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Sulfuric Acid Decomposition

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Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company,
for the United States Department of Energy's National Nuclear Security Administration
under contract DE-AC04-94AL85000.

Overview

- Project objectives
- Description of current experimental tests
- Experimental results (Gelbard et al., “High Pressure Sulfuric Acid Decomposition Experiments for the Sulfur-Iodine Thermochemical Cycle,” SAND2005-5598, 2005.)
- Summary & Plans for next experimental series leading to Integrated Lab-Scale (ILS) experiments

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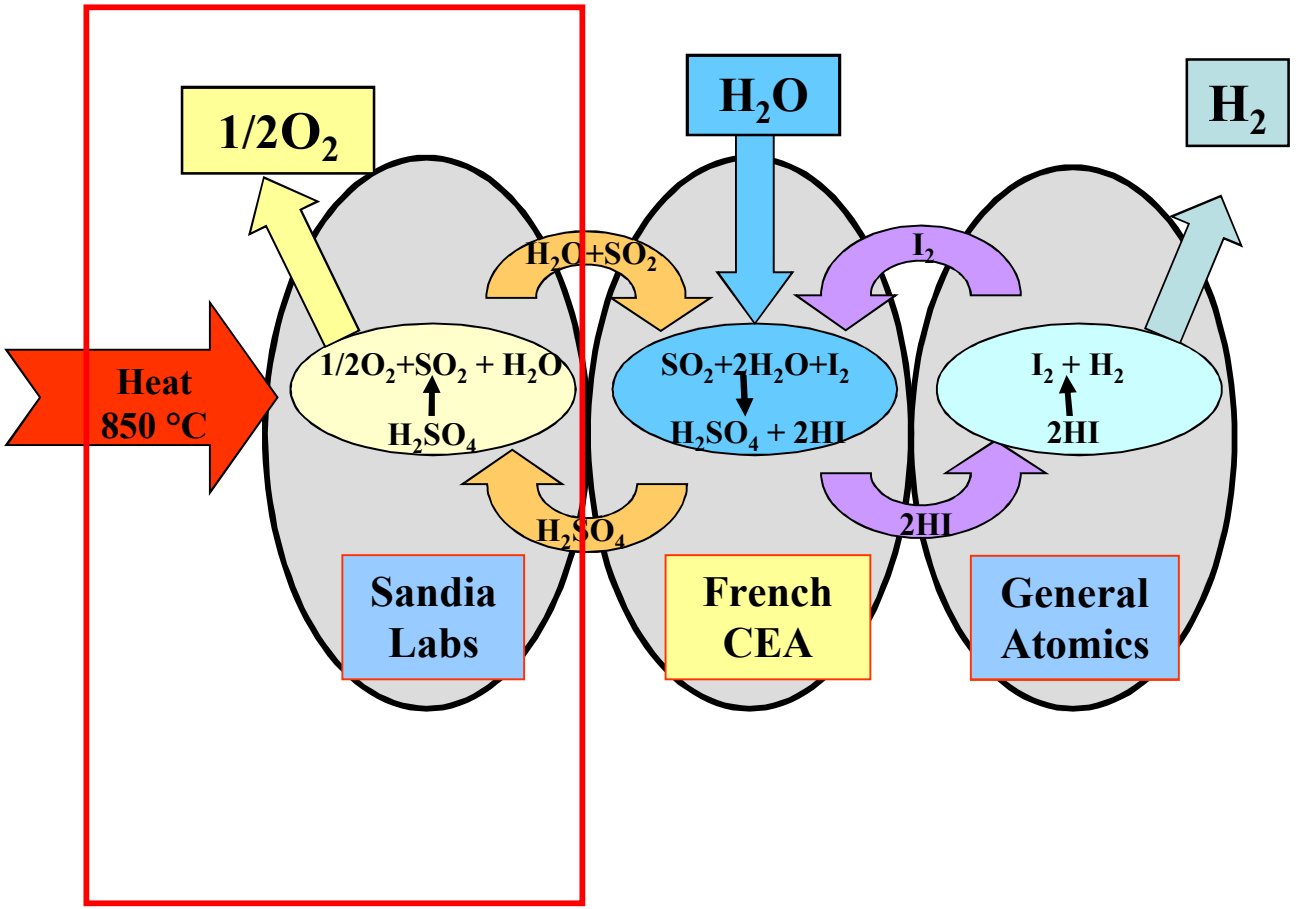
Sulfur-Iodine Thermochemical Cycle



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Objectives

- **Demonstrate laboratory-scale process with engineering materials**
  - Design, construct, assemble, and test laboratory-scale apparatus and instrumentation
  - Philosophy: “First get it to work, then improve incrementally ”
- **Assess process performance and materials**
  - Variations in acid concentration, temperature, and pressure
  - Catalyst: conversion, poisoning, acid concentration, temperature, pressure
  - Engineering materials (starting with Hastelloy, Incoloy, ceramics, and glass)
- **Integrate into a closed-loop SI cycle: Integrated Lab-Scale (ILS) Experiments**
  - Real-time instrumentation for process control

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Status of Accomplishments

- Modular boiler, superheater, decomposer, and fluid collection units built and successfully tested six times.
- Three pressurized acid tests performed.
- Tested real time gas flow measurements to directly obtain acid conversion fraction.
- Assembling FT-IR for SO3, SO2, and H2O in-situ concentration measurements
- Assembling DCHX (Direct Contact Heat Exchanger) which will recycle heat and unreacted acid

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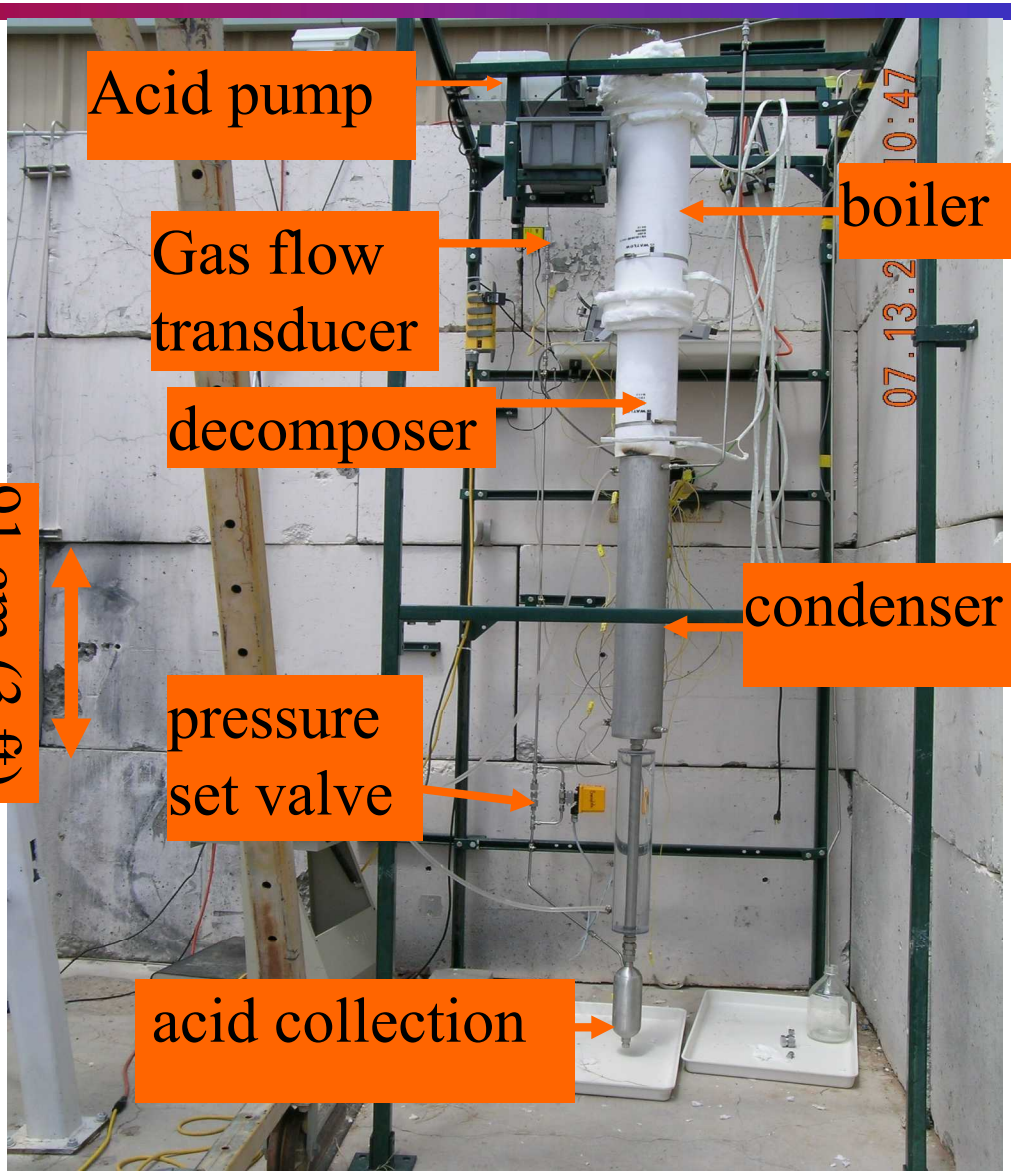
Sulfuric Acid Decomposition Apparatus



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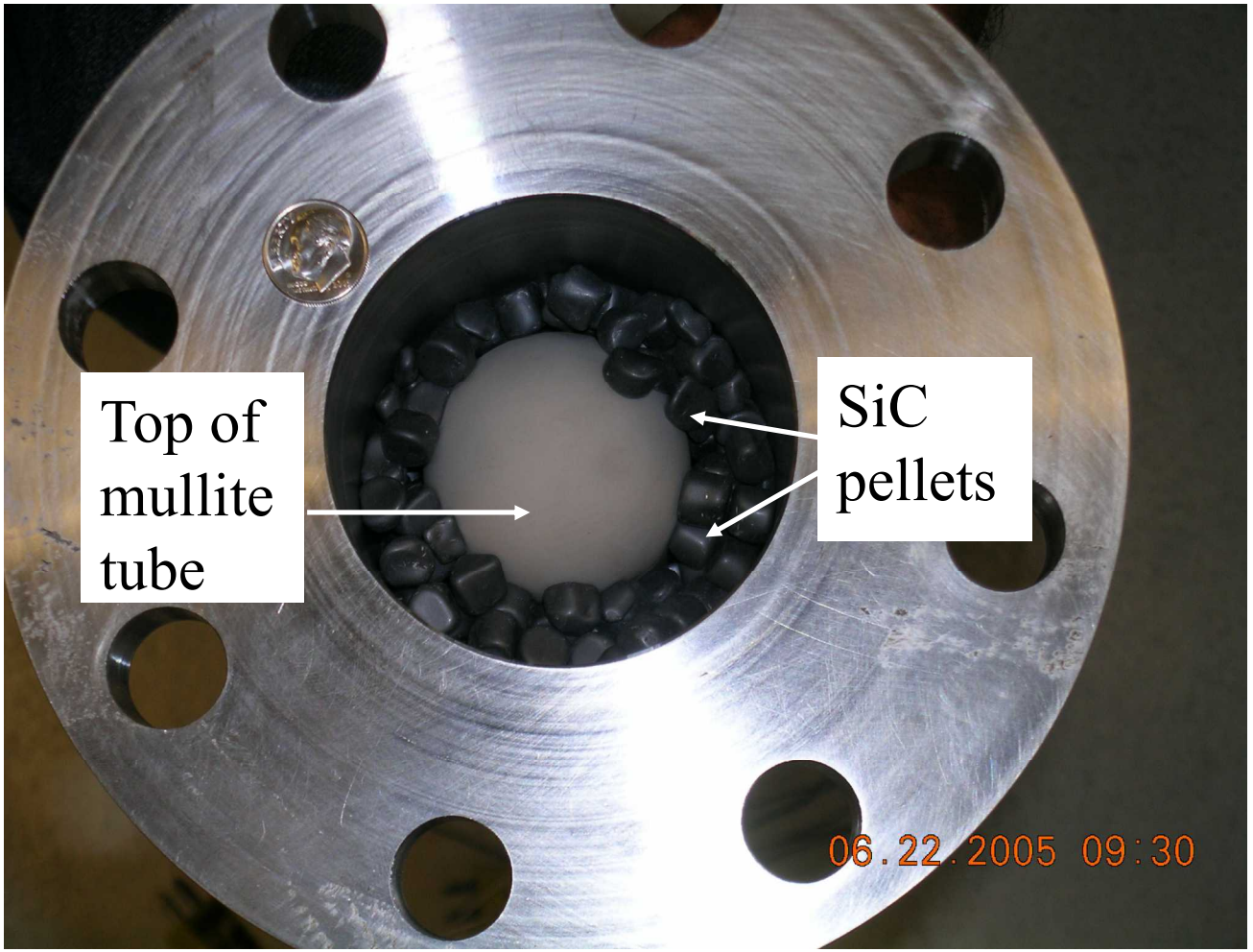
Top of Boiler



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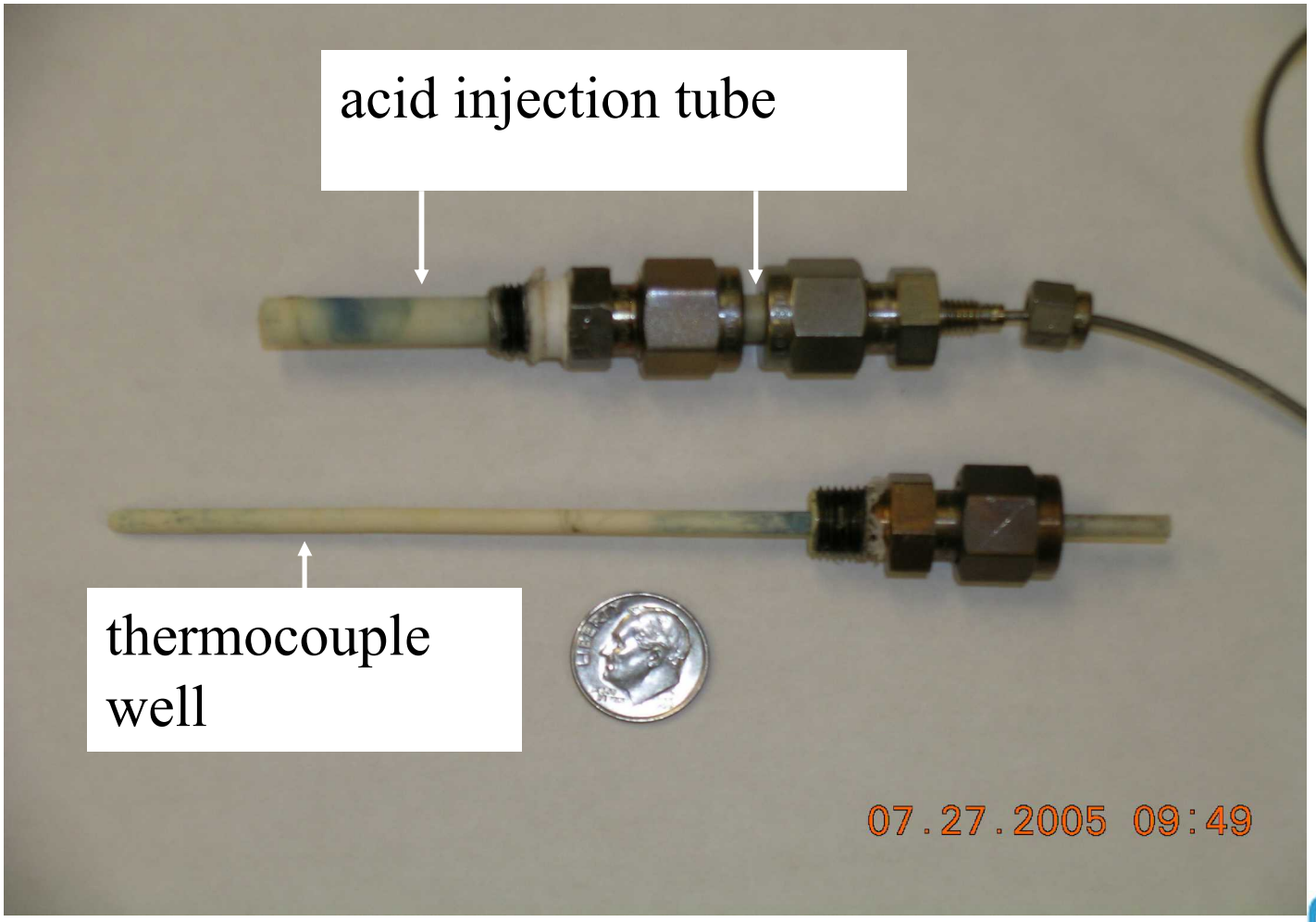
Ceramic Injector and Thermocouple Well



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Platinum Catalyst on Zirconia

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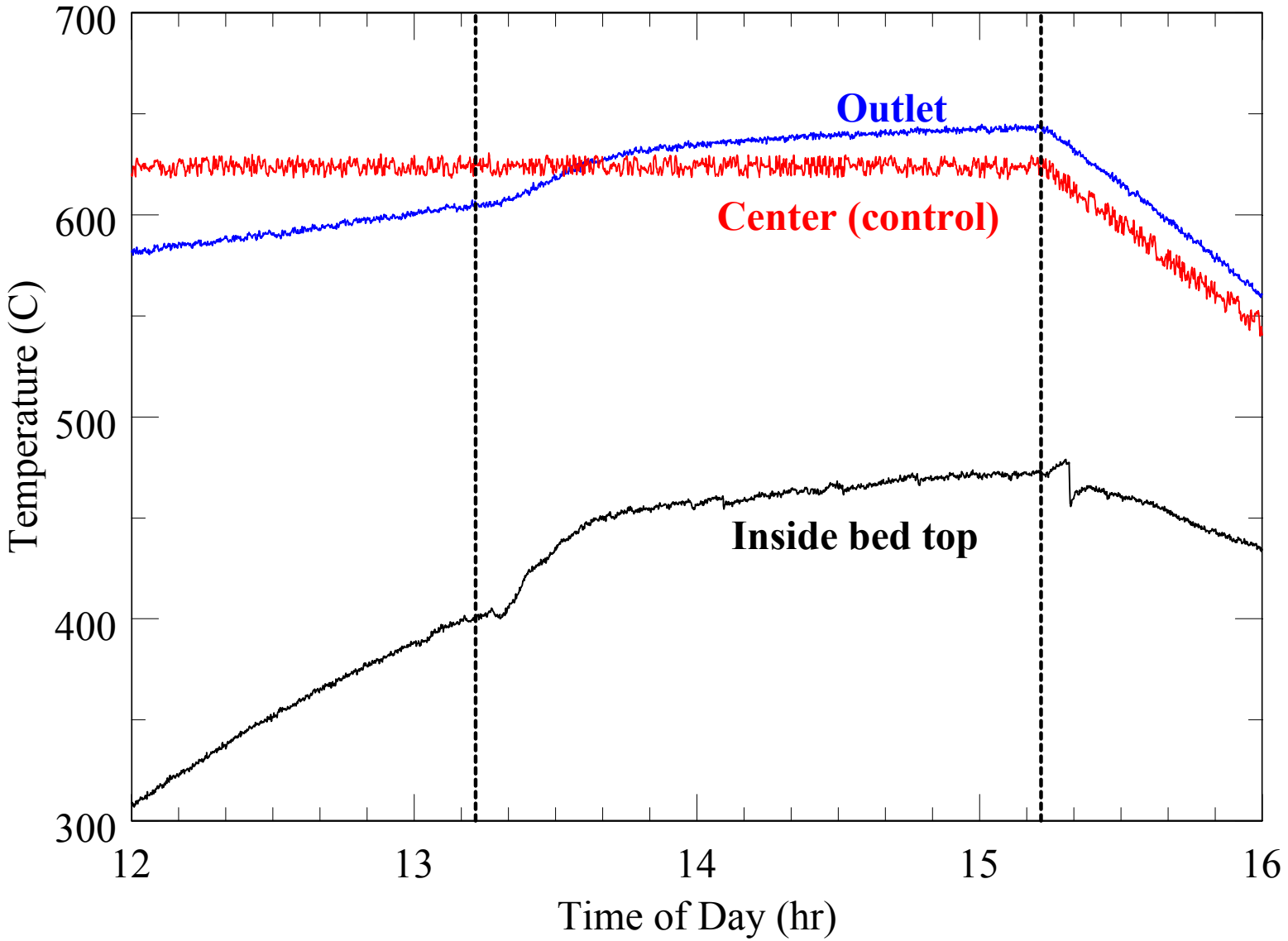
Boiler Temperature Profile



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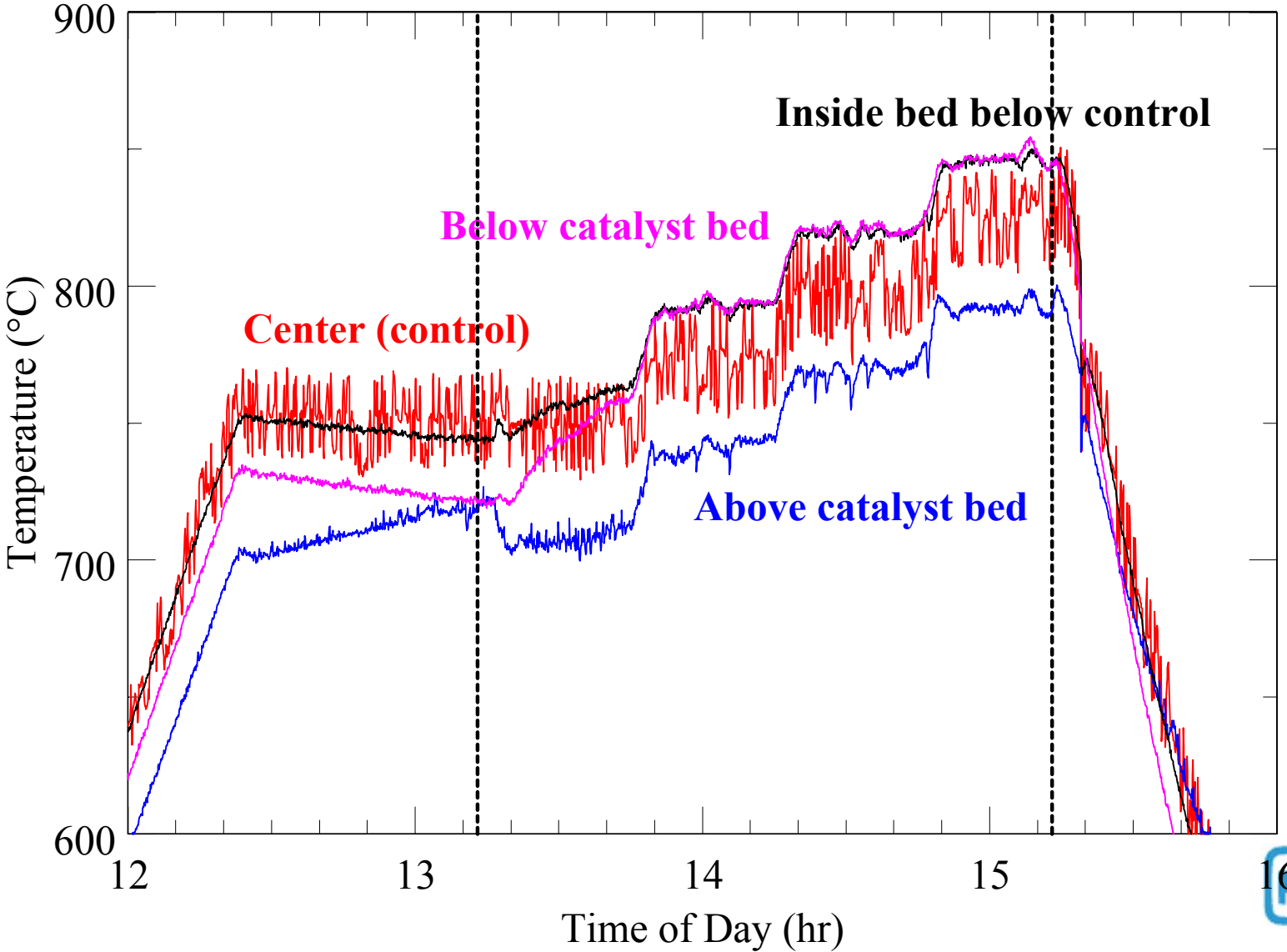
Decomposer Temperature Profile



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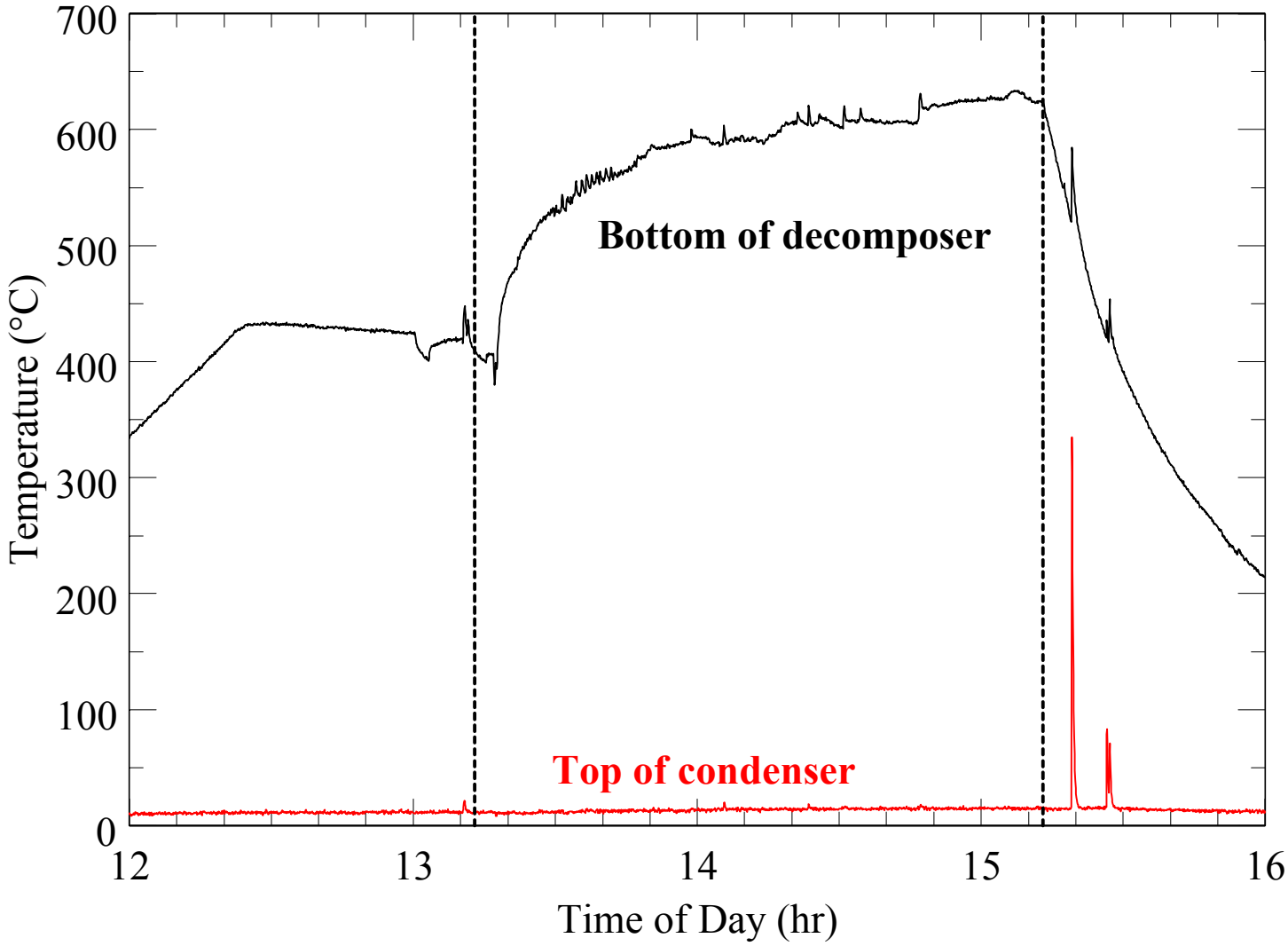
Bottom of Decomposer & Top of Condenser Temperature Profiles



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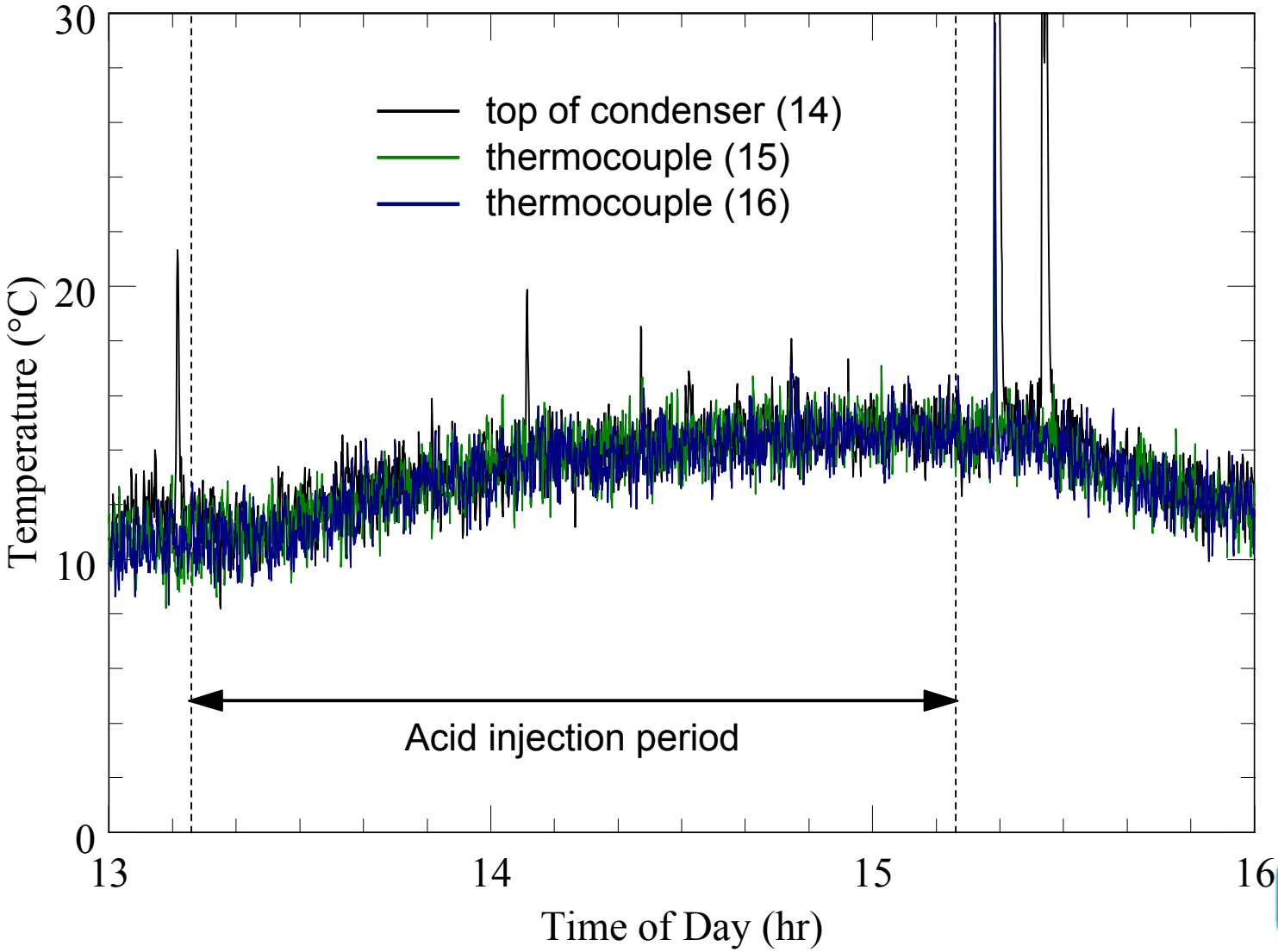
Condenser Temperature Profile



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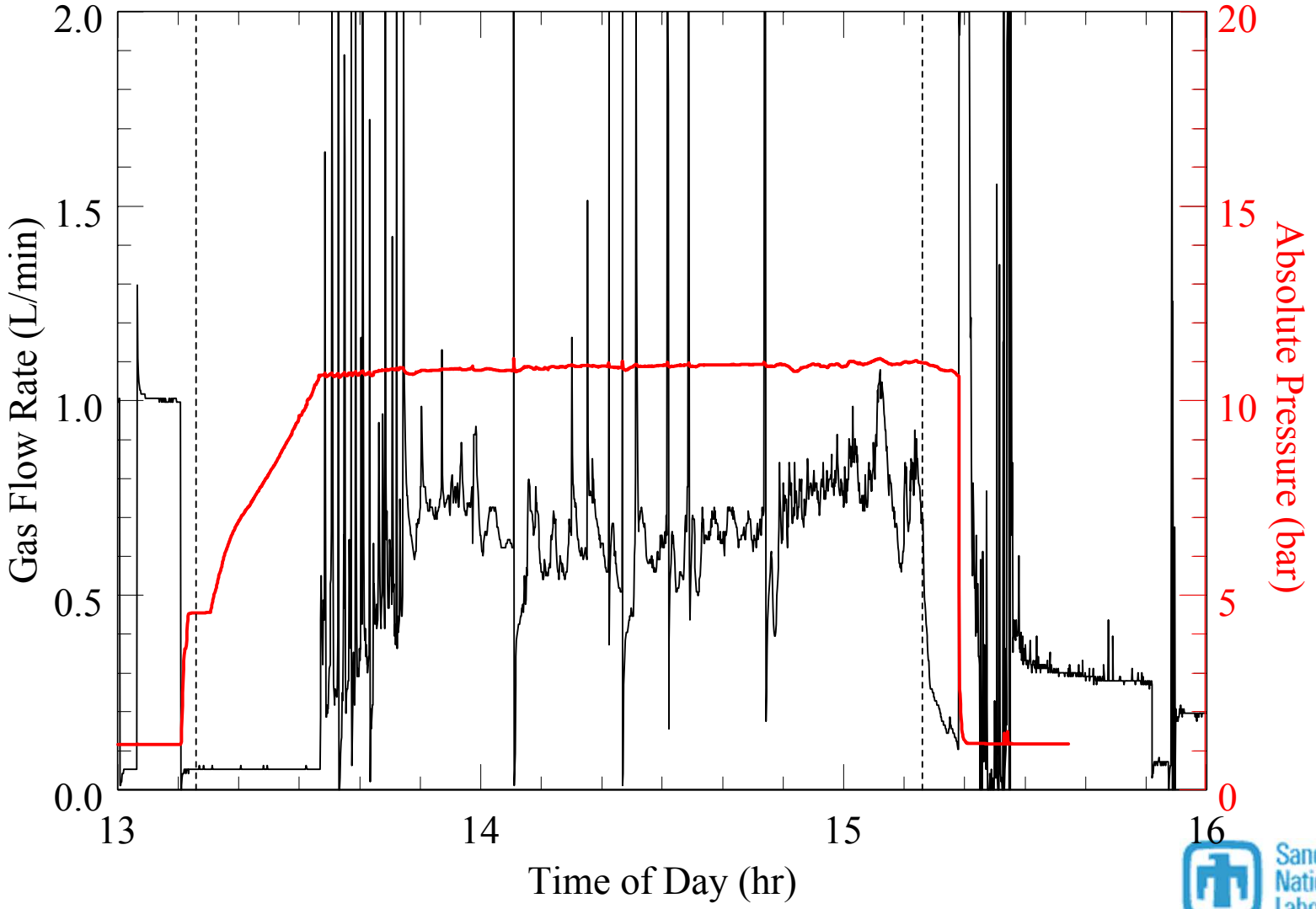
Flow Rate & Pressure



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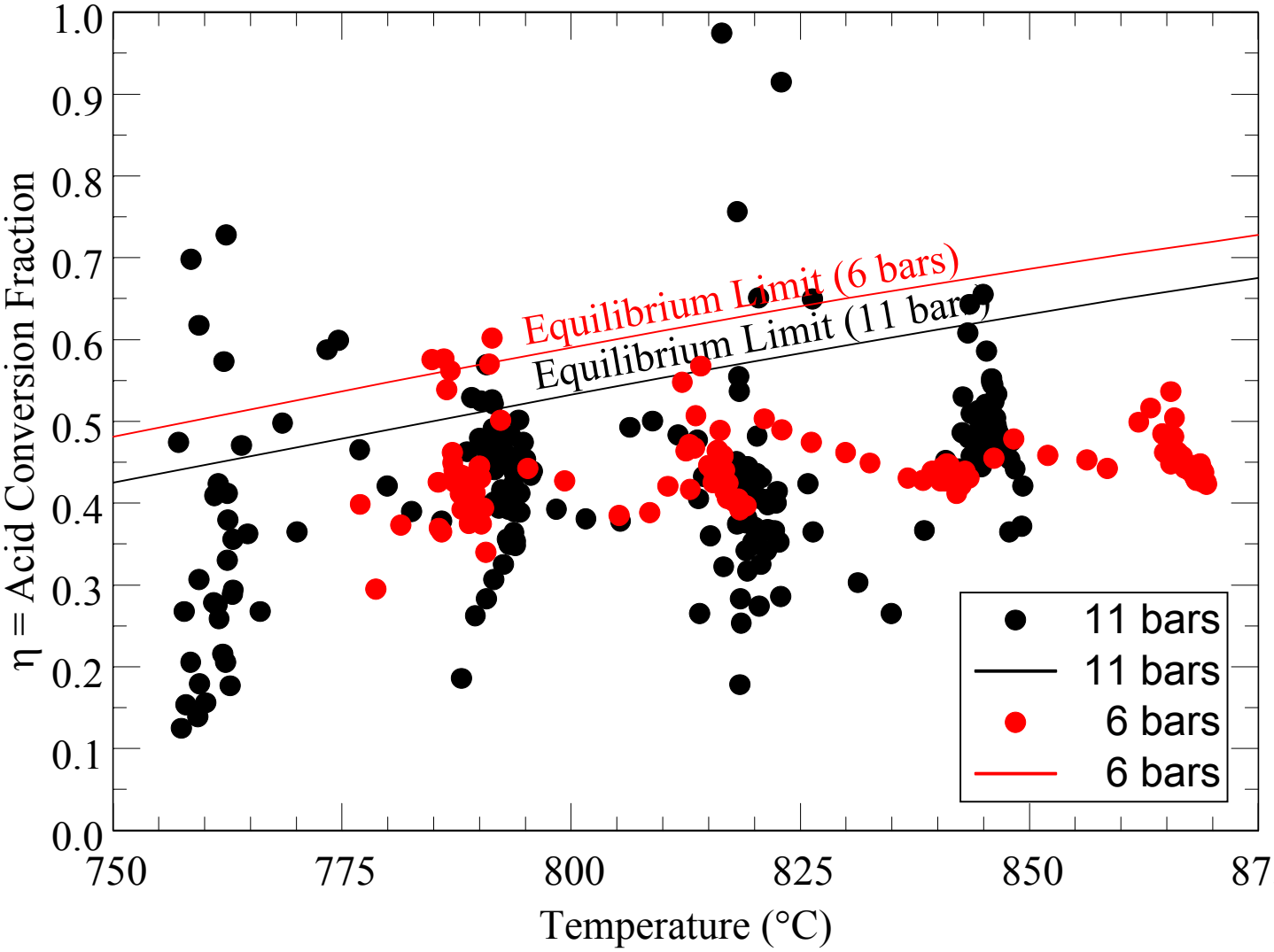
Acid Conversion Fraction



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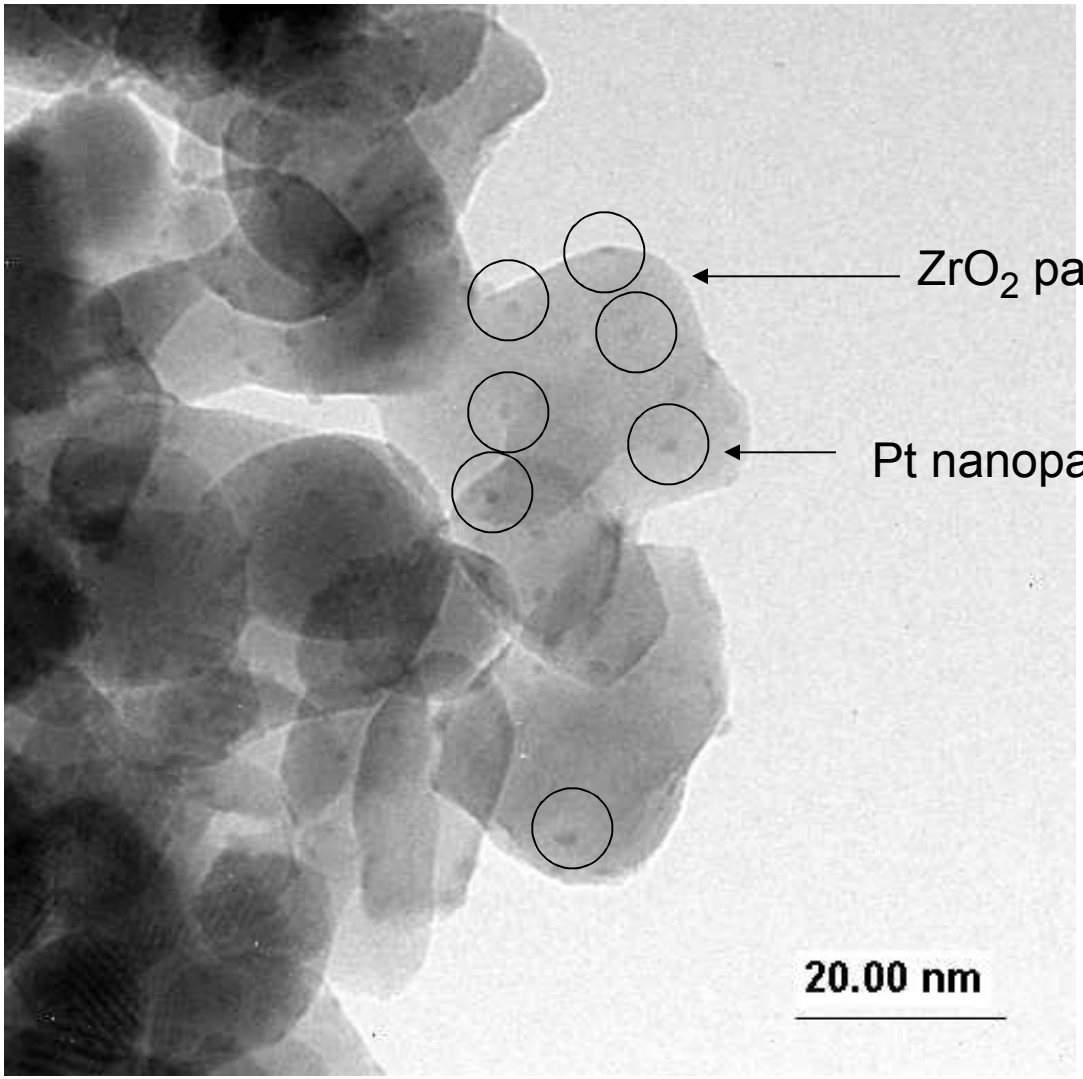
Pretest TEM of Catalyst
(Tom Headley, SNL)



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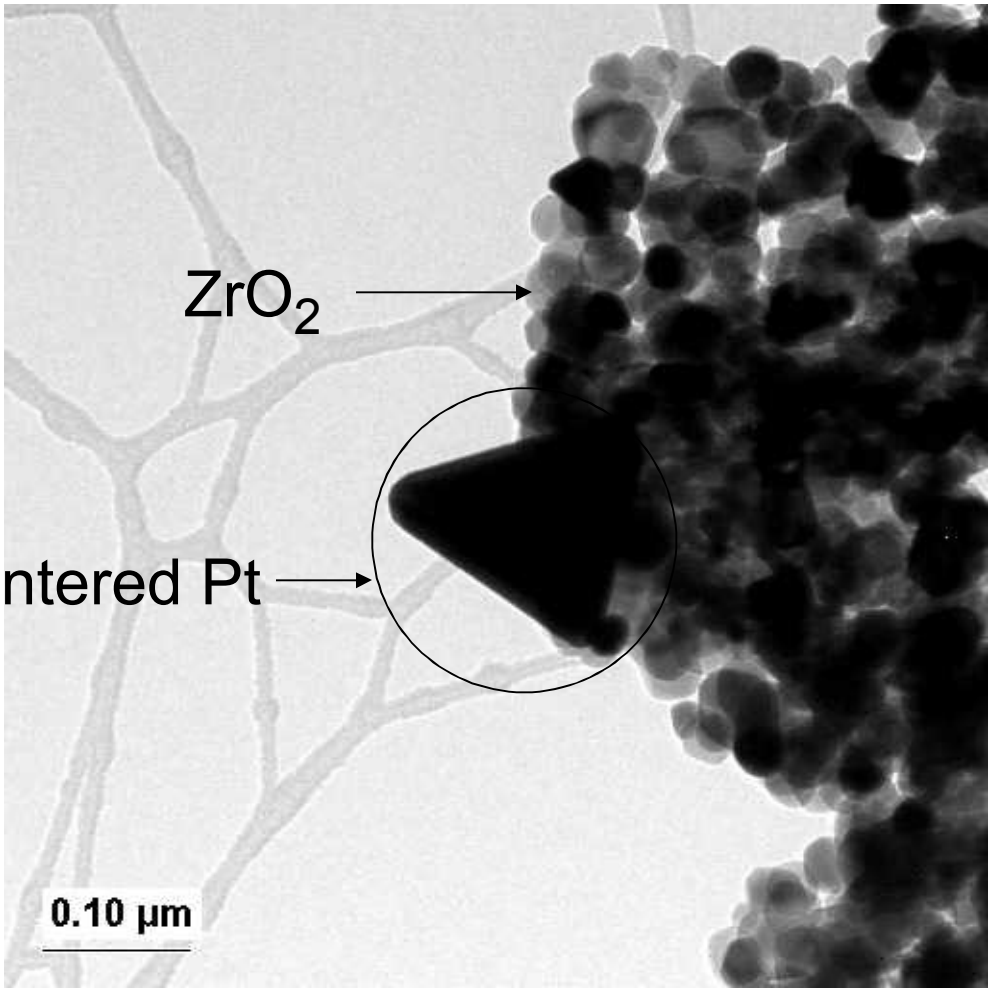
Post test TEM of Catalyst (Tom Headley, SNL)



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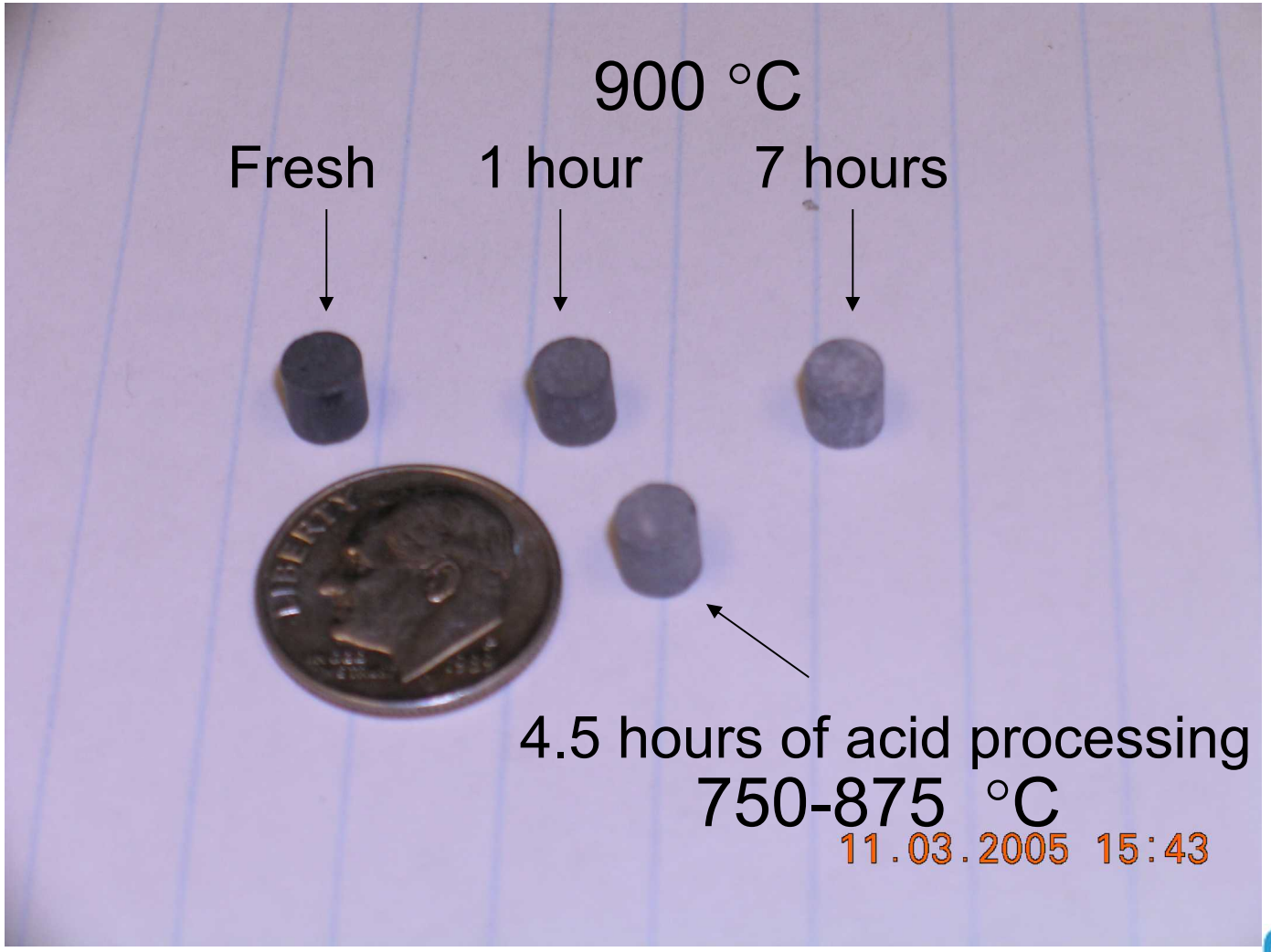
Comparing Catalyst Heated in Air and Used in Acid Decomposition Experiments (Bob Moore, SNL)



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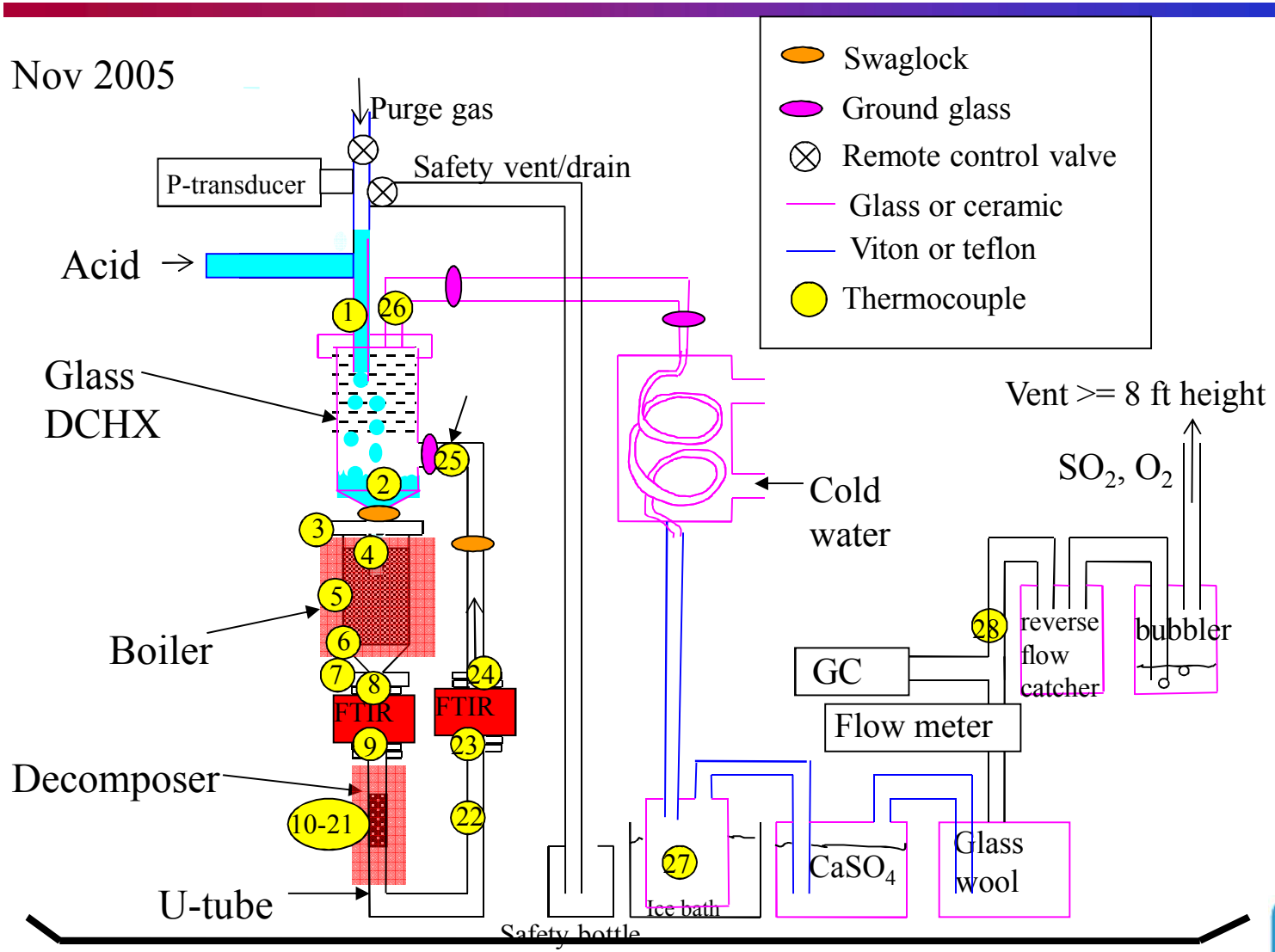
DCHX & FT-IR


Nov 2005
Swaglock
Ground glass
Remote control valve
Glass or ceramic
Viton or teflon
Thermocouple
Purge gas
Safety vent/drain
P-transducer
Acid
Glass DCHX
Boiler
Decomposer
U-tube
Cold water
Vent >= 8 ft height
SO_2, O_2
GC
Flow meter
reverse flow catcher
bubbler
FTIR
FTIR
$CaSO_4$
Glass wool
Ice bath
Safety bottle
Catch-pan
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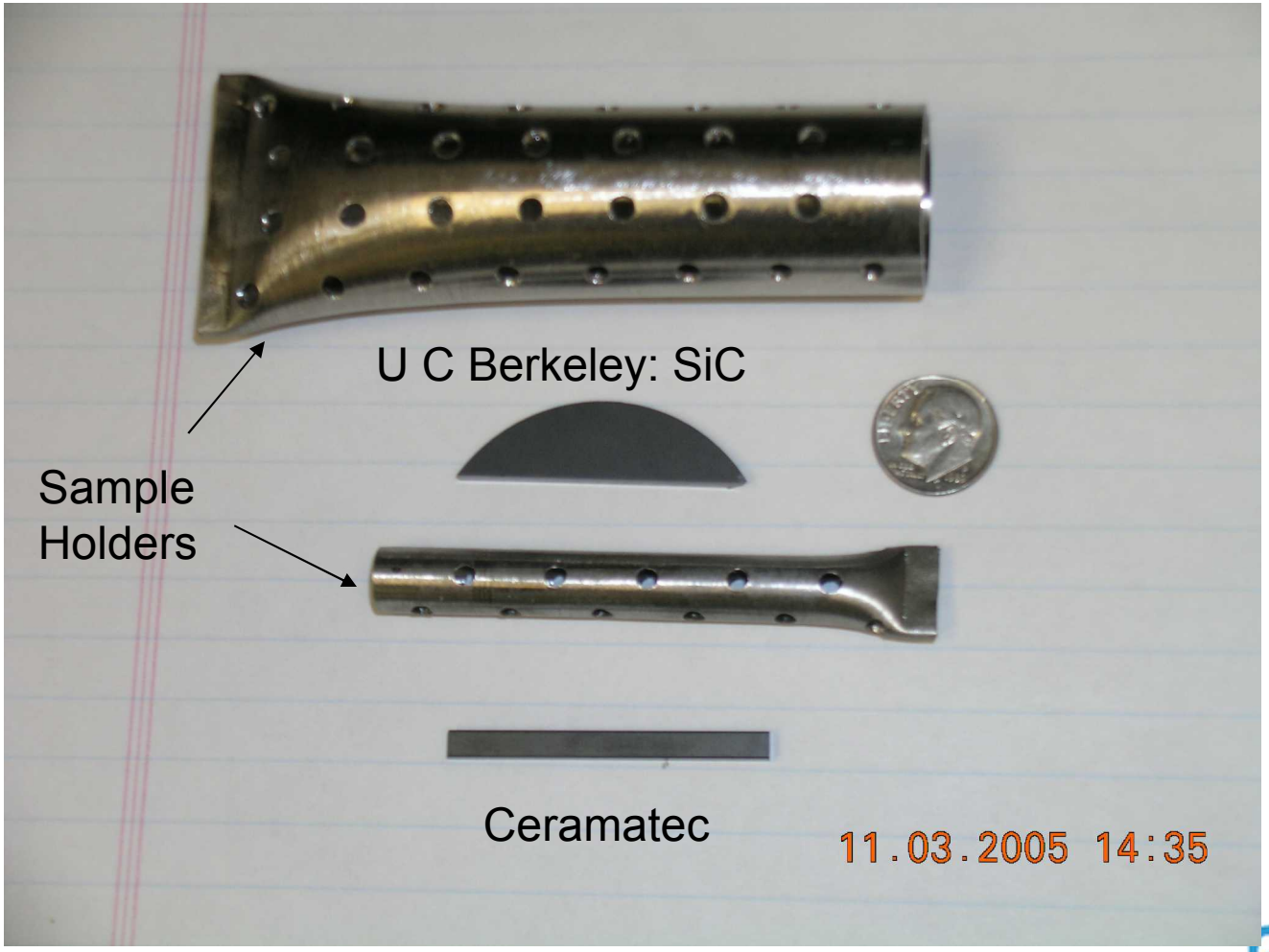
Corrosion Coupon Testing



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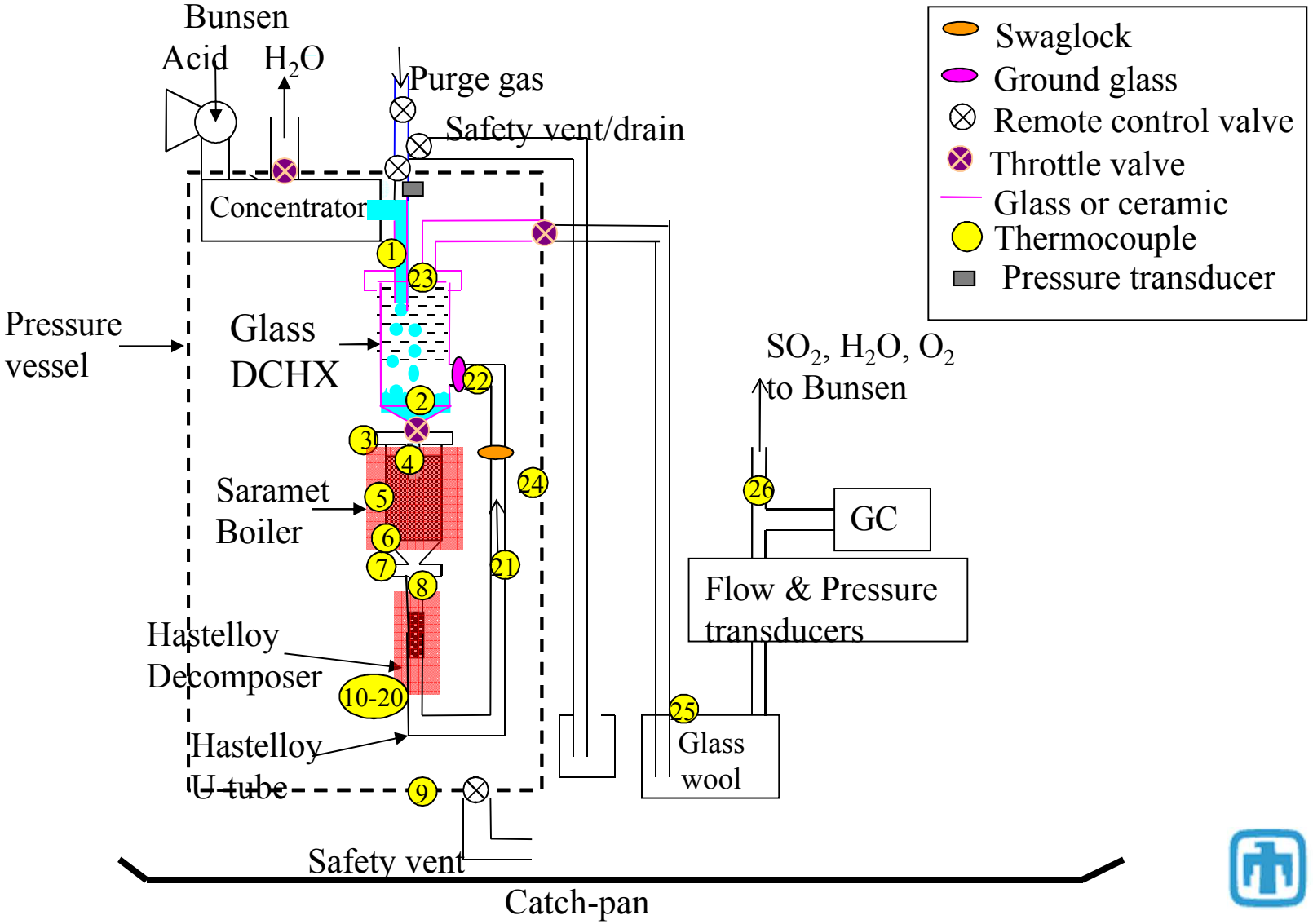
Schematic of Sulfuric Acid Section



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Future Plans Leading to Integrated Lab-Scale Experiments

- Test of adding DCHX and FT-IR (Nov-Dec 2005)
  - Heat and unreacted acid recovery
  - In situ real-time SO3, SO2 and H2O concentrations
  - Coupon corrosion testing under prototypic conditions
- Test of Pressurizing with DCHX (May 2006)
  - 6 bars
  - Assess ceramic replacements for components (boiler and DCHX)
- Test of adding concentrator to apparatus (August 2006)
- Demonstrate full Section 2 for ILS experiments (Dec 2006)

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Summary

- Pressurized lab-scale apparatus fabricated, assembled, tested, and operational
  - Completed first series of pressurized acid tests (2, 6, and 11 bars)
  - Demonstrated real time measurements which are needed for process control
  - Acid conversion fraction 15-20% below theoretical maximum determined by equilibrium
- Plans for FY06
  - Test FT-IR for in situ SO_2, SO_3 and H_2O measurements
  - Test Direct Contact Heat Exchanger to recover heat and recycle undecomposed SO_3
  - Develop and test concentrator that can operate continuously for days/weeks
  - Near completion of full sulfuric acid section including concentrator, DCHX, boiler, decomposer. Prepare for Integrated Lab-Scale operations.