multiple gasification campaigns
- Measured raw gas to ppb-level halide and sulfur contaminants



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- Plan called for tests at GTI Flex-Fuel Facility and DOE/Southern Company PSDF
 - GTI FFTF tests conducted in 2004, 2005
 - PSDF test delayed by Southern Company into 2006
- Upcoming tests at two sites
 - Chemrec black liquor gasifier at Weyerhaeuser's New Bern Mill, NC (June 2005)
 - Fluidized bed gasifier at GTI Flex-Fuel Test Facility in Des Plaines, IL (August 2005)
- Development Stage work completion by 2nd Q FY06
 - Document findings by October 2005



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- Limited testing opportunities
 - Available biomass gasification and syngas cleanup sites
 - Multiple fuels, gasifiers, and gas cleanup systems improve validation of techniques
 - Hydrocarbon production
 - Contaminant level



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Development Stage success will deliver specifications, protocols, and methods for the analysis of syngas quality from pilot, demonstration, and commercial thermochemical biomass conversion processes.



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- FY05 expenditures – approx. \$1.5 million
- Providing on-line monitoring and diagnostic measurements for gasification-based processes
 - Transportable capability
 - Full range of syngas major and minor species
- Evaluating syngas reforming and cleanup technologies
 - Raw syngas to ultra-clean levels
- Enables cost-effective process evaluation and process optimization