Enabling Chip-Scale Trace-Gas Sensing Systems With Silicon Photonics

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Outline

- **Oil and Gas Industry use case for innovative trace gas sensors and sensor networks**
- **Evanescent field waveguide spectroscopic sensor design**
- **Spectral extraction, noise analysis, and long-term stability**
- **Integration of an on-chip reference cell, III-V / Si hybrid laser, and III-V photodetector**
- **Fugitive methane management solution early field test results**
- **Outlook**

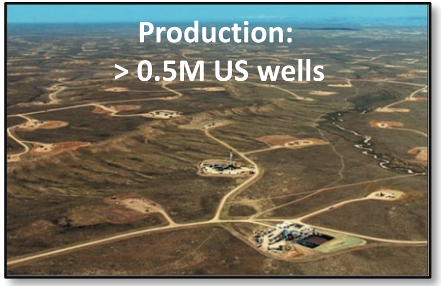
Why Manage Methane Emissions?

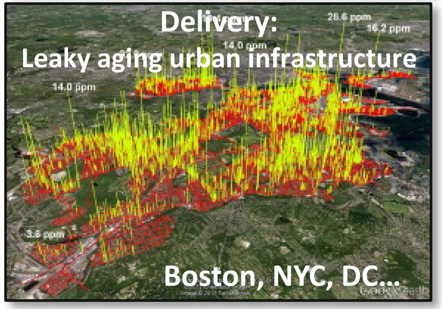
Natural gas is considered as a source of clean energy:

- Compared with coal, burning natural gas produces ½ as much CO_2 per unit of energy generated
- "Bridge fuel" for lowering emissions while transitioning from fossil fuels to renewable energy sources
- ***But….***

Leaking more than ~2-3% of natural gas produced, processed, stored, and delivered would negate its greenhouse gas advantage:

- ***Various estimates place leakage rate at 1.6%-10% of total production! (depending upon location/study)***
  - *D.T. Allen et al., PNAS 2013; A. R. Brandt et al., Science 2014; Inventory of U.S. Greenhouse Gas Emissions and Sinks, U.S. EPA.*





N. Phillips et al., Env. Pol. 2013

Fugitive emissions can eliminate advantage over burning coal

Urban safety implications

http://www.huffingtonpost.com/2015/03/26/east-village-explosion_n_6950116.html
http://edition.cnn.com/2014/03/15/us/aging-gas-infrastructure/

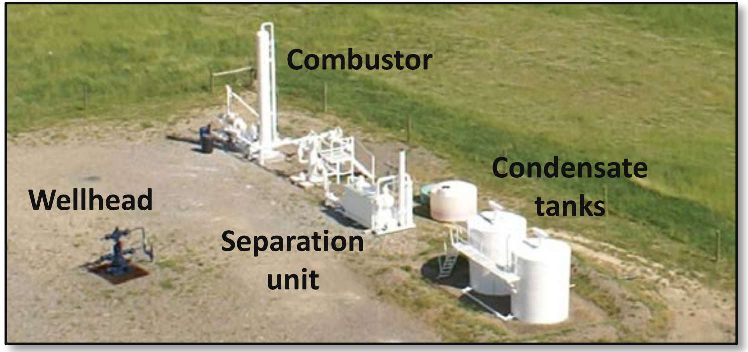
Fugitive Methane Emissions in Natural Gas Processing

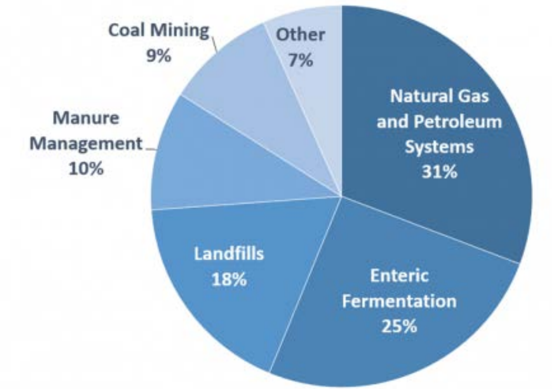
Methane (CH_4) is the second largest contributor to global warming after CO_2:

- Global warming potential of CH_4 is ~20-35 × greater than CO_2
  - *Alvarez et. al., Proc. Nat. Acad. Sci., 109 (17), pp. 6435-6440, (2012).*
- 10%-30% of global warming impact from human activity

> 0.5 Million active oil and gas wells in the U.S.:

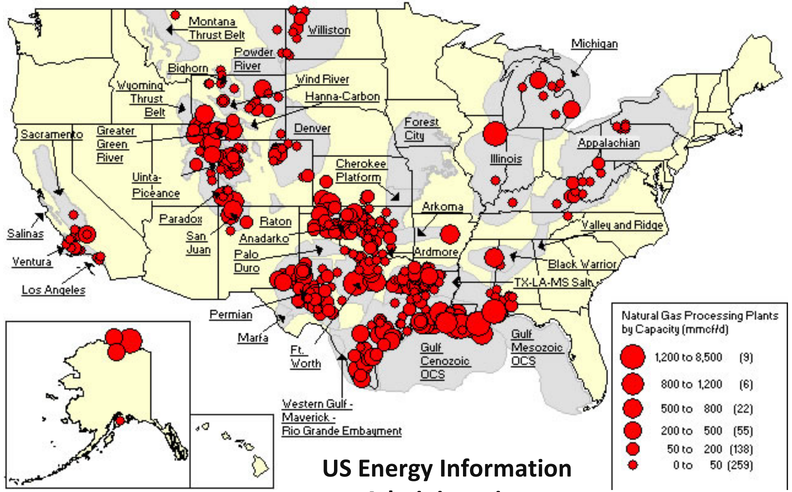
- ~30% of U.S. anthropogenic methane emissions





U.S. Methane Emissions By Source

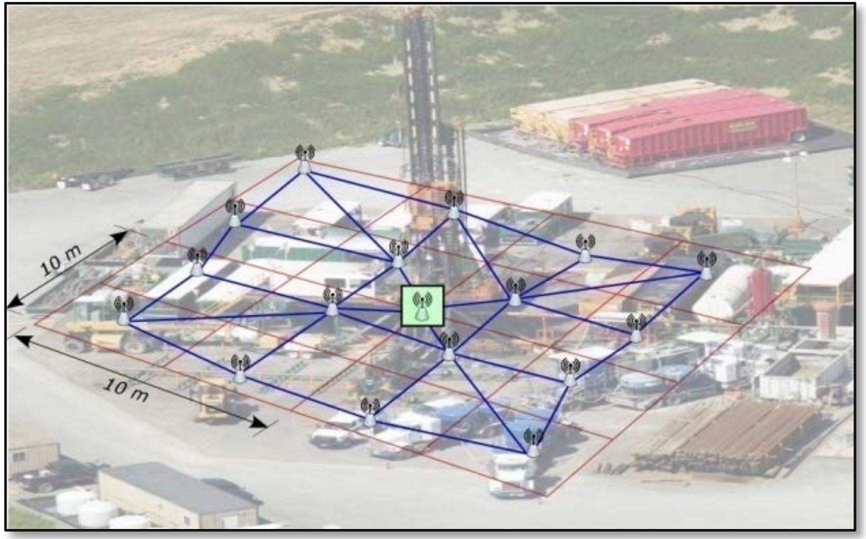
U.S. EPA (2017). Inventory of U.S. Greenhouse Gas Emissions and Sinks: 1990-2015.



US Energy Information Administration

Use Case for Innovative Sensor Networks

An Intelligent Multi-Modal Methane Measurement System (AIMS)



Technological driver: ARPA-E MONITOR Program

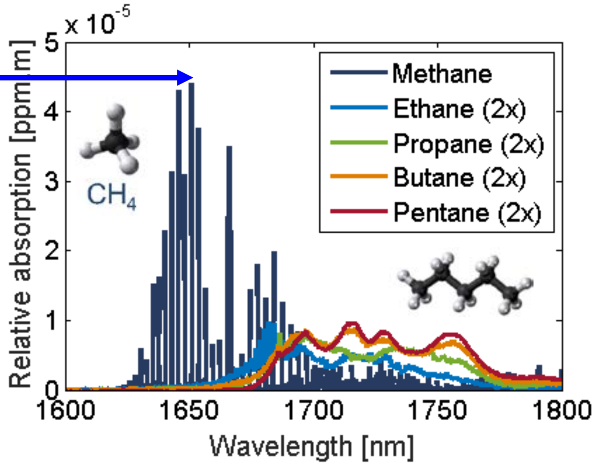
- Cost-effective sensor network enabling continuous monitoring for CH_4 leak detection, localization, and repair
- *No viable technology today: Alignment of performance with required cost point is very challenging with today's technology*

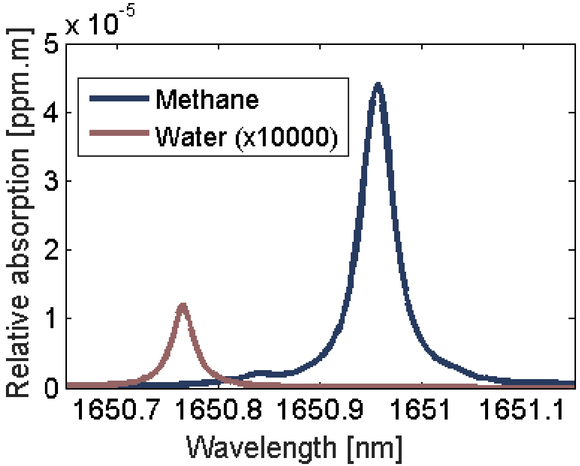
Opportunity – Apply Physical Analytics / IoT Solutions to:

- Significantly reduce fugitive CH_4 emissions across the oil and gas industry
- Improve production efficiency and safety, reduce cost
- Comply with emissions regulations
- *Harness the full potential of natural gas as a clean fuel*

Achieving Molecular Selectivity with Optical Spectroscopy

CH_4 R(4) line in the in the $2\nu_3$ overtone band





Typical composition of natural gas

naturalgas.org

Methane	CH_4	70-90%
Ethane	C_2H_6	0-20%
Propane	C_3H_8	
Butane	C_4H_{10}	
Carbon Dioxide	CO_2	0-8%
Oxygen	O_2	0-0.2%
Nitrogen	N_2	0-5%
Hydrogen sulphide	H_2S	0-5%
Rare gases	A, He, Ne, Xe	trace

Chemi-resistive VOC sensors offer sensitivity, low cost, low power, *but*:

- Not selective to only CH_4 - other VOCs, humidity, etc.
- Can produce false positives

Optical spectroscopy near 1651 nm *uniquely* identifies CH_4:

- Low overlap with constituents of natural gas
- Virtually no cross sensitivity to water

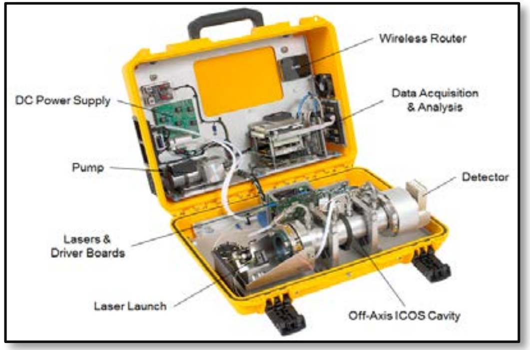
Sensitivity, Size, Power Consumption, ***COST***… Today's Commercial Sensors Don't Meet Needs

What makes spectroscopic sensors so expensive?
- Precision instruments ~ppb sensitivity require by-instrument calibrations
- Optical multi-pass cells, ring-down cavities, off-axis cavities, AR coatings
- Active optical alignment
- Low as-manufactured laser wavelength yield
- Thermal control and stabilization
- Use of expensive mid-IR lasers and/or image sensors (some)

Technical objective then becomes:
- Build a practical instrument, not a scientific instrument
- Don't burn power on active stabilization
- Engineer for high yield, high volume, low maintenance field operation, and **LOW COST**

5000ppm, 23cm, 2.5kg, battery powered

2ppb, 45cm, 15kg, 60W



1ppm, 35cm + external pump, 1.9kg, 2W

2ppm, 27cm, 2.7kg, 5W

Silicon Photonic Optical Trace Gas Sensor: Key Technical Innovations

Solution for deployment of economical, low-power, continuously monitoring sensor networks

IBM technology value proposition:

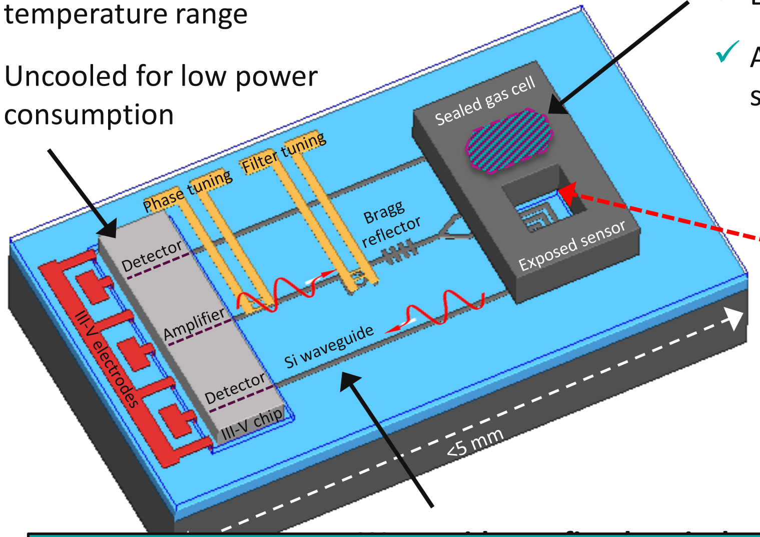
- **Selectivity to molecule of choice**
- **Orders of magnitude lower cost**
  - < $250/sensor (in volume)
- **Low power consumption**
  - < 1 Watt
- **Leverages volume manufacturing**
  - Same infrastructure used to print billions of transistors on a single microprocessor

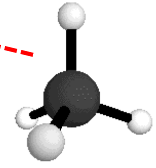
Integrated tunable laser and detector:

- ✓ Operation across wide ambient temperature range
- ✓ Uncooled for low power consumption

On-chip gas reference cell:

- ✓ Built-in self-calibration
- ✓ Autonomous long-term sensor accuracy





Methane molecule

Sensor sensitivity target: ~5-10 ppmv CH_4

Compelling Technological Advantages

	Commercially Available Optical CH_4 Sensors	Integrated SiPh Chip Sensor
Sensitivity	0.1-1 ppmv	**5 ppmv**
Power	2-10 W	**~0.6 W**
Size	~50 cm	**~5 cm**
Weight	3-10 kg	**~200 g**
Cost	$10k-$25k USD	**$0.25k USD**
Figure of Merit **Sens-power-$-size ($ppm^{-1}$/(W.k$.m))**	**~0.5**	**22**

> 40x improvement in Figure of Merit

SiPh technology value proposition:

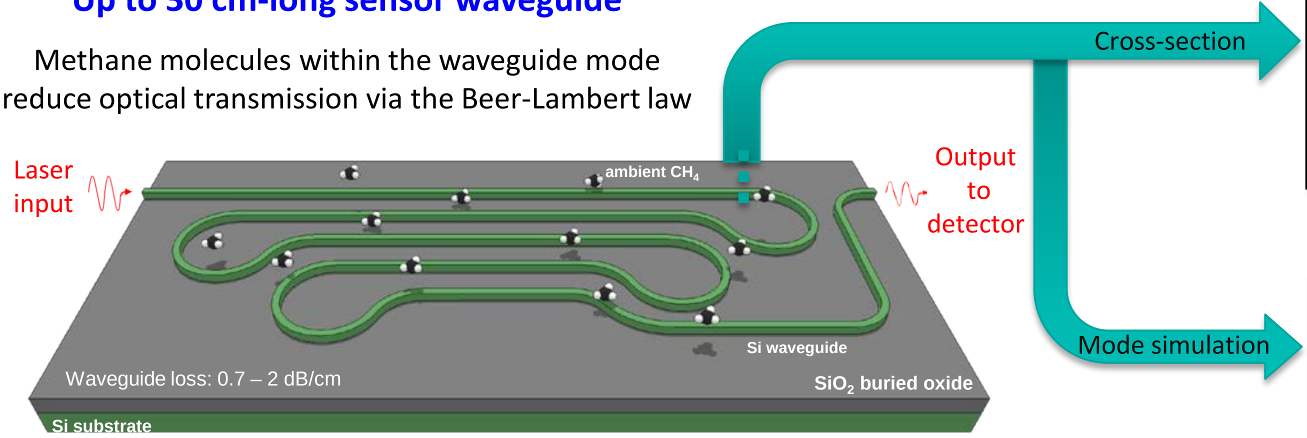
- Orders of magnitude lower cost
- Low power consumption
- Compactness
- Leverages volume manufacturing
- Extensible to a broad range of applications
- ***Facilitates economical, large-scale deployment of continuously monitoring sensor networks***

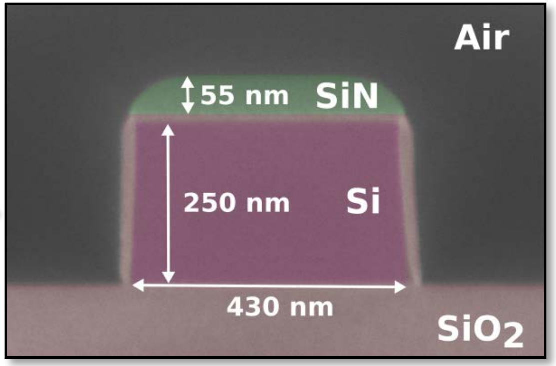
References:
[1] http://www.axetris.com/en-us/lgd/products/lgd-f200/lgd-f200-a-ch4
[2] http://www.tdlsensors.co.uk/products.html
[3] http://www.geotechuk.com/products/landfill-and-biogas/portable-gas-analysers/tdl-500.aspx

Evanescent Field Trace Gas Sensing

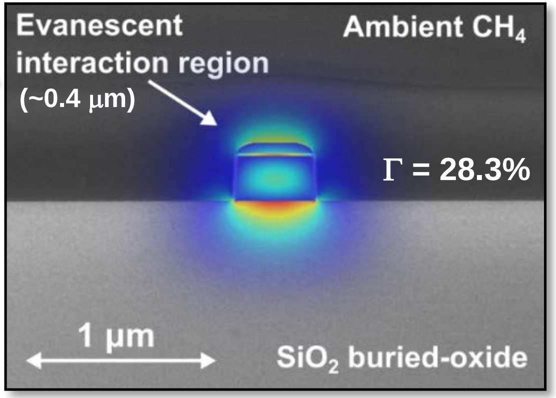
Up to 30 cm-long sensor waveguide

Methane molecules within the waveguide mode reduce optical transmission via the Beer-Lambert law





Waveguide cross-section



TM_{00} electric field profile (E_y)

Direct laser absorption spectroscopy via Beer-Lambert Law

$$I_t = I_0 \exp\left[-\mathrm{L} \cdot \frac{T_R}{T} \cdot p \cdot S \cdot \chi(\nu - \nu_0) \cdot C_r \cdot \Gamma \cdot L_p \right]$$

Concentration: C_r

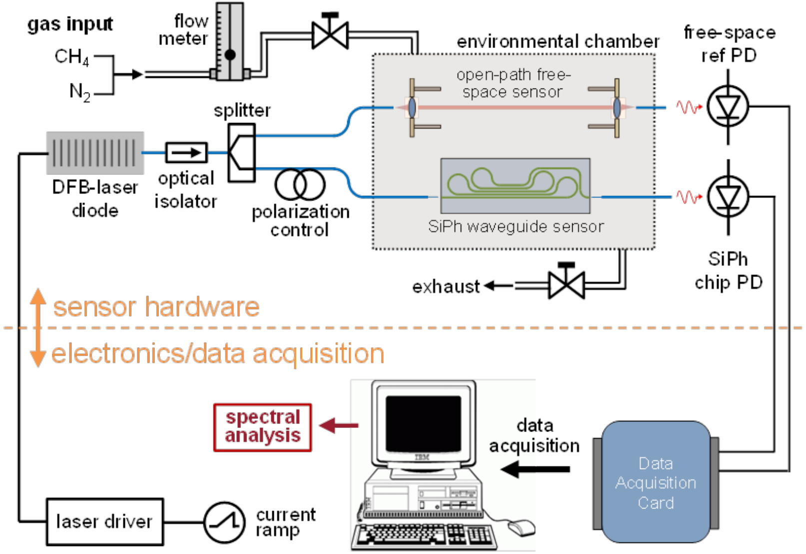
Absorption coefficient: $\mathrm{L} \cdot \frac{T_R}{T} \cdot p \cdot S \cdot \chi(\nu - \nu_0) \cdot C_r$

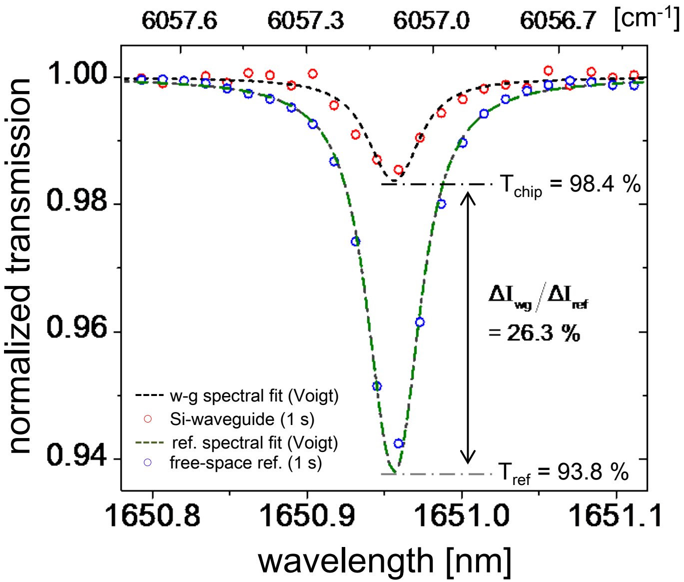
Effective path length: $\Gamma \cdot L_p$

- L - Loschmidt constant
- T_R – reference temperature
- p – partial pressure
- S – integrated linestrength
- χ – lineshape function
- C_r – relative concentration
- Γ – overlap factor
- L_p – physical path length

E. Zhang, L. Tombez, J. S. Orcutt, S. Kamlapurkar, G. Wysocki, W. M. J. Green, CLEO 2016.

Benchtop System for CH_4 Measurements



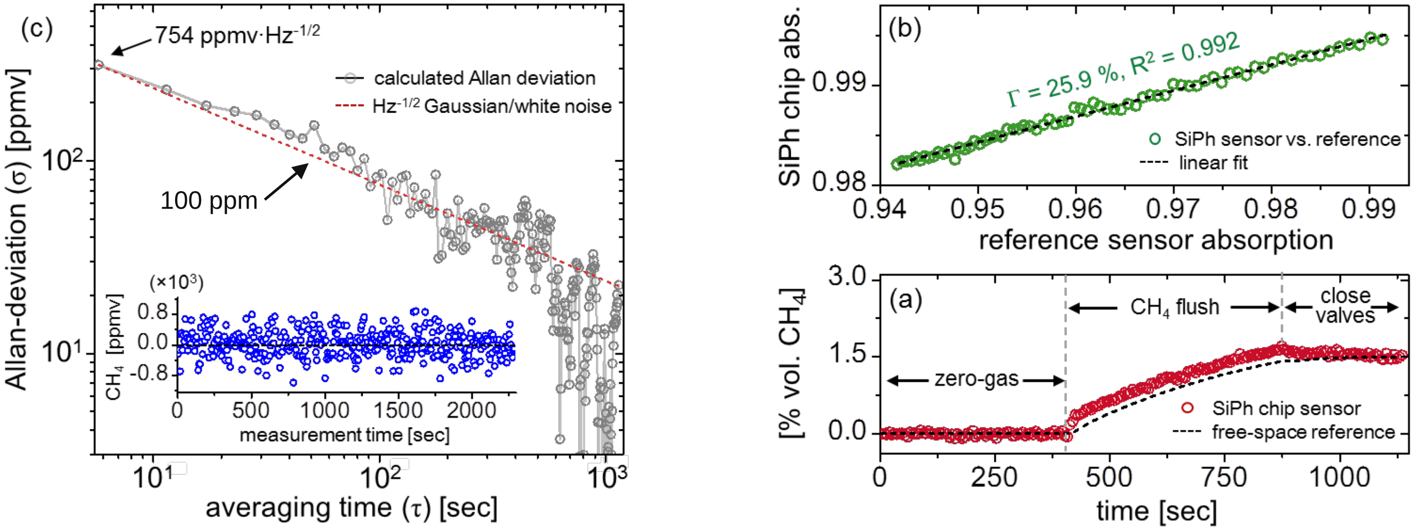


- 1.65 µm DFB laser probing CH_4 in the $2\nu_3$ overtone band
- 100 Hz laser current ramp for direct laser absorption spectroscopy
- Uncooled amplified InGaAs detectors
- 16 kS/sec or 1 MS/sec per channel ADC
- Simultaneous reference/waveguide sensor data acquisition

Comparison of SiPh waveguide and reference CH_4 spectra:

Simulated Γ = 28.3%
Experimental Γ = 26.3%

Sensor Stability and Accuracy Analysis



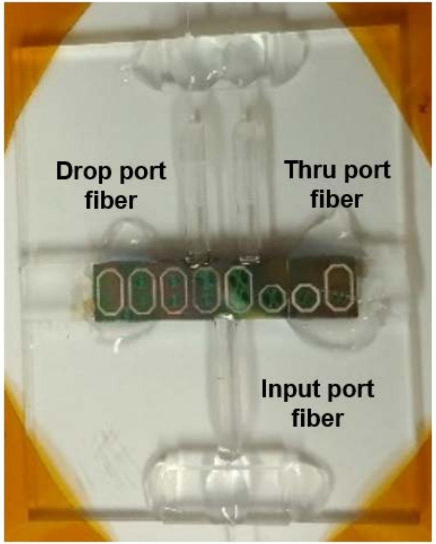
- ~2000 sec. zero-gas stability test with Allan-deviation stability analysis (5 sec. averaging)
- 754 ppmv·Hz$^{-1/2}$ sensitivity with a Dynamic Etalon Fitting algorithm
- White noise-limited performance to ~1000 seconds
- Waveguide sensor noise-equivalent absorption: **$(NEA)_{wg} = 8.4 \times 10^{-4}$ Hz$^{-1/2}$**

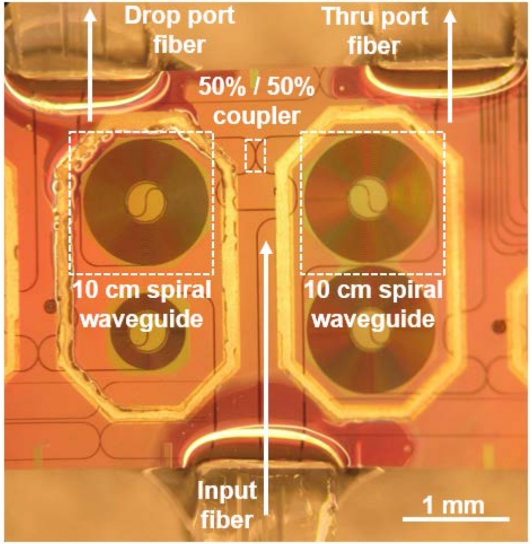
E. Zhang et al., CLEO 2016.

Methane Minimum Detection Limit

Packaging, fabrication, and design:

- Mechanical stability via fiber pigtailing
- Sample both thru port and drop ports simultaneously
- Improved sensitivity expected with next-generation samples:
  - Larger mode overlap, lower propagation/coupling losses

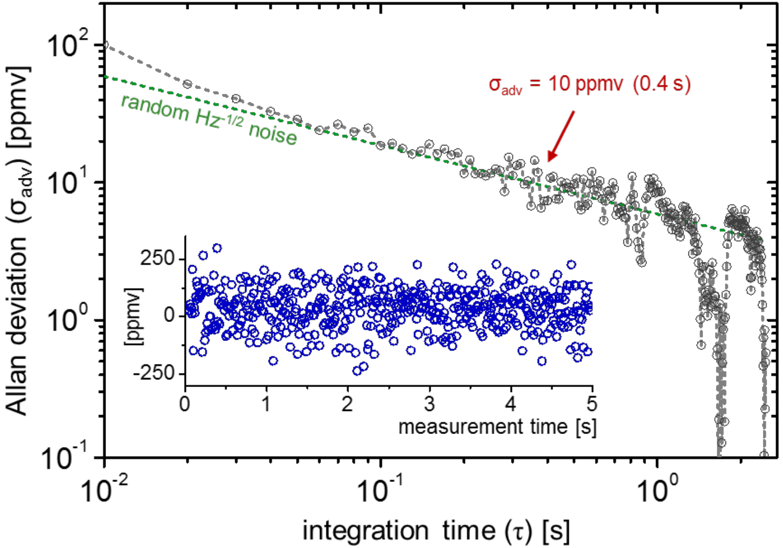




Minimum detection limit

30 cm waveguide, Γ = 25%

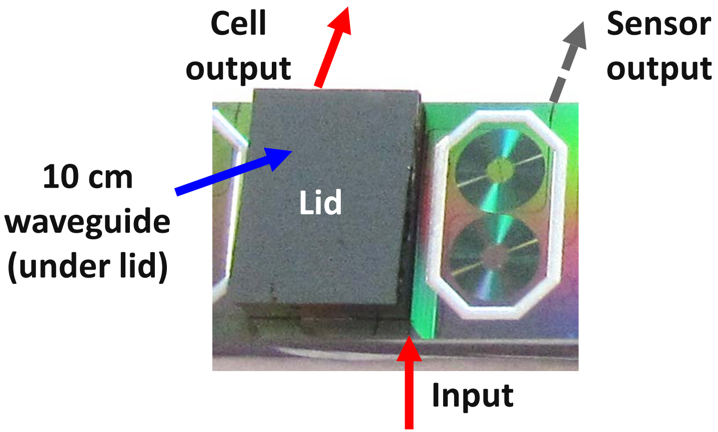
→ 7.5 cm effective path length



C. C. Teng, C. Xiong, E. J. Zhang, Y. Martin, M. Khater, J. Orcutt, W. M. J. Green, Gerard Wysocki, CLEO 2017.
E. J. Zhang et al., unpublished.

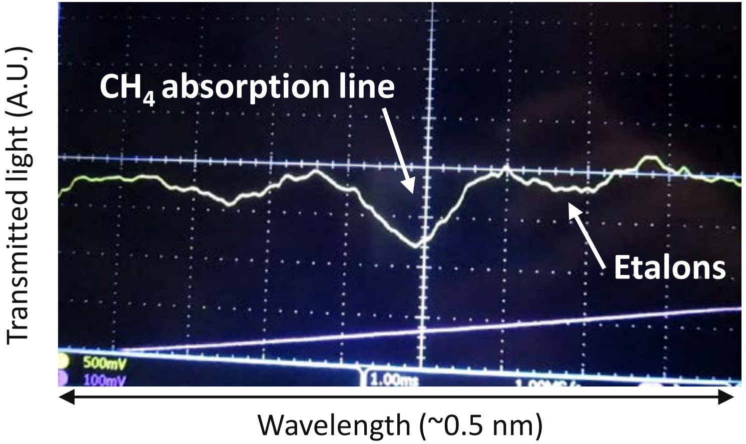
On-Chip Integrated CH_4 Reference Cell

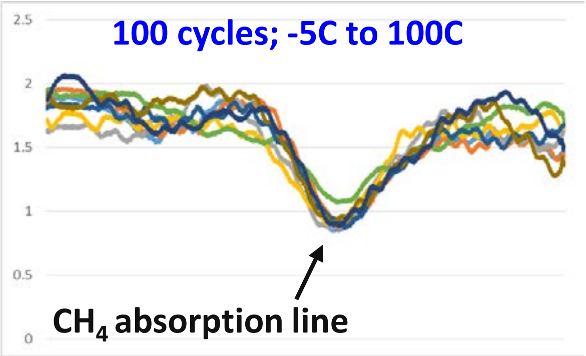
Test configuration



- **Etalons shift with temperature:**
  - Methane absorption line does not
- **Stress testing - cell remains sealed after:**
  - 2 months in ambient lab conditions
  - Thermal cycling; -5C to 100C
  - 20 hours at 107C

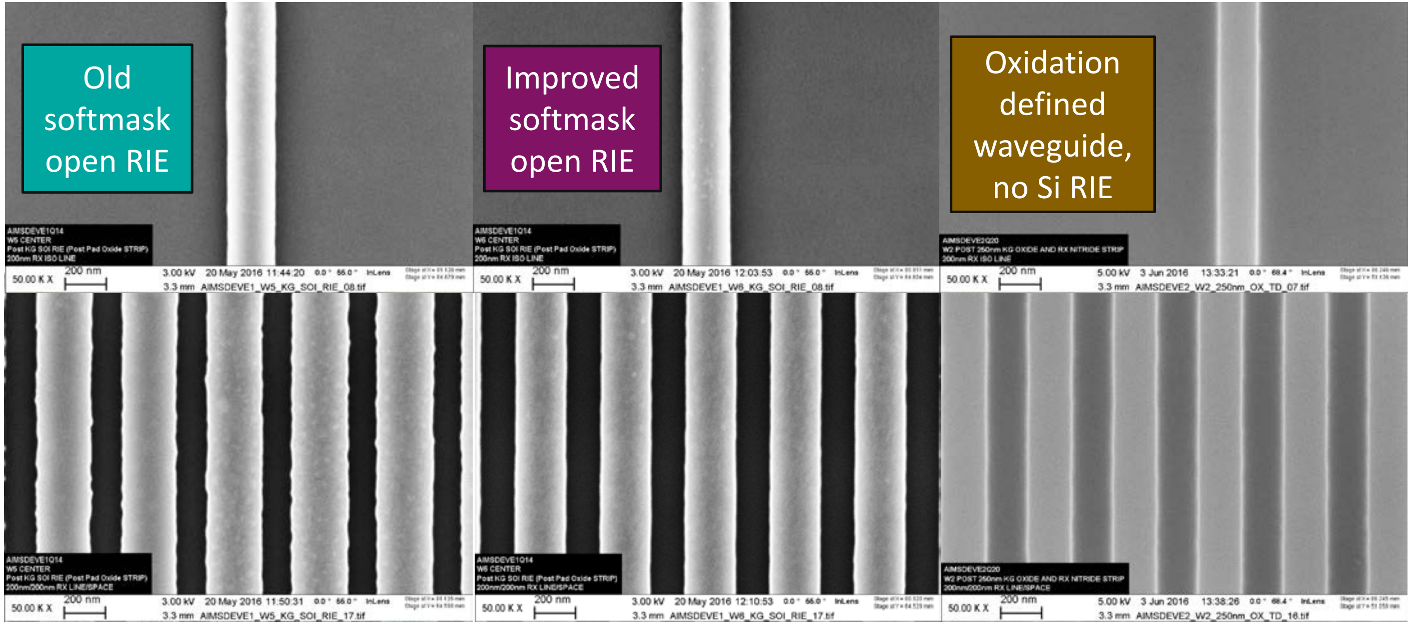
Line scanning spectroscopy while heating chip





Sequence of line scans for chip ΔT ~2-5 C.

Line Edge Roughness Generates Internal Etalons



	Old SM Open RIE	Improved SM Open RIE	Oxidation defined waveguide
LER – Isolated (nm)	**3.32 ± 0.20**	**2.45 ± 0.39**	**2.89 ± 0.20**
LER – Array (nm)	7.71 ± 0.45	3.41 ± 0.26	3.30 ± 0.10

Initial positive tone litho process had 5.7 nm LER

New softmask open etch has notable improvement compared to POR:
- Optical measurements to corroborate

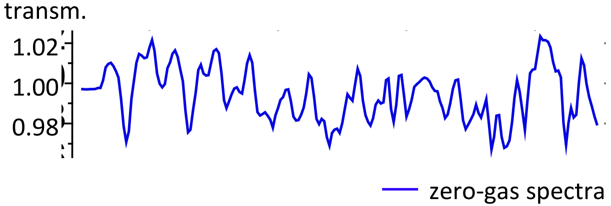
Oxidiation defined waveguides have LER comparable to new process:
- 250nm of SiO_2 grown with SiN mask to recess Si

Internal etalon amplitude depends strongly on polarization:
- Reduced significantly for TM mode compared to TE mode

Consequences of Miniaturization and Internal Etalon Mitigation

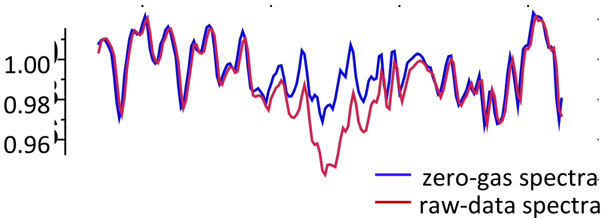
Conventional etalon subtraction

SiPh waveguide etalon background



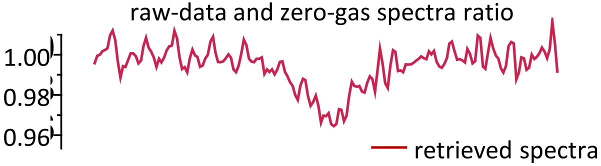
- *High index contrast of Si generates distributed reflections, multi-path interference*

With 2.87% CH_4

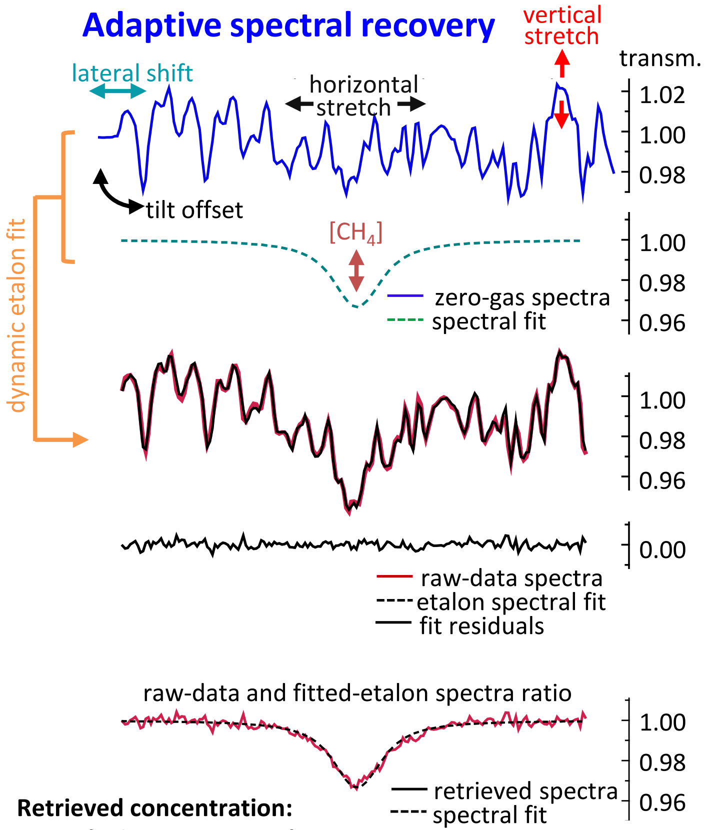


- *Drifting etalon spectrum can mask and cross-talk with the weak absorption signal*

Retrieved CH_4 spectra



Dynamic etalon fitting: Adaptive spectral recovery

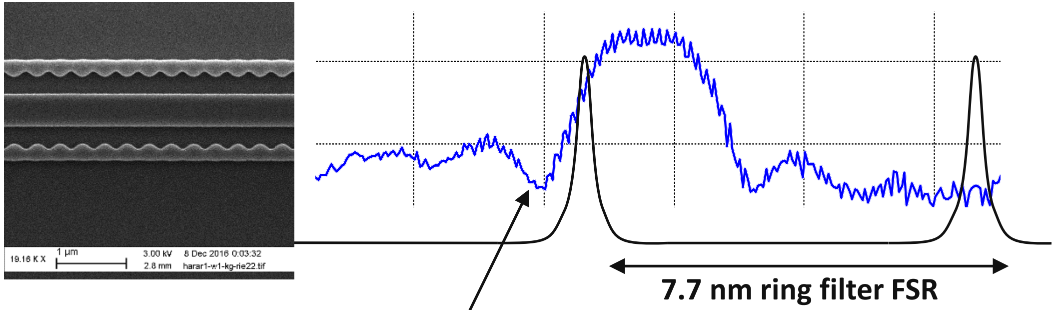


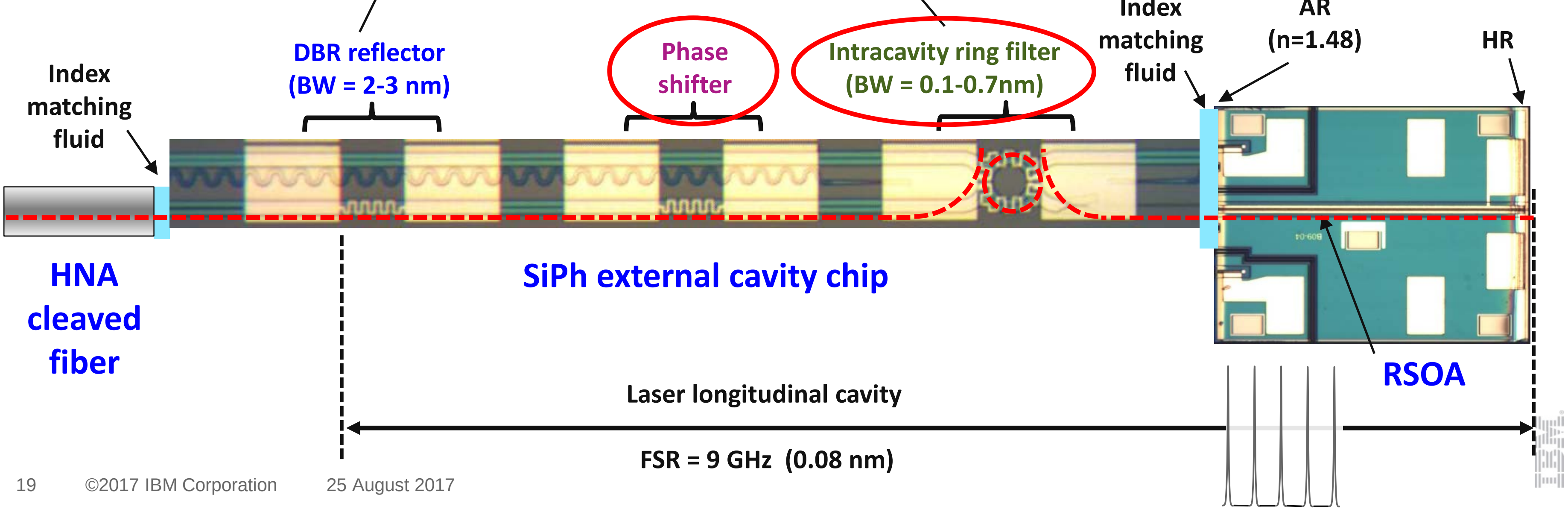
Retrieved concentration: 2.87% (reference CH_4 cell)

E. Zhang, L. Tombez, J. S. Orcutt, S. Kamlapurkar, G. Wysocki, W. M. J. Green, CLEO 2016.

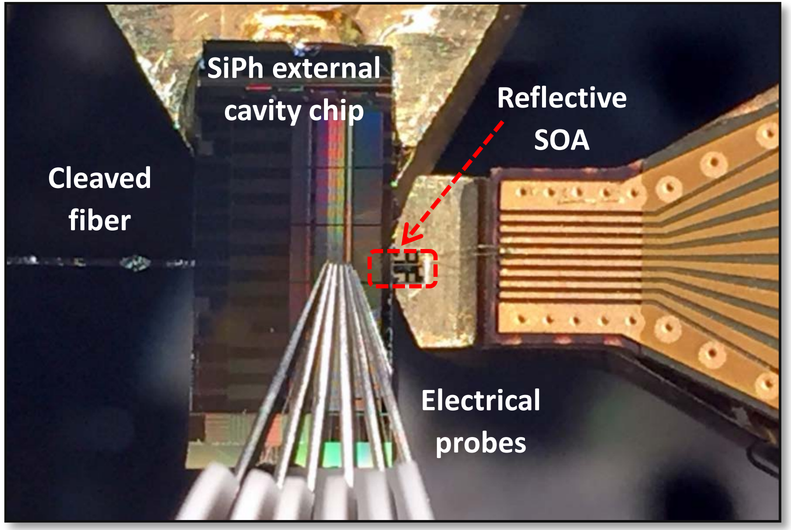
External Cavity Laser Design and Test

- III-V reflective SOA provides round-trip gain
- Longitudinal cavity formed by Bragg reflector and high-reflection coating
- Thermally tunable intracavity ring filter and phase shifter select a single longitudinal mode
- Compensate for temp, manufacturing tolerances

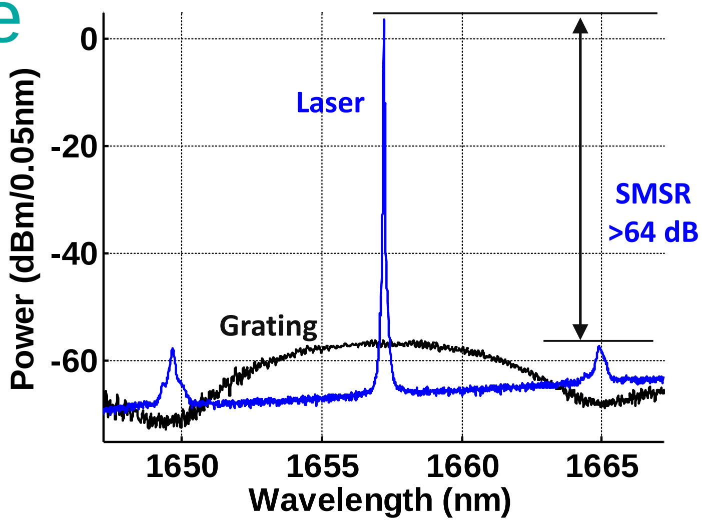




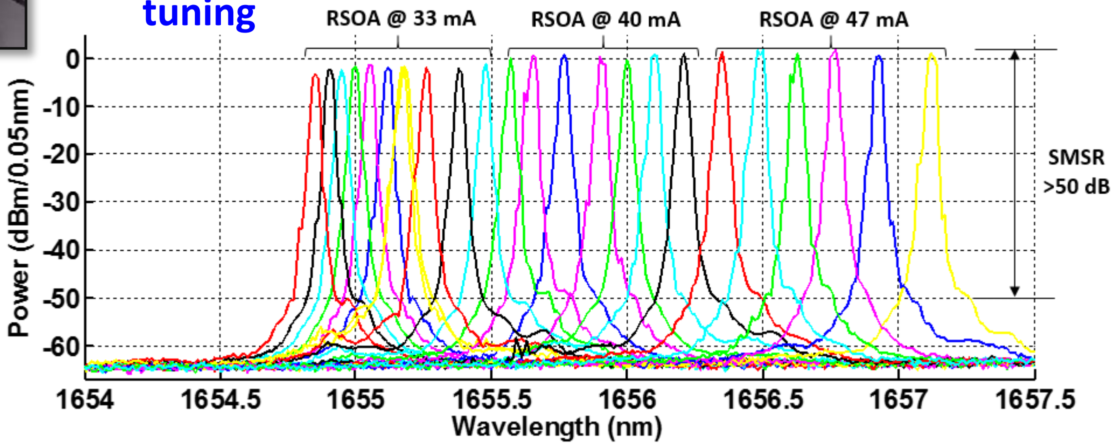
Hybrid III-V/Si Laser Performance



Laser spectrum



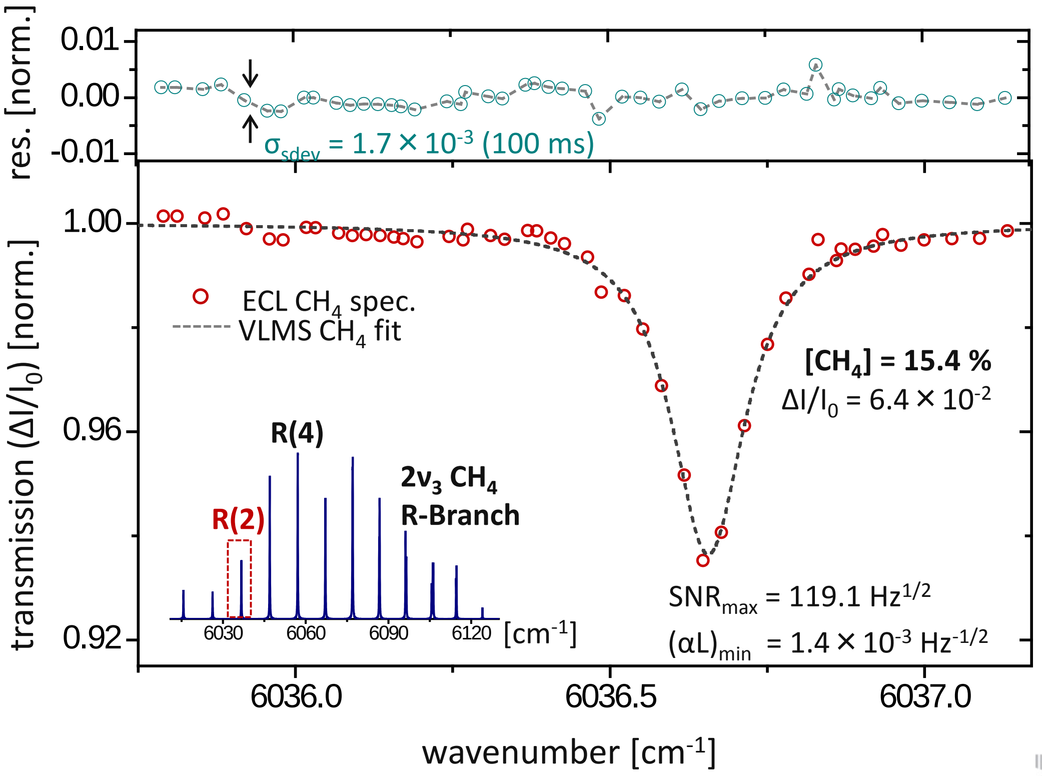
Wavelength tuning



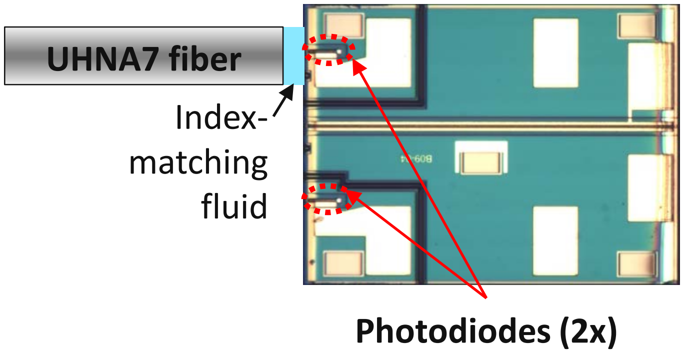
- Single-mode operation from 1650 - 1670 nm
- > 45 dB side-mode suppression ratio
- 2 - 8 nm mode-hop free tuning (depending upon DBR bandwidth)
- 0.5 mW output power (fiber-coupled)
- > 1 mW output power (on-chip)

Methane R(2) Spectral Acquisition

- CH_4 spectroscopy performed on R(2) line (weak, λ = 1656.5 nm) using hybrid III-V/Si laser and a fiber-coupled CH_4 reference cell
- Line fit accurately reproduces the concentration extracted with a commercial DFB laser
- Minor lithographic tweak to DBR grating required to target R(4) line

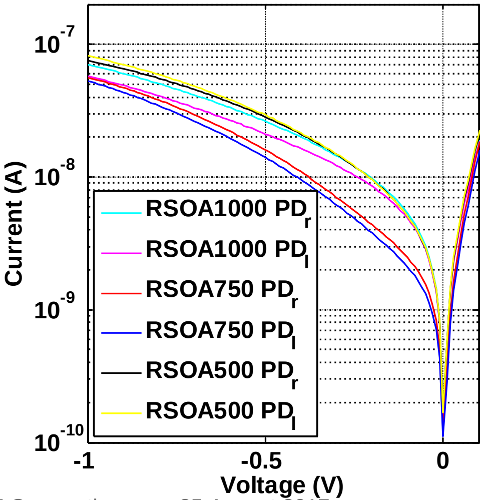


Photodetector Characterization: Dark Current and Responsivity

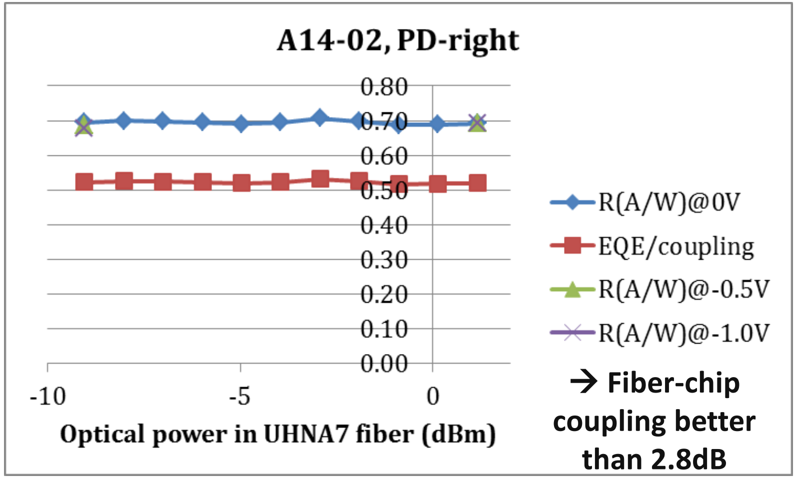


- Same QW epitaxy used for photodetectors; not optimized for performance
- Dark current within ~10x of optimized telecom-band photodetectors (<10^{-8} at -1V)

Dark current (22 °C)



Responsivity at 1650 nm (22 °C)



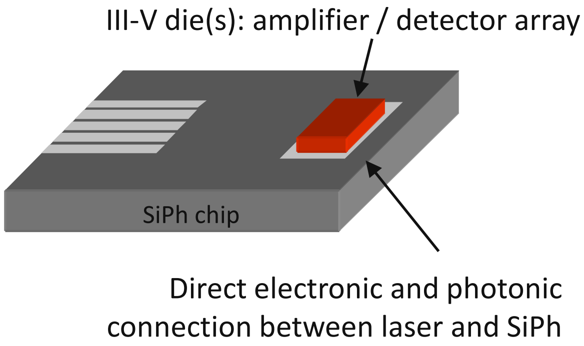
III-V Gain / Detector Chip Attach

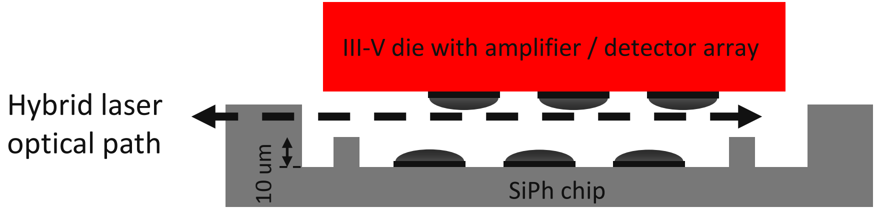
IBM differential:

- Full automation in standard CMOS assembly tooling
- Single or multiple III-V die flip-chipped to SiPh
- Disruptive scalability in volume and cost

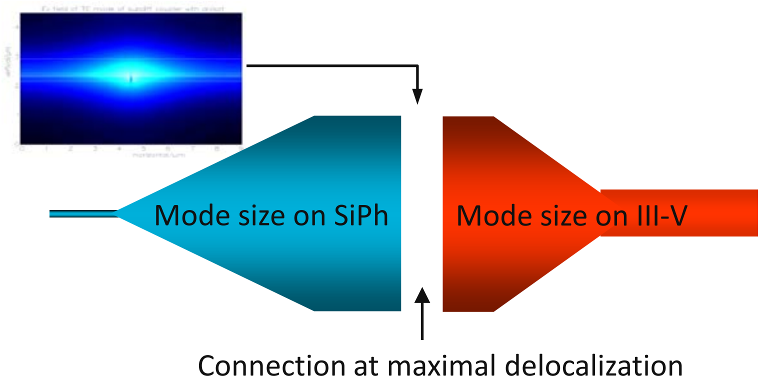
Innovation:

- Mode shape engineering to relax tolerance
- Solder surface tension re-aligns III-V chip
- Superior thermal characteristics





Pick and place (±10 μm), then anneal (< ±1 μm)



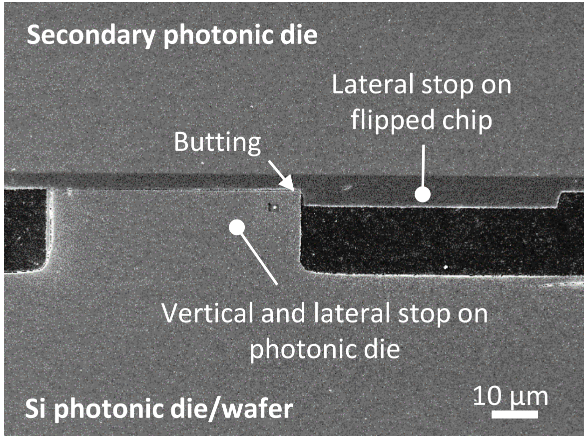
Key challenge:

- Sub-micron tolerances for passive alignment

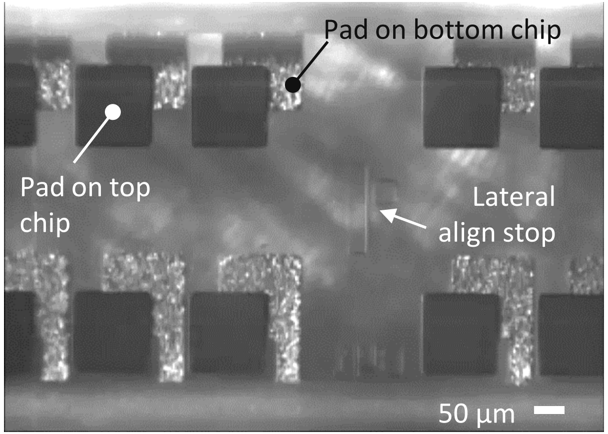
T. Barwicz, et al., GFP 2015; J.-W. Nah et al., ECTC 2015; Y. Martin et al., ECTC 2016.

Solder Induced Self-Alignment

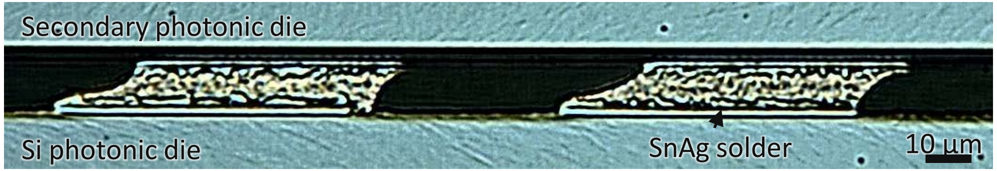
Cross-section of stops after assembly



Infrared view through assembly at anneal



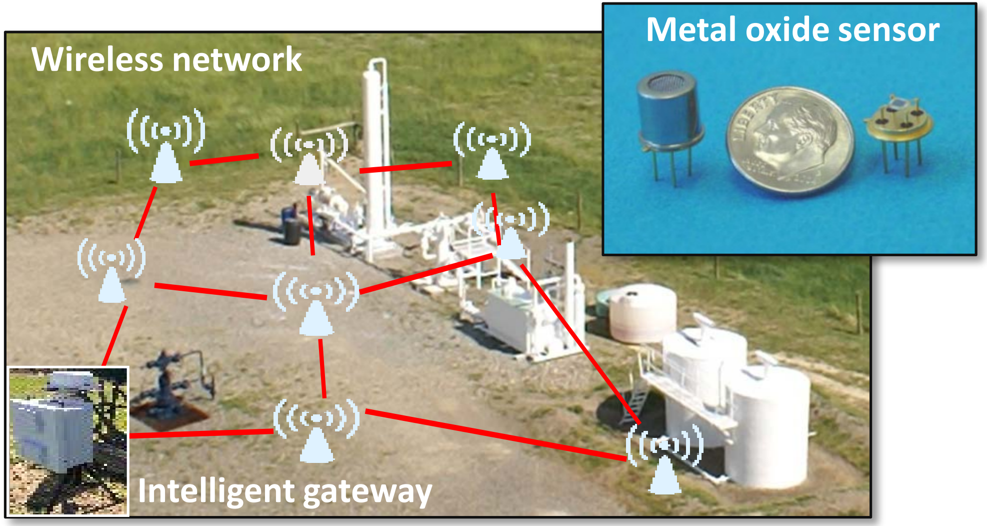
Cross-section of solder pads after assembly

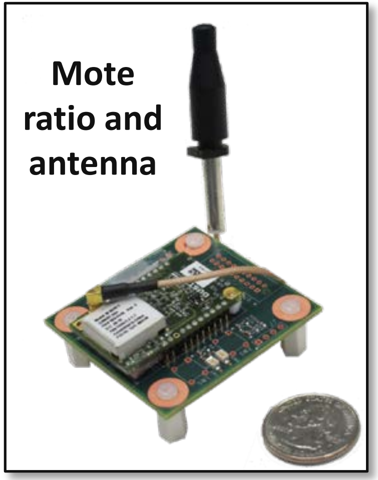


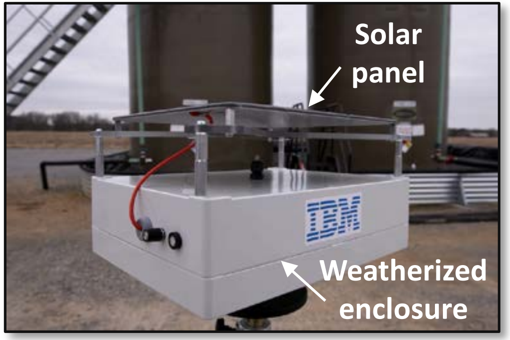
- Patterning limits accuracy at butting of lithographically defined stops.
- Solder pads offset by design for sustained force at butting (*J.-W. Nah et al, ECTC 2015*)

Wireless Sensor Nodes

- **Methane sensors:**
  - Hot metal oxide chemi-resistor
  - Off-the-shelf, cost-effective
  - Sensitivity ~1 ppm, response time ~0.5 sec
  - Non-selective: broad sensitivity to VOCs, humidity
- **Power and packaging:**
  - Solar power harvesting for remote operation
  - Robust all-weather enclosure
- **Intelligent nodes and network:**
  - Dynamic “hopping” communication pathway for mote failure tolerance
  - Remote gateway aggregates CH_4 / wind data, links to cloud
  - Analytics partitioned between mote, cloud



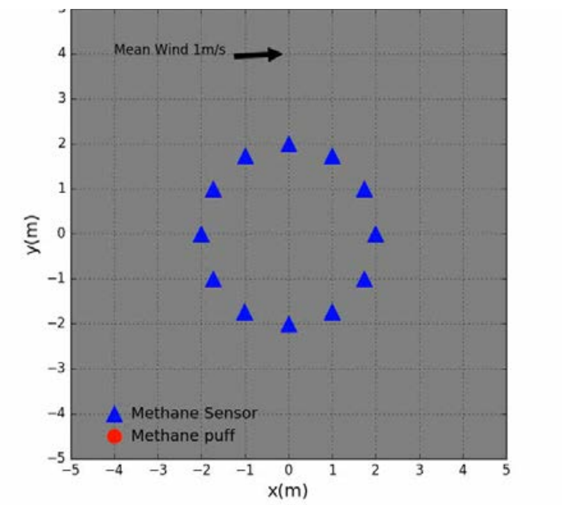


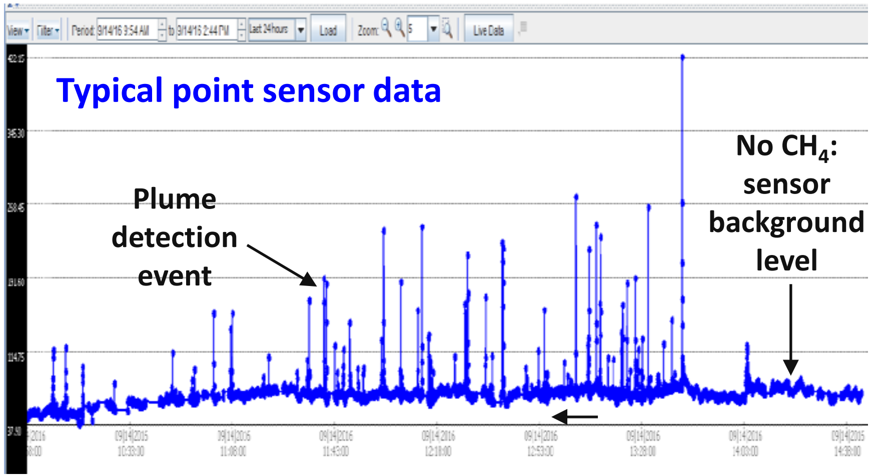


Meandering Plume Model

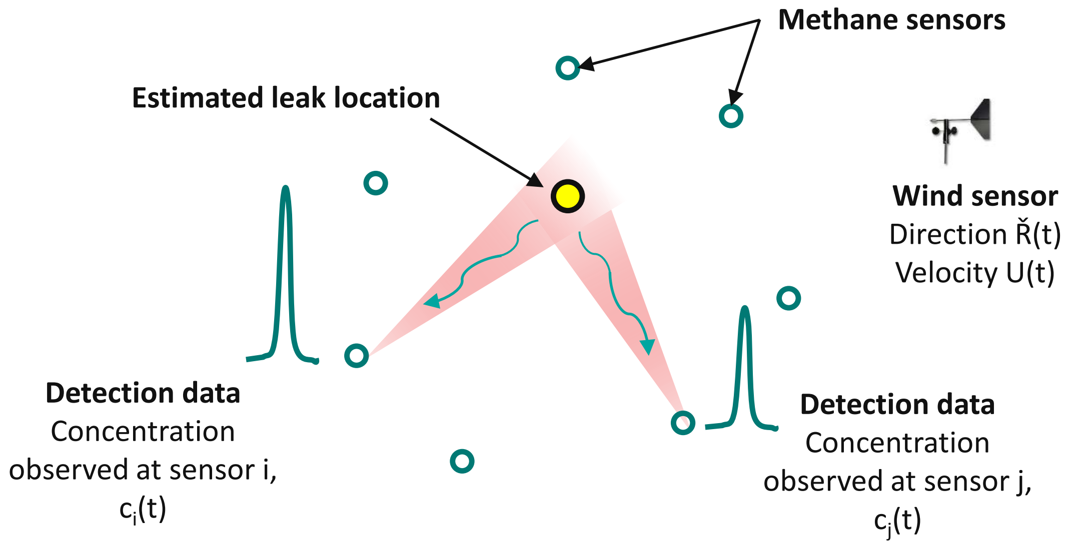
- **Within the "near-field", CH_4 plume dispersal dominated by airflow, turbulence, obstacles… not diffusion**
- **Distributed network of point sensors which can:**
  - Resolve short CH_4 peaks (~1-10 sec duration) at low concentration (~1-100 ppm), without saturation
  - Characterize wind direction and velocity
- **Network communication protocols must be time synchronized to:**
  - Correlate CH_4 and wind data time series
  - Optimize power management, communication bandwidth, and computational workload
- **Use physical models and statistical data analytics to infer location and magnitude of the CH_4 source**

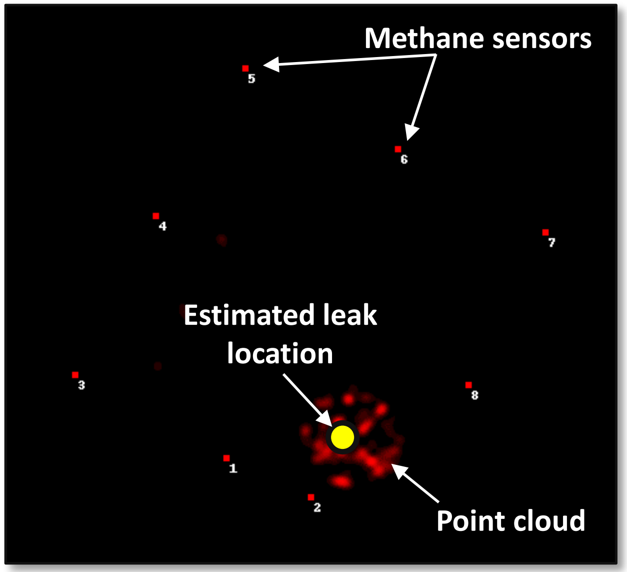
Simulation of a CH_4 leak plume





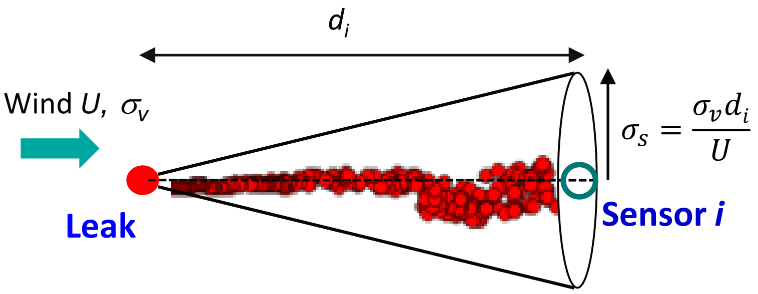
Leak Location Estimation





- Peaks observed at methane sensors are combined with wind direction data to estimate the most likely direction the plume took to arrive at the sensor
- Intersection points are generated for all peaks versus all other peaks to create a point cloud
- Centroid estimation using many such points allows the estimation of the leak position
- Cluster analysis and spatial filtering further improves this estimate

Source Magnitude Estimation



With an estimate for leak position, use the short range meandering plume equations to estimate source magnitude for each peak, recorded by each sensor:

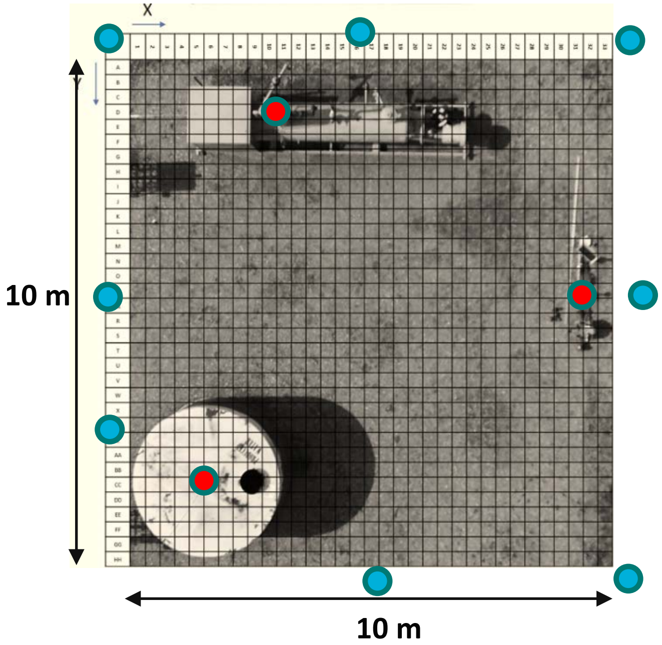
$$\boldsymbol{Q_i = \pi \sigma_s^2 U c_{p,i}}$$

where:

Q_i = source magnitude estimate based on sensor peak (gms/sec)
$c_{p,i}$ = peak concentration at sensor (gms/meter^3)
d_i = distance to source (meters)
σ_v = variation of wind velocity over methane peak interval (meters/sec)
U = average wind velocity over methane peak interval (meters/sec)

- Plume meanders: Radius sampled grows linearly with distance d_i from sensor *i*, according to wind variation σ_v
- Largest CH_4 peaks $c_{p,i}$ at each sensor correspond to the most direct path from leak to sensor
- Average the maximum Q_i values from each sensor to obtain best source magnitude estimate Q_{est}

Field Test System Validation

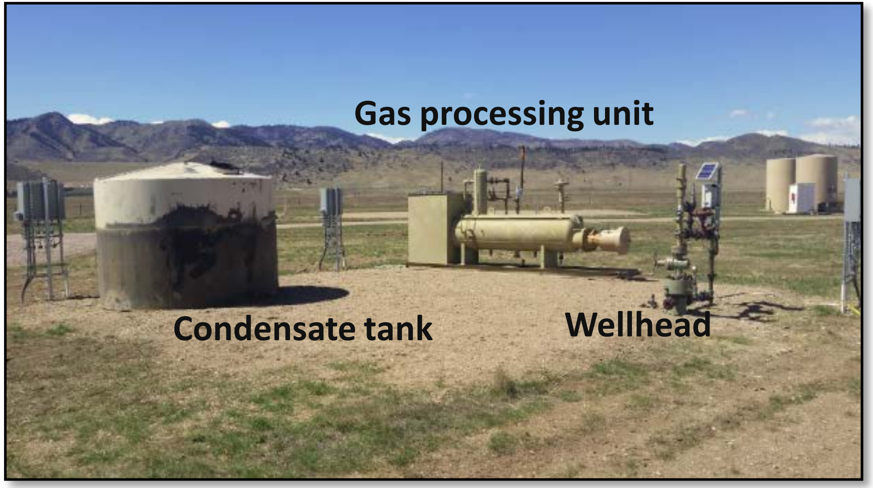


Known leak location

Estimated leak location

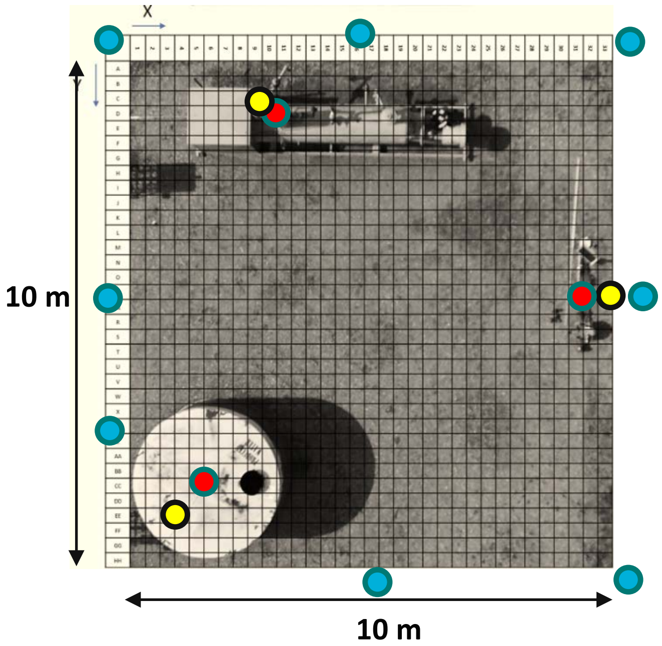
Methane sensor location

METEC Pad 2: Locations of Test Leaks



- Testing performed at the Methane Emissions Technology Evaluation Center – METEC (Colorado State University)

Field Test System Validation



Known leak location
Estimated leak location
Methane sensor location

Location of source

Site	Known leak position (m)		Estimated leak position (m)		Error (m)
	X	Y	X	Y	
Tank hatch	1.1	-3.8	1.25	-4.25	**0.48**
GPU	-3.5	0.25	-3.6	0.42	**0.22**
Wellhead	1.5	3.0	1.95	3.76	**0.88**

Magnitude of source

Site	Known flow rate (SCFH)	Estimated flow rate (SCFH)	Error (SCFH)	Error (%)
Tank hatch	32	34	2	**7%**
GPU	32	29	-3	**8%**
Wellhead	32	33	1	**4%**

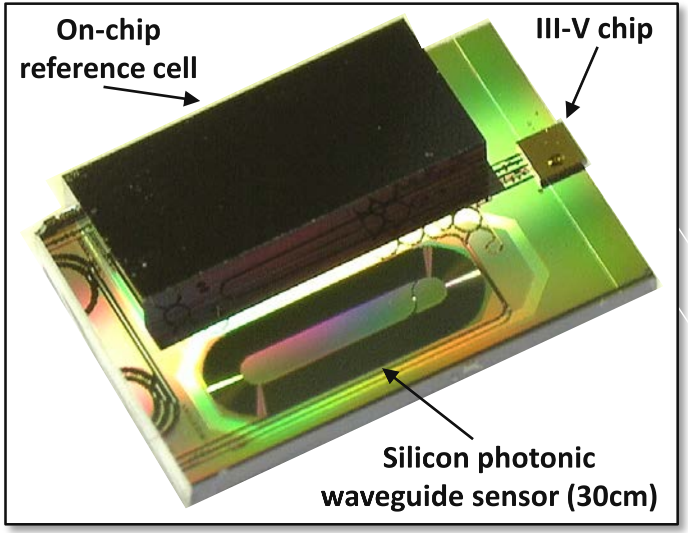
- Good performance with single sources
- Approaches to handle multiple simultaneous sources are under development

Key Upcoming Milestones

- **Demonstrate III-V / Si laser and fully-integrated SiPh sensor assemblies:**
  - Single mode tunable laser required for 1650nm methane line scanning
  - ***Facilitates economical, large-scale deployment of continuously monitoring sensor networks***

- **Field testing of a "hybrid" methane leak detection system:**
  - Replace several chemi-resistors with SiPh optical sensors
  - Deploy a functional sensor network, demonstrate leak detection / localization at O&G partner sites
  - Study leak rate accuracy, species cross-sensitivity, false-positives rate, overall "hybrid" system performance improvements

Mechanical prototype of full sensor assembly



Sensor deployed at industry partner's wellpad

Thank You!

IBM T.J. Watson Research Center

