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Broadly Tunable Mid-Infrared Hydrocarbon Sensor

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Outline

- **Summarize project**
- **Summarize barriers, pathways, and metrics**
- **Review progress to date (past year)**
- **Describe future plans (coming year)**
- **Describe commercialization plans**

Broadly Tunable Mid-Infrared Hydrocarbon Sensor (CPS #17141)

VG04-168-2

Goal: To develop a prototype broadly tunable mid-infrared laser spectrometer which can be used in the petrochemical industry for process control (and later for environmental monitoring)

Challenge: Existing gas analyzers don't allow processes to be controlled in real time because gas must be delivered to a remote location; laser-based analyzers can be located close to the process stream, but need a wide tuning range to detect multiple hydrocarbon species

Benefits: Greater efficiency of hydrocarbon cracking process, leading to reduced energy consumption during manufacturing and lower production costs; potential US energy savings of 5 trillion BTU per year if fully implemented

FY05 Activities: Finish optimizing a key optical component of gas analyzer; design and build portable prototype gas analyzer; test gas analyzer on calibrated gas mixtures in laboratory, then in a working process stream

Participants: Physical Sciences Inc., Analytical Specialties, Inc. (ASI), Dow Chemical, U.S. Department of Energy (SBIR program)

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Broadly Tunable Mid-Infrared Hydrocarbon Sensor (CPS #17141)

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Barrier-Pathway Approach

Barriers	Pathways	Critical Metrics
Inadequate tuning range of commercially-available laser sources	Produce custom-made laser source with a wider tuning range	Tuning range >200 cm^{-1} (achieved in Phase I)
Slow response time of existing gas analyzers (remote location)	Perform measurement near the process stream	Portable instrument capable of measuring at least three hydro-carbons to a precision of ~1% with a response time of a few seconds

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Phase II Deliverable: Conceptual Design

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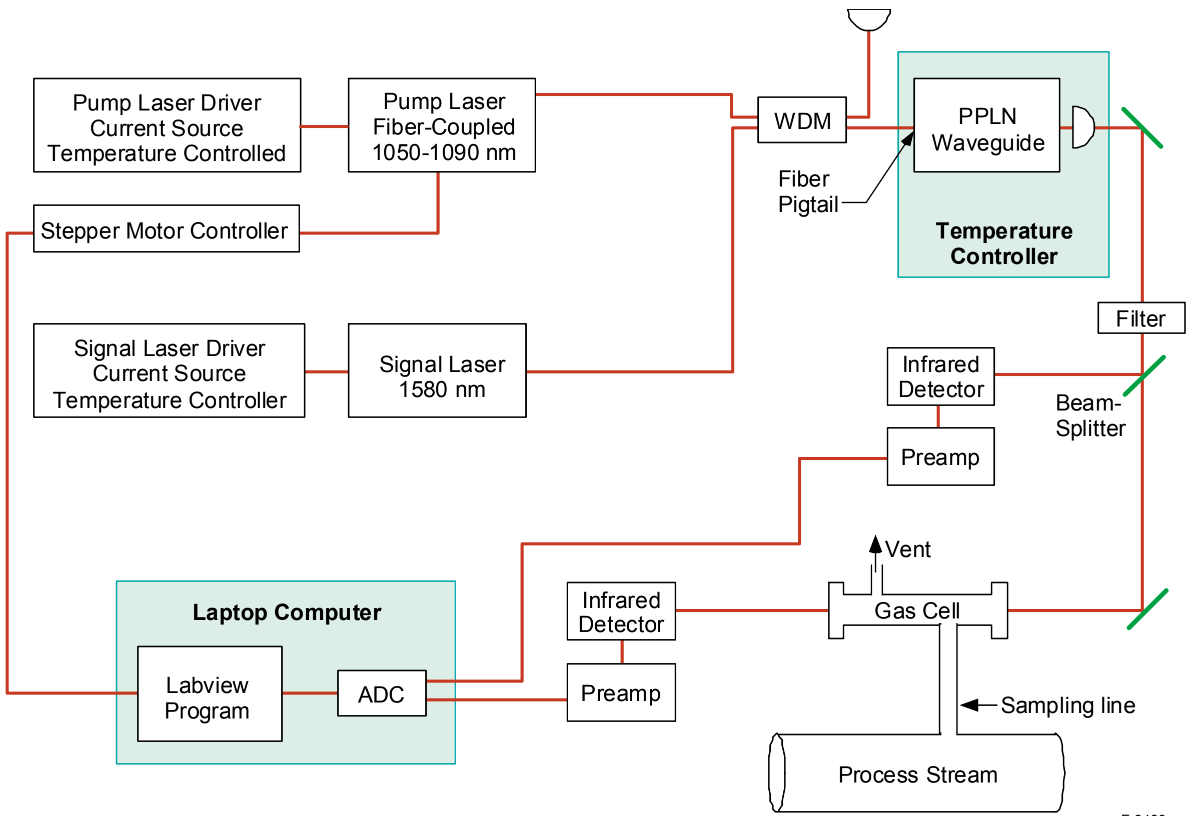
- **Broadly tunable mid-IR gas analyzer based on frequency mixing of near-infrared semiconductor lasers**
- **Key optical component is the optical waveguide used for frequency mixing**



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Project Tasks

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1. **Design, fabricate optimized waveguides for efficient MIR generation**
2. **Design and fabricate portable prototype gas analyzer**
3. **Demonstrate speciation of calibrated gas mixtures in the laboratory**
4. **Demonstrate speciation on process gases in a petrochemical facility**

Task 1: Importance of Efficient Waveguides

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- **Assume 5 mW pump power, 10 mW signal power can be coupled into waveguide**

- **Calculate MIR output power as a function of device efficiency**

Scenario	η_{dev} (%/W)	Output Power (μW)
Pessimistic (Phase I result)	1.0	0.5
Realistic (Phase II improvements)	5.0	2.5
Optimistic (theoretically possible)	50	25

- **Calculations (presented below) and past experience suggest that MIR powers ≥ 1 μW are needed**

- **Serious effort to increase η_{dev} to 5%/W is justified**

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Progress to Date (Task 1)

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1. Design, fabricate optimized waveguides for efficient MIR generation

- improved computer model of waveguide fabrication process developed and tested; will speed optimization because computer experiments are faster than real ones; first generation of waveguides designed
- new photolithographic masks (to define the shape of the waveguides and ensure phase-matching in the nonlinear optical process) designed and fabricated
- waveguide mask includes multiple designs of the “taper” so that the optimum taper design can be found experimentally
- one set of waveguide chips has been patterned in the cleanroom, is awaiting annealed proton exchange (APE) in our laboratory
- testbed for waveguide characterization modified to accommodate the the change from 810/1070 nm mixing to 1070/1580 nm mixing
- computer-controlled DAQ system for waveguide characterization developed

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Task 1 Progress: Computer Model of APE

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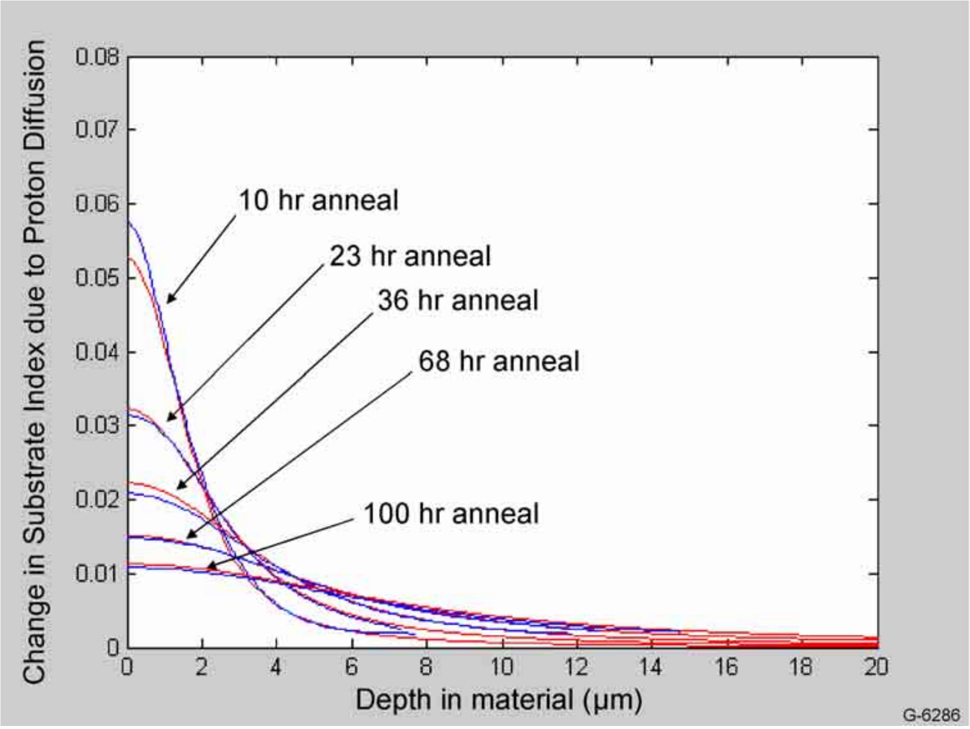
- **Index of refraction at 632.8 nm calculated as a function of depth for planar waveguide, compared with experimental measurement**



- **Good agreement between calculation and experiment; computer model can be used to design waveguides with confidence**

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Progress to Date (Tasks 2-4)

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2. Design and fabricate portable prototype gas analyzer

- hardware for fiber "pigtailing" (permanently attaching fiber to waveguide) identified
- signal-to-noise calculations performed to guide the choice of infrared detectors and detection schemes
- hardware and software for rugged, portable DAQ system identified
- typical process gas conditions (composition, temperature, pressure) at the measurement location specified, measurement requirements (speed and precision) defined

3. Demonstrate speciation of calibrated gas mixtures in the laboratory

- propylene identified as a key target species (along with ethylene, methane); need to measure methane/propylene and propylene/ethylene ratios

4. Demonstrate speciation on process gases in a petrochemical facility

- site visit to petrochemical facility conducted, measurement location identified

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Task 2 Progress: Signal to Noise Calculation

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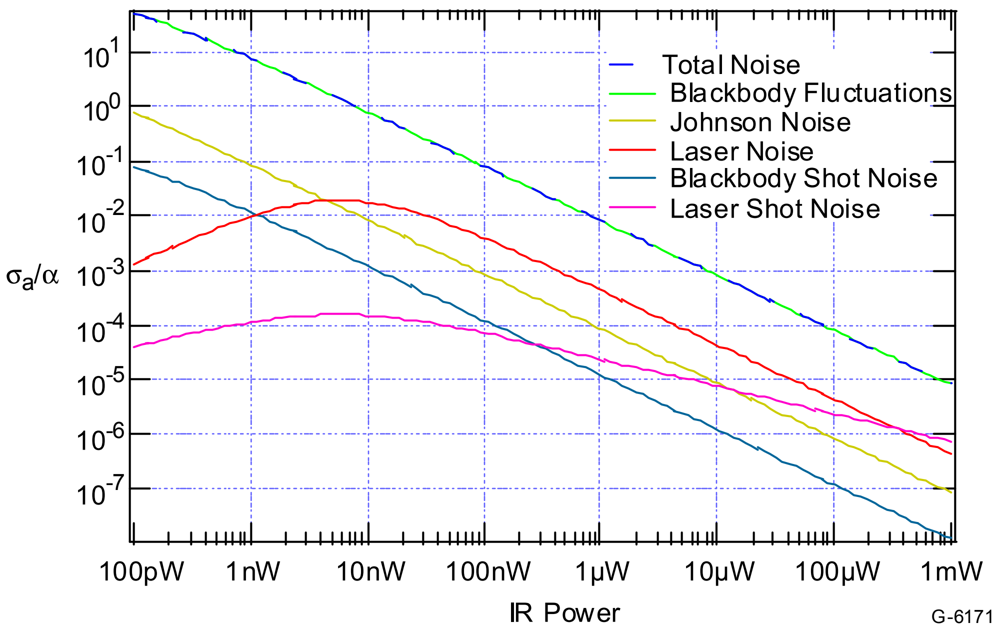

- **Concentration measurement error plotted as a function of laser power for an assumed set of operating conditions**
- **Adequate measurement precision predicted for laser powers ≥1 µW**

- **$\alpha = 1$**
- **T_{det} = -90°C**
- **γ_{back} = 0.1%**
- **γ_{laser} = 1 %**
- **$\sigma_{ex} = 0$**
- **$I_{bias} = 0$**
- **Δf = 500 Hz**
  - (500 points; 1 s acquisition time)
- **λ = 3.3 µm**
- **$A_{det} = 4.91 \times 10^{-8}$ m^2**
  - (250 µm diameter)
- **$\tau_{sam} = 0.7$**
- **$\tau_{ref} = 0.85$**
- **$\tau_{BS} = 0.7$**

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Future Plans

1. **Design, fabricate optimized waveguides for efficient MIR generation**
   - fabricate, test channel waveguides
   - iterate design, fabrication, and testing to improve device efficiency

2. **Design and fabricate portable prototype gas analyzer**
   - review ruggedness requirements, design gas analyzer accordingly
   - purchase components, fabricate gas analyzer

3. **Demonstrate speciation of calibrated gas mixtures in the laboratory**
   - design, fabricate temperature-controlled, short path length gas cell
   - measure MIR spectra of three target species, develop detection strategy
   - purchase calibrated gas mixtures, measure species concentrations

4. **Demonstrate speciation on process gases in a petrochemical facility**
   - install prototype near process stream at petrochemical facility
   - measure species concentrations in process stream

Commercialization Strategy

- **PSI will work with ASI to develop and test prototype hydrocarbon process analyzers**
- **ASI's principal business is specialized analyzers for the US petrochemical industry**
- **ASI provides unique access to important, unmet petrochemical analyzer needs**
  - process definition
  - sensor specifications, packaging, environmental requirements
  - access for prototype testing in Phase II at Dow Chemical
- **Dow is is the major end-user of process control instruments developed by ASI, will provide access to facilities during Phase II**

Summary and Conclusions

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- Difference-frequency generation with tuning range of 250 cm^{-1} in 3.4-micron wavelength region demonstrated in waveguides with proper choice of pump, signal wavelengths
- Tunability can be exploited to perform speciation on hydrocarbon mixtures, with potential use in petrochemical manufacturing
- Phase II program is designed to produce a portable prototype gas analyzer
- First year of Phase II program has laid the foundation for prototype construction: progress on designing more efficient waveguides, defining application requirements, developing packaging strategies, attracting commercial interest
- Prototype will be fabricated and tested in second year of Phase II program

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