

## **DOE Sensors & Automation 2006 Annual Portfolio Review<sup>+</sup>**

### **High-Speed Gas Analysis with PHASED\* MGA Gen.1 on NeSSI\*\***

\* Phased Heater Array Structure for Enhanced Detection; MGA=μGas Analyzer

\*\* Micro Gas Analyzer; New Sampling and Sensor Initiative by CPAC

Part B of “Wireless and Sensing Solutions Advancing Industrial Efficiency”

**Ulrich Bonne, PI**  
**Honeywell Labs, Plymouth, MN 55441**

+ Black Headers = DOE-requested info

Purple Headers = Advisors-requested info

Blue Headers = Technical paper presentation

# High-Speed Gas Analysis with PHASED MGA Gen.1 on NeSSI\*

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Leonard Hilton, Nancy Iwamoto, Jerry Evans, Honeywell International  
Rob Synovec, Vanessa Reid, Adam McBride, Chemistry Department/CPAC, University of Washington, Seattle, WA;  
Cyrus Herring, Kirsten Kunze and David Kellner, Caviton, Inc., Champaign, IL

## Abstract

As part of the DOE-OIT project on "Wireless and Sensing Solutions Advancing Industrial Efficiency", this sensor and sampling system's development goal is to field test of PHASED\* MGA and other microanalytics on NeSSI-II\*\* in the plants of collaborating members of the processing industries. The presentation of progress on this project will cover: 1) Further development of film materials such as Nanoglass and DB-5; 2) Experimental evaluation of such materials with diverse analytes (C1–C10); 3) Micro-machining and performance evaluation (preconcentration and separation) of PHASED chips and associated electronics; 4) Detector development to improve the performance of the on-board TCD, integrate the MDD as a two-dimensional detector, and development of an alternative CID; 5) Finalization of system packaging; and 6) Design and development of the NeSSI hardware and software for field testing, communication component selection and evaluation, and preparation for pilot-plant tests, all of which help to realize this cross-industry micro-analytical platform of the future, which will save capital, installation, maintenance, and plant operation costs.

**Acknowledgments:** This project is made possible with cost-shared funding by DOE-OIT, Honeywell, and technical collaborators: Michelle Cohn, UOP; Rob DuBois, Dow Chemical; Jeff Gunnell, ExxonMobil; Frank Schweighardt / Dean Chin-Fatt, Air Products; Joe Andrisani, DuPont; Mel Koch, CPAC; Steve Doe, Parker-Hannifin; and David Simko, Swagelock, under the project entitled "Wireless and Sensing Solutions Advancing Industrial Efficiency"

•PHASED =Phased Heater Array Structure for Enhanced Det.; NeSSI = New Sampling Sensor Initiative; MGA=μGas Analyzer

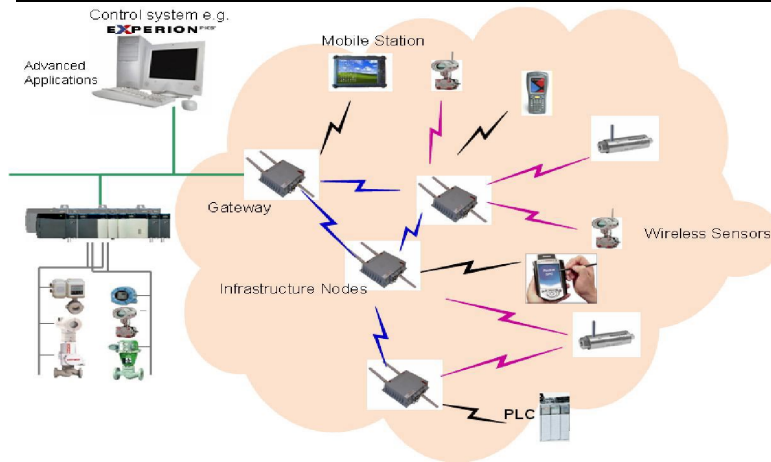
**Presenter Bio:** Ulrich Bonne, Sr. Research Fellow, joined the Honeywell Labs at Plymouth, MN, in 1965, and received his MS and PhD in Chemical Physics from the University of Göttingen, Germany. His present R&D interests are focused on micro gas analyzers and sensors for industrial, environmental and medical applications.

# Project Description

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- **Need:** Modular, standardized, IS and networked sampling system (NeSSI-II) to support composition analyses of process streams
- **Goal:** Field testing of PHASED and other microanalytics on NeSSI-II in the plants of collaborating members of the processing industries
- **Core Technology:** Hi-Speed, Multi-Stage Pre-Conc., Affordable  $\mu$ GC; NeSSI-1.5 configuration meeting mini-VOC needs
- **Novel/Transformational Element(s):** PHASED  $\mu$ GC on SP-76 of NeSSI-II
- **Initial Industry for Application:** 2 field test sites: 1) Helium production, and 2) HCl in butane
- **Crosscutting Applicability:** Industrial applications in Refining/Petrochemical/Chemical, Mining/Minerals/Materials, Food/Ag/Forestry, Pharmaceutical, Natural Gas, Air Quality and Medical Diagnostics
- **Key Project Deliverable(s):** Report on results from 2 field tests

# Wireless & Sensing Solutions for Improved Industrial Efficiency

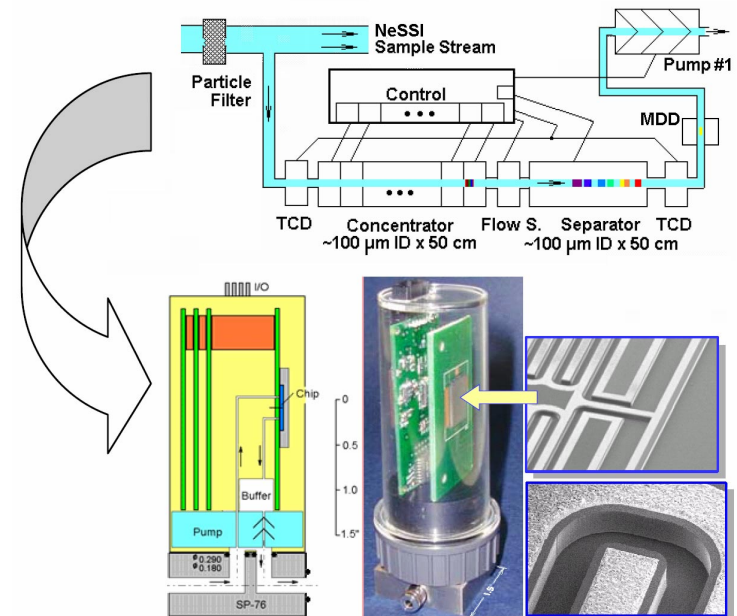


## Part A: Wireless Network for Secure Industrial Applications

- Robust industrial wireless infrastructure
- High data security
- Lowest total cost of ownership
- Low power requirements and extended battery life
- Enhanced availability and coexisting with other RF systems

## Part B: PHASED Gas Composition MicroAnalyzer

- MEMS based micro gas chromatograph
- Real-Time, on-/by-line measurement of process streams
- High-Speed, 1-10 seconds analysis time
- 20-50-Stage, sub-second pre-concentration
- NeSSI-Compatible Microanalytics
- Low-Power requirements
- Affordable



# NeSSI and PHASED

## Microanalytics for Process Control Solutions Technical Concept – NeSSI-I, -II, -III

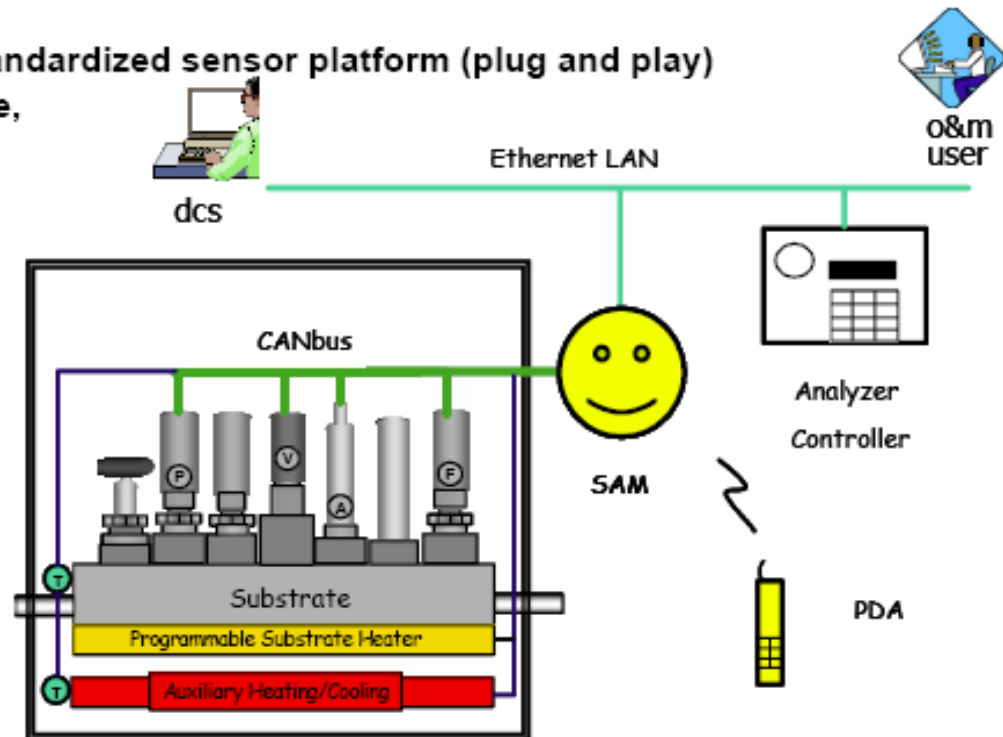
### • NeSSI-III Sampling System:

- Modular, IS, networked, standardized sensor platform (plug and play)
- Pressure, flow, temperature, and composition
- Micro-components with lower-cost life cycle
- Supports microanalytics:

**PHASED  $\mu$ GC**

### Applications:

- In-situ process stream monitoring
- Ind. process control and automation
- Petroleum refineries, chemical processing plants, metals & food processing



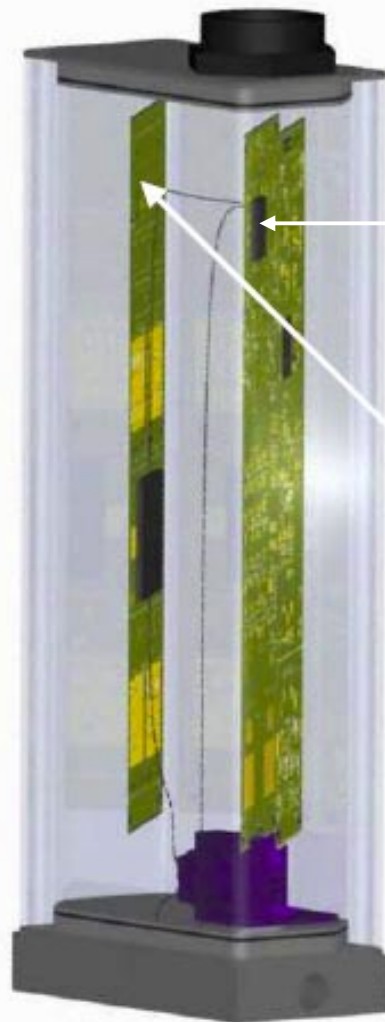
**Process monitoring with lower-cost of life-cycle  
and improved safety**

# Technical Progress

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- Progress this year has been made across all areas of the project, and is detailed in the following slides
  - **Separation** – New column materials have substantially improved separation performance
  - **Detection** – A 50-70x more sensitive TCD has been designed and 90% processed. Likewise, a new Chemical Ionization Detector (CID) has been designed and 90% processed, and a more compact version of the Micro-Discharge Detector (MDD) has been completed to fit into the PHASED-on-NeSSI SP-76 housing
  - **Test Configuration** – The hardware configuration for the test system and the NeSSI platform is complete; software to support the test configuration is designed and is in development

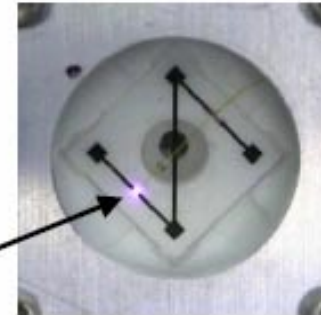
# PHASED on NeSSI: Design



**Power & Signal I/O**

**PHASED Chip**  
w/Capillary I/O

**MDD**



**Housing for  
PHASED w/TCD  
and MDD on  
NeSSI**

**Sampling Pump**

**SP-76**  
1.5 x 1.5" x 2

Tom.Rezachek@Honeywell.com,  
Fouad.Nusseibeh@Honeywell.com and  
Leonard.Hilton@Honeywell.com



# PHASED on NeSSI: Fabrication

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Top Cover

Side Cover

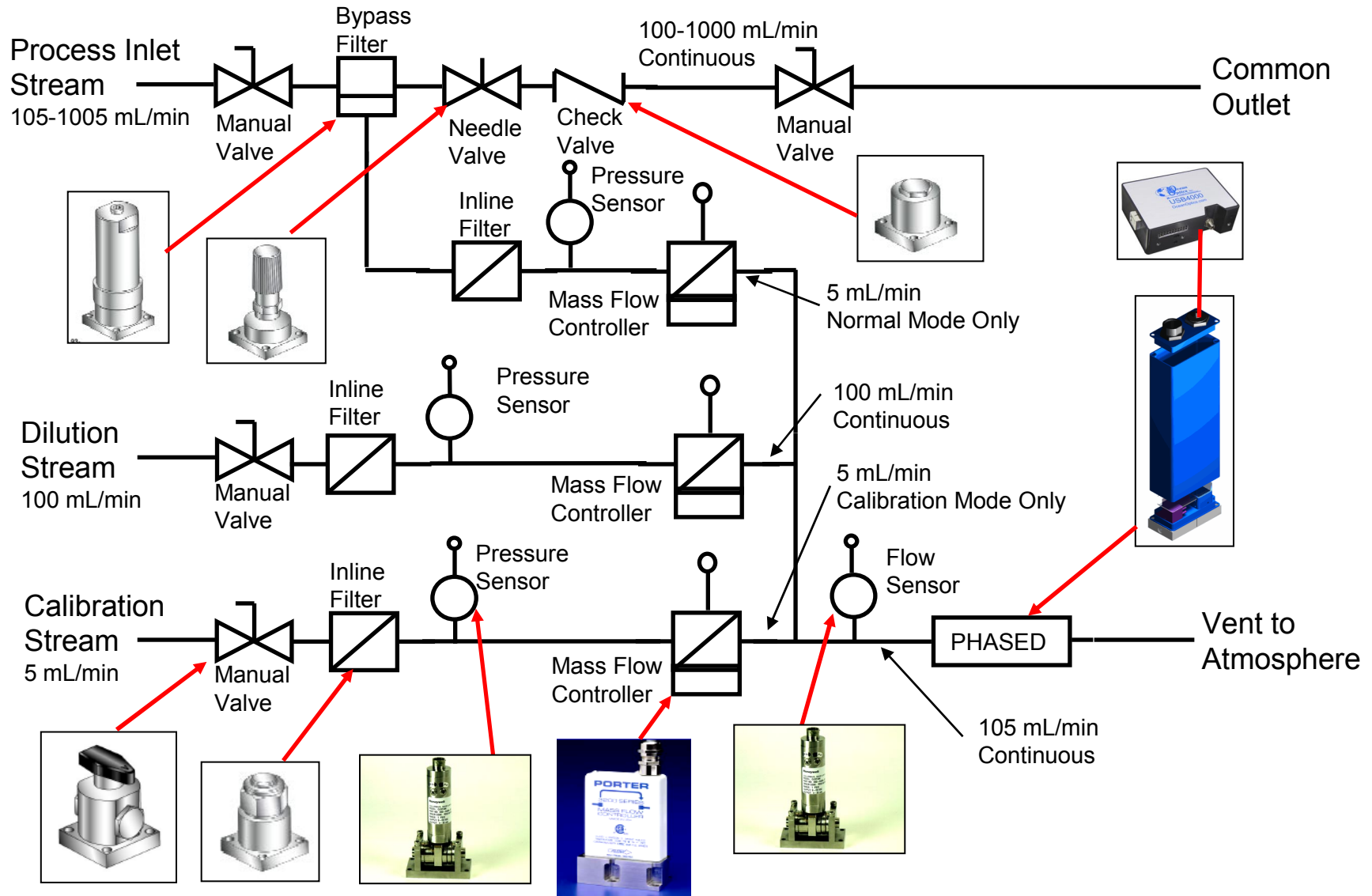
Circuit Boards

Buffer Volume  
on Base Cover

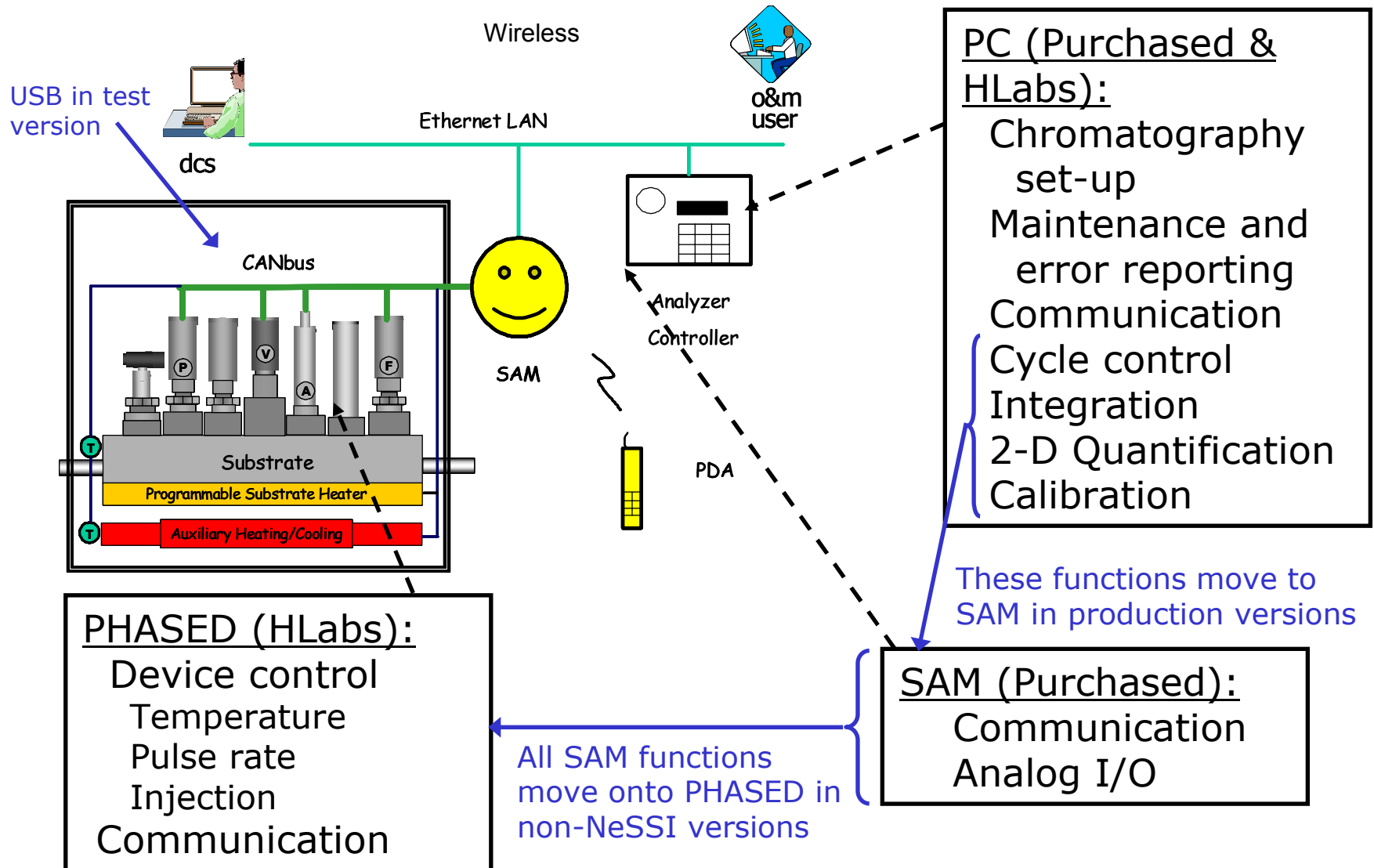
2 SP-76 Blocks



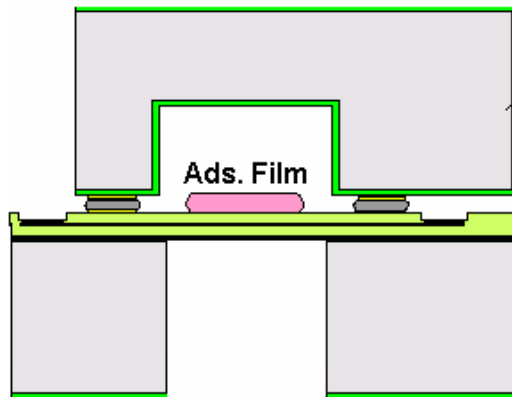
# Flow Diagram of PHASED-on-NeSSI



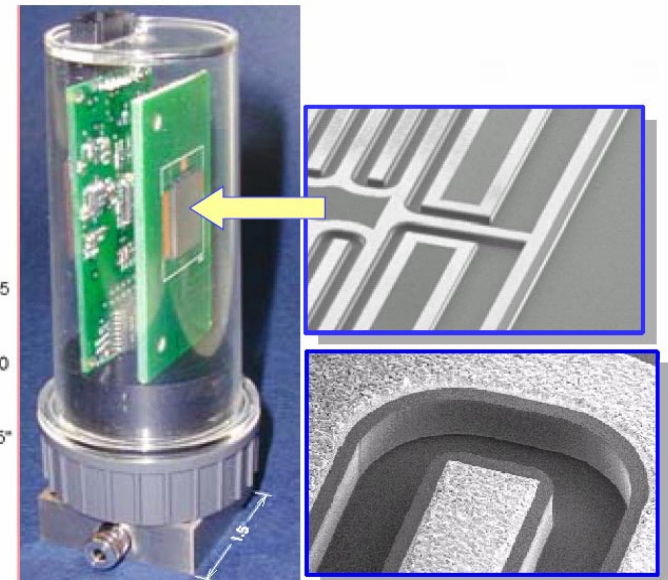
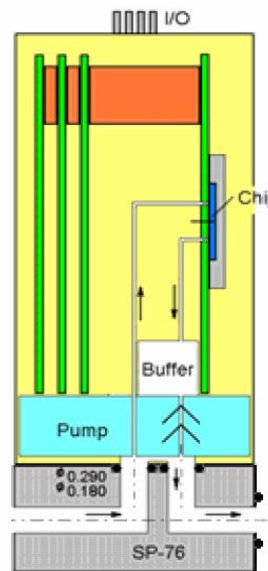
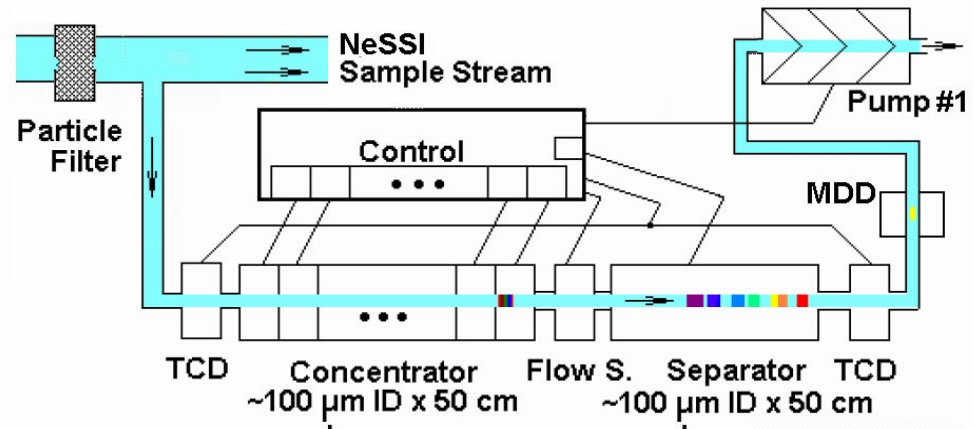
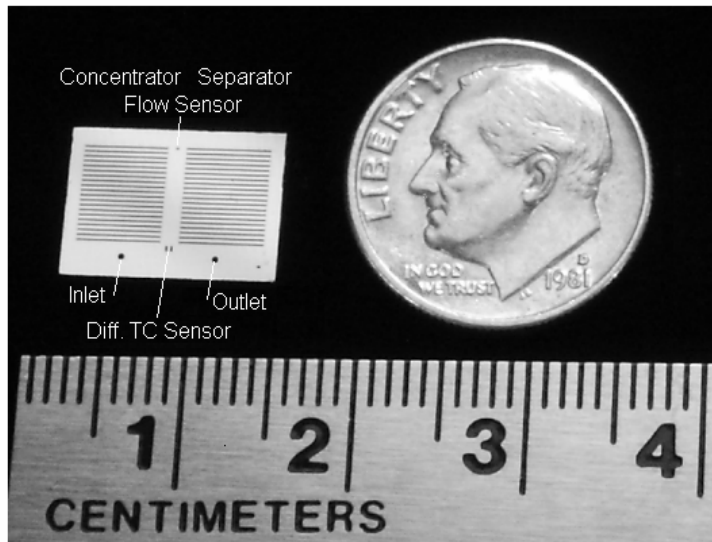
# PHASED-on-NeSSI Software Architecture



# PHASED Analyzer and Device Packaging



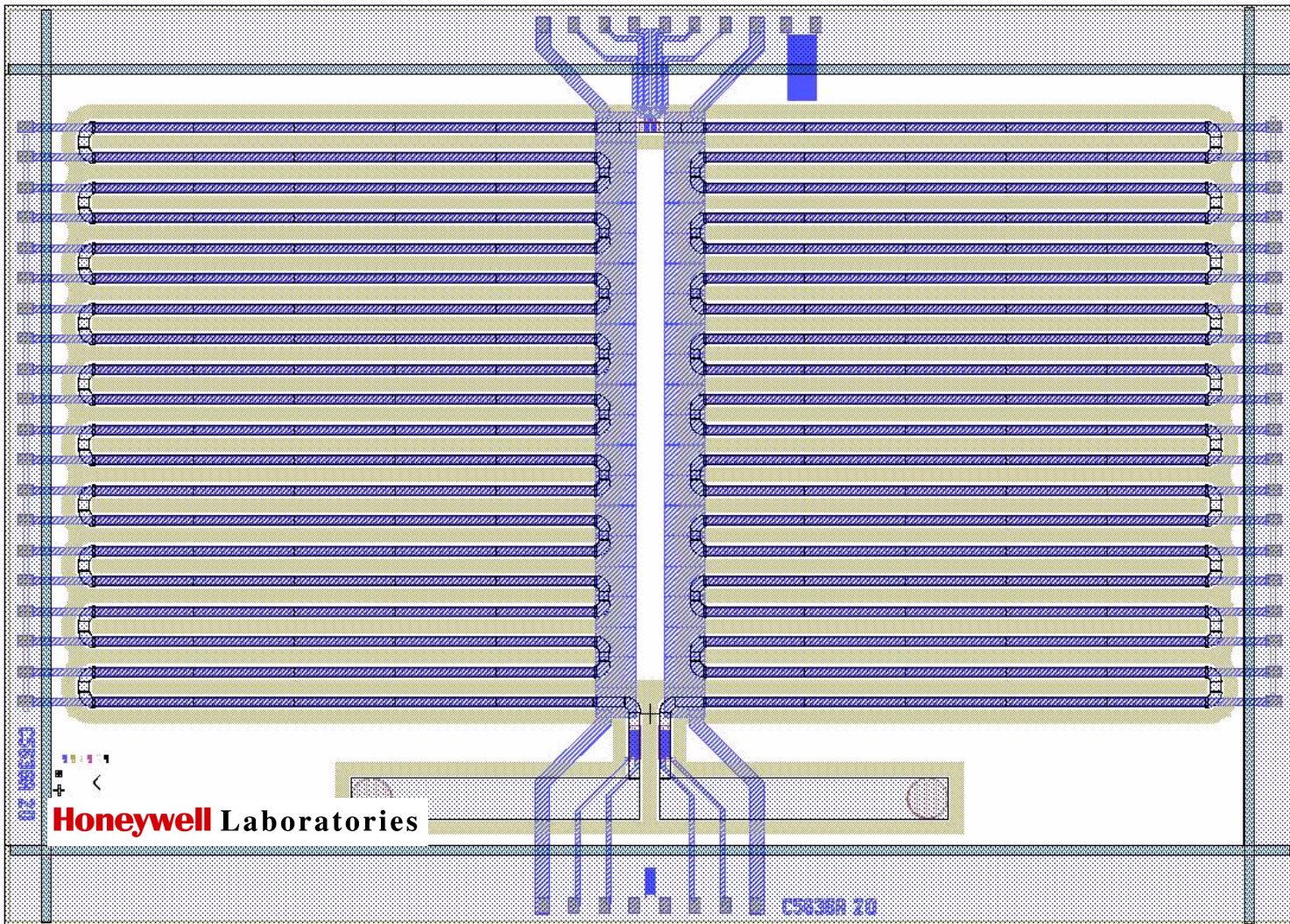
**PHASED Cross Section**



**NeSSI**



# PHASED MGA Gen.1, with Integrated Sensors, Concentrator and Separator.



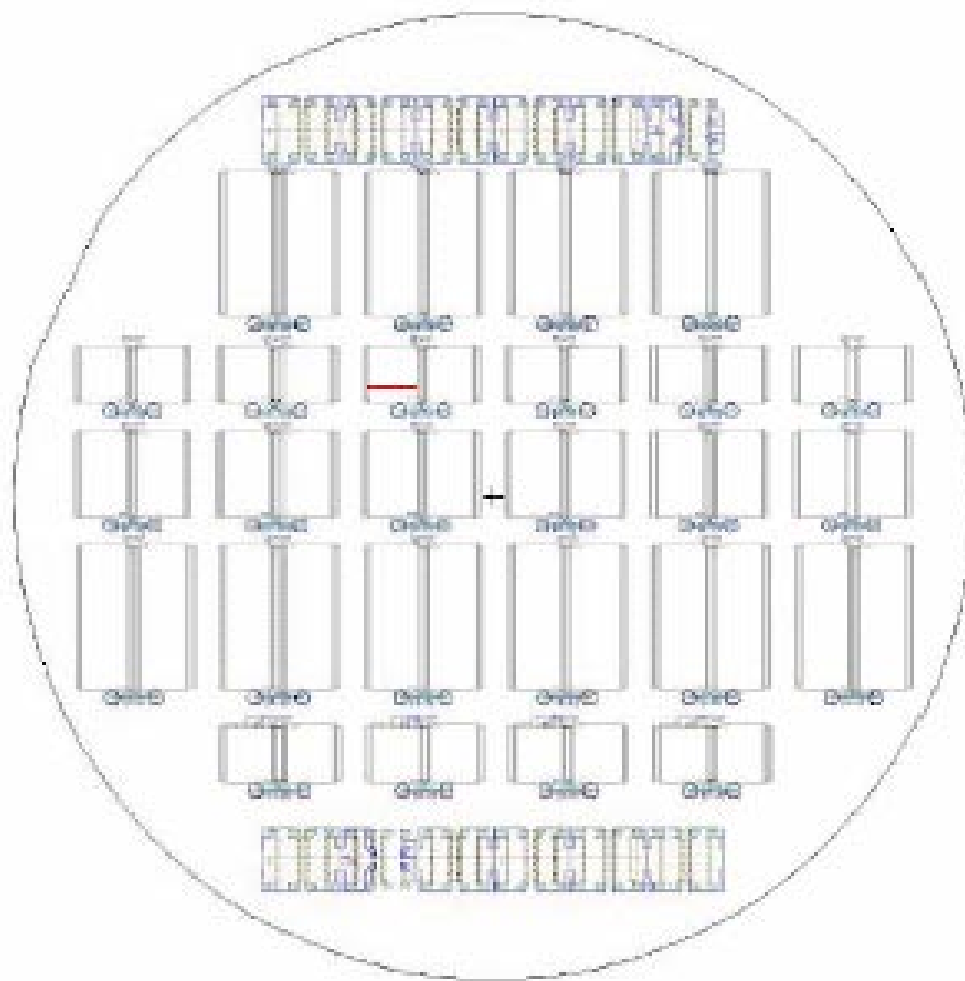
20-Element Pre-Concentrator,

Diff. TC,

20-Element Separator

# Layout of PHASED on a 4" Wafer

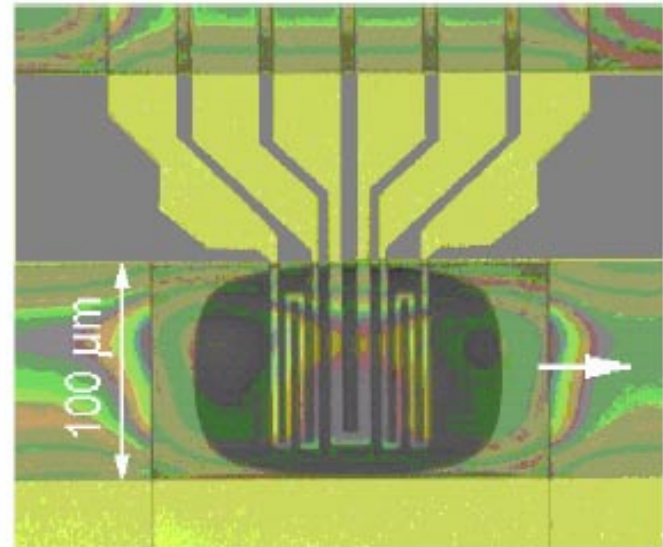
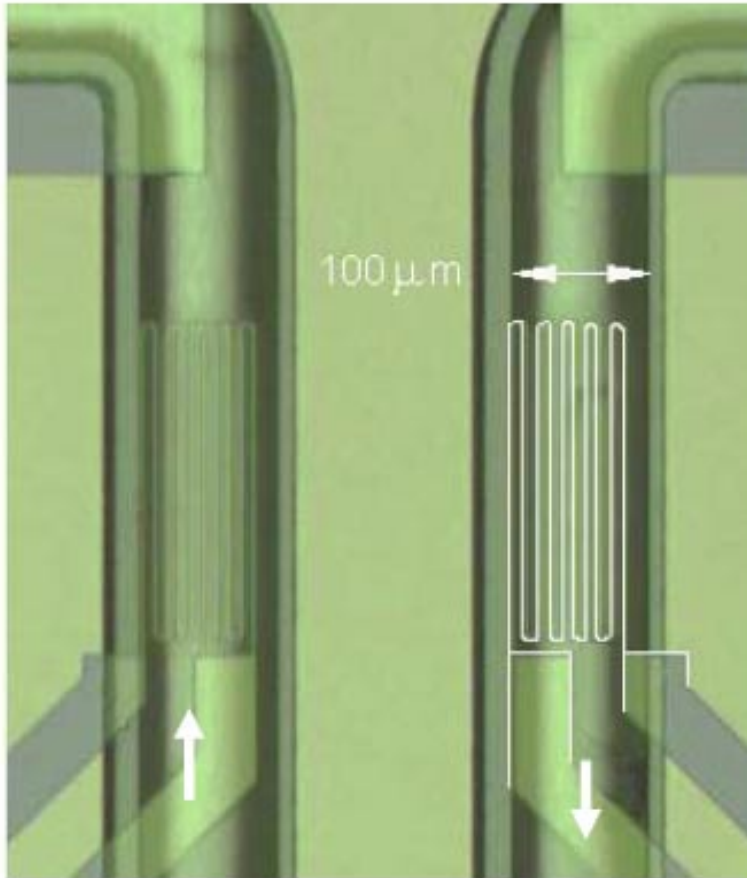
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# Sensors Integrated into PHASED

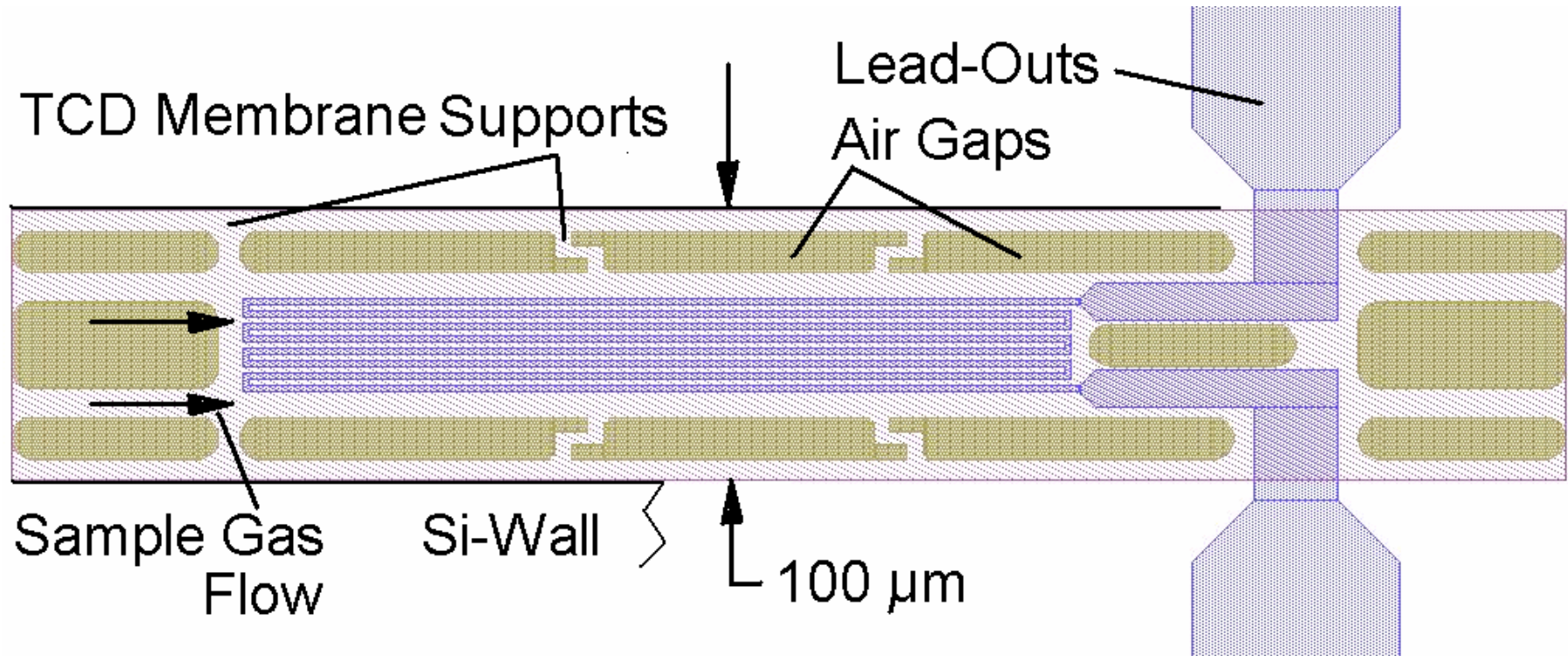
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PHASED  $\sim 100 \times 250 \mu\text{m}$  TCD &  $100 \times 72 \mu\text{m}$  Flow Sensors  
on a  $1\text{-}\mu\text{m}$  Membrane, in a  $100\text{-}\mu\text{m}$  Channel

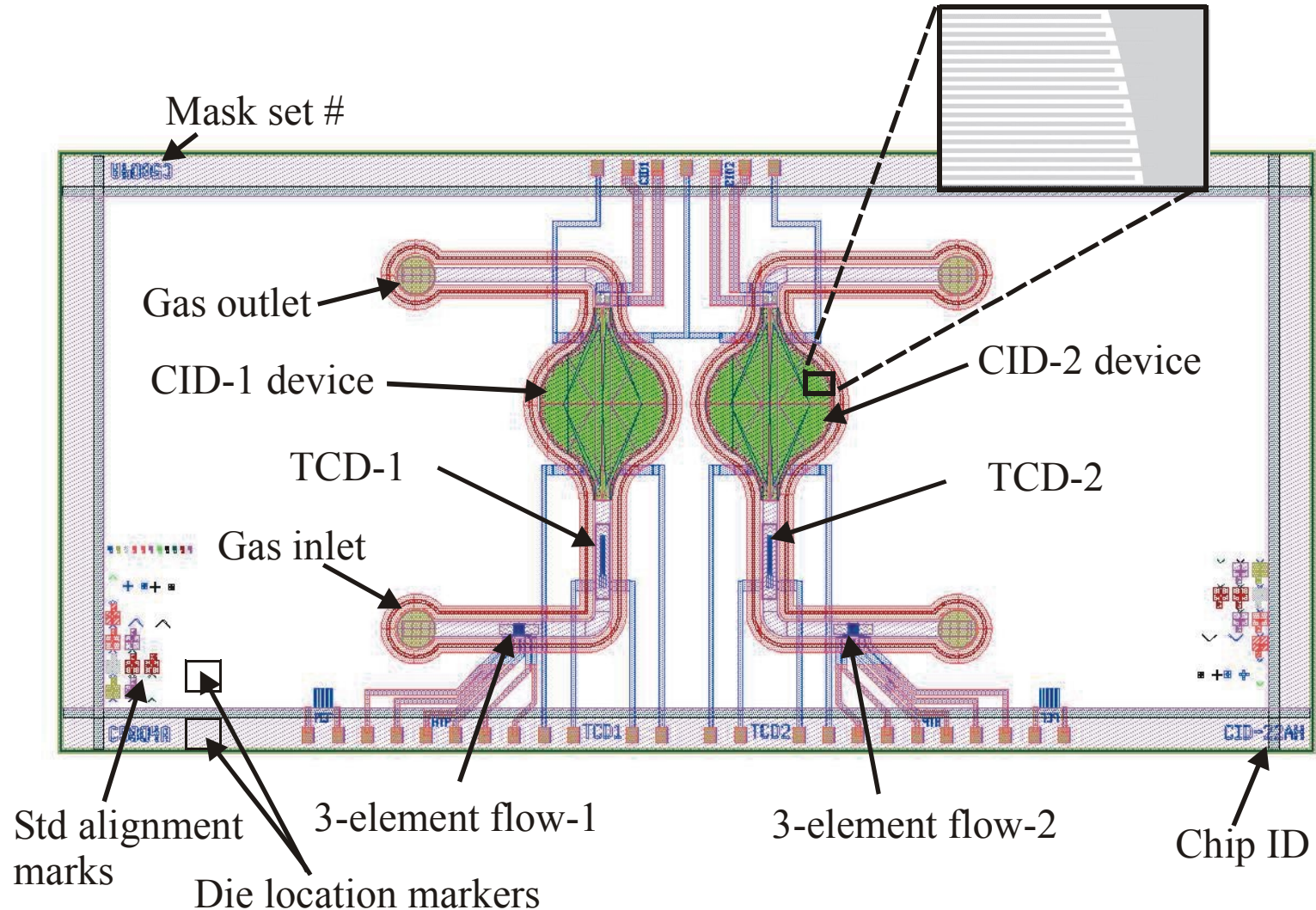


# Improved TCD Design: Better Thermal Isolation

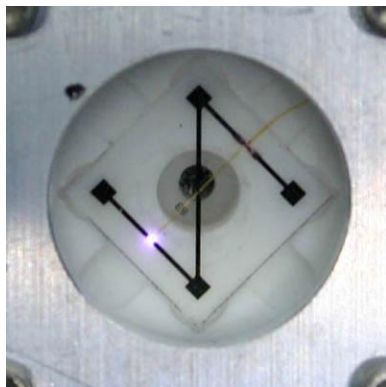


Sensitivity gain due to: Thermal barrier created by air gaps.  
Two-sided gas contact

# New Chemical Impedance Detector Design

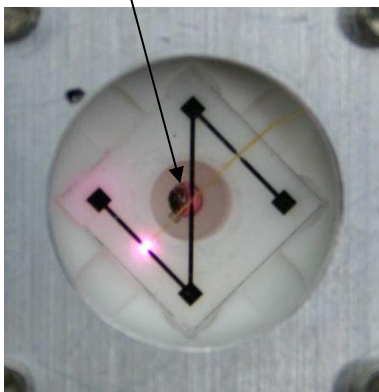


# Micro-Discharge Detector Performance



**CO<sub>2</sub> Discharge**

**Fiber Optic Cable**



**He Discharge**

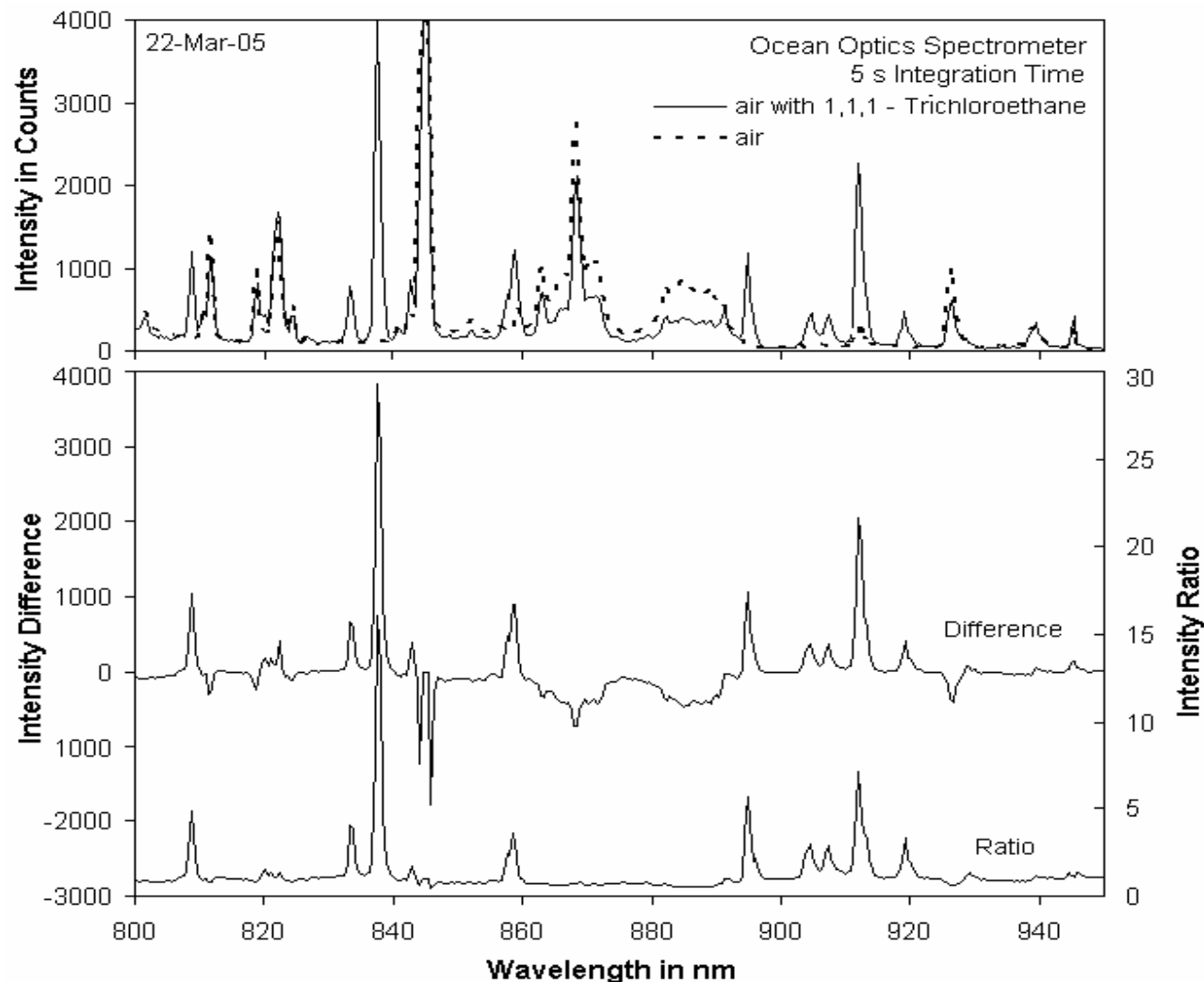
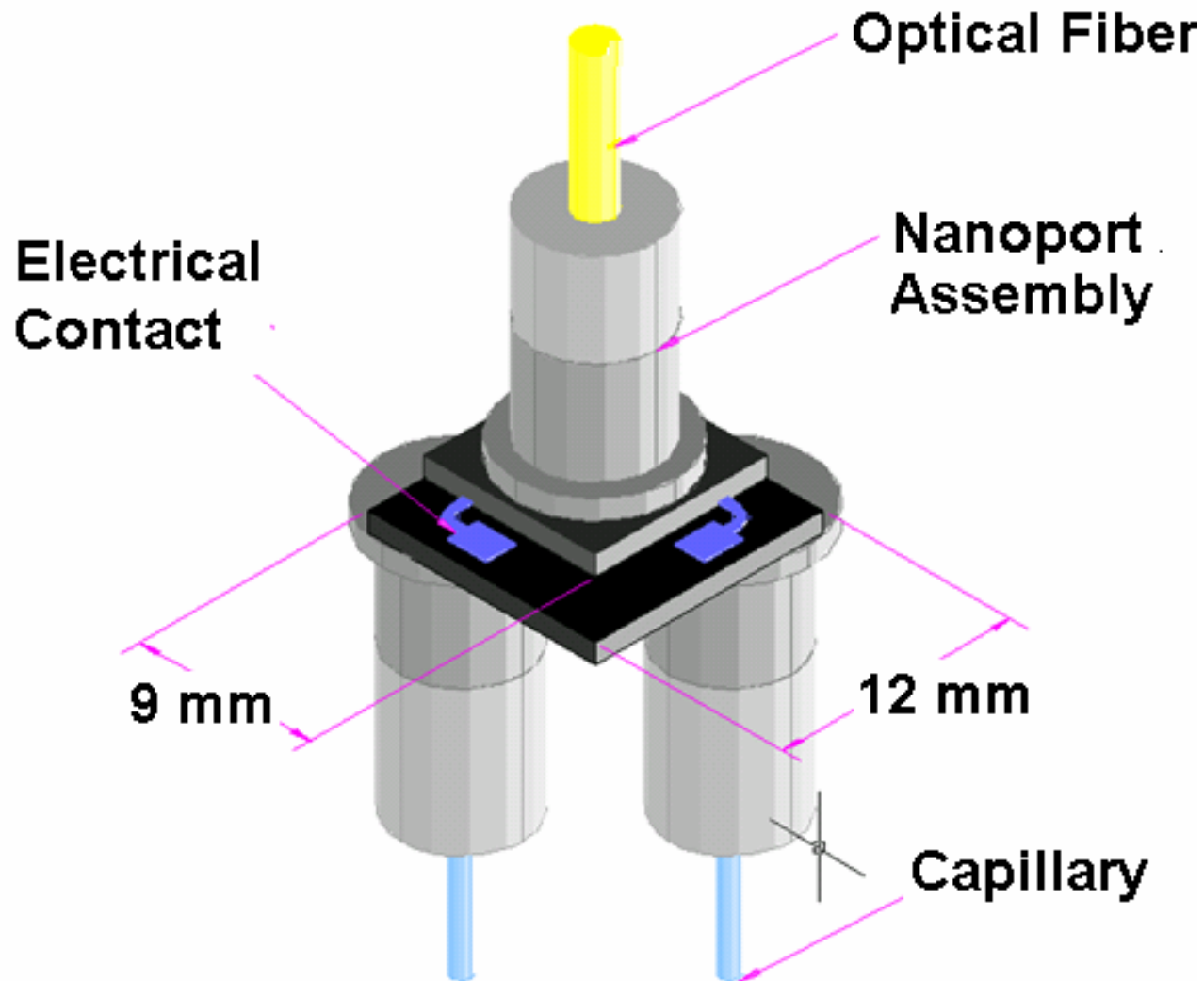


Fig. 3. MDD Trichloroethane Detection In Air. By Caviton, Inc.

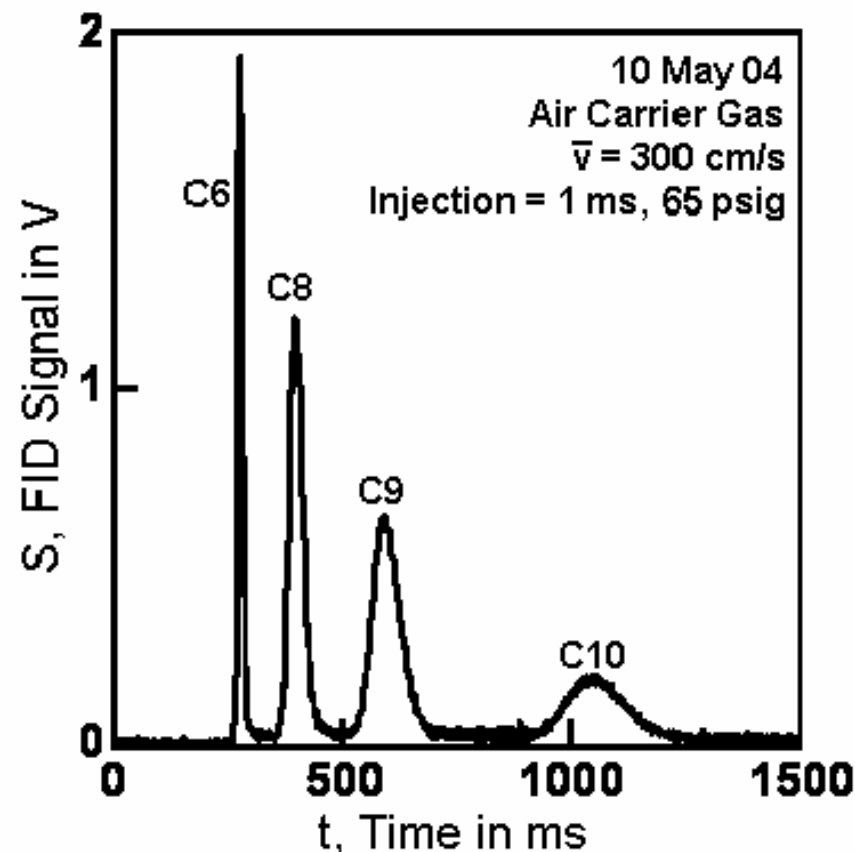
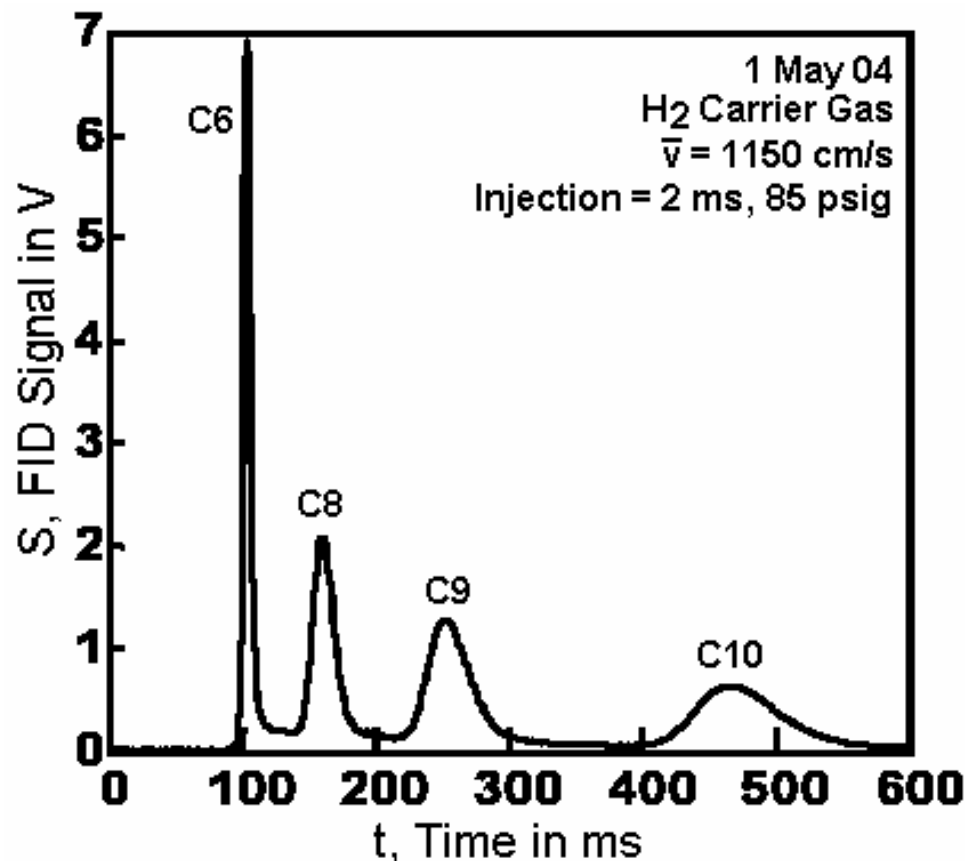
# Improved MDD Package Design

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# Separation Performance – Nanoglass, 2004

## •Separation Performance: H2 vs. Air Carrier Gas



### Separation of Hexane, Octane, Nonane and Decane.

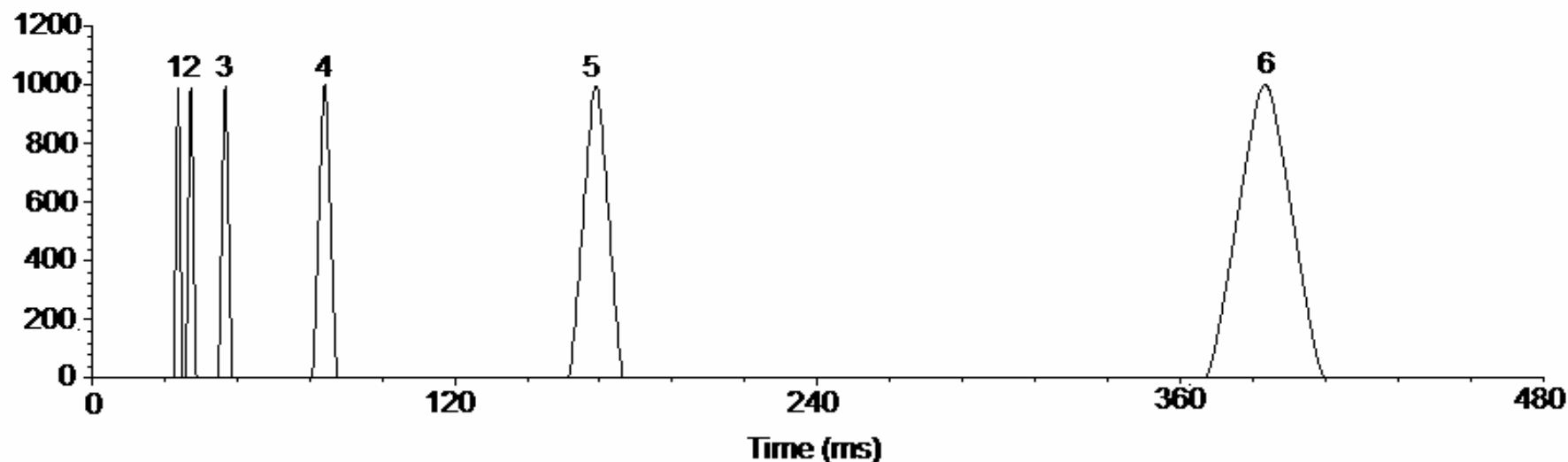
Capillary #3: 1-m in length, 100  $\mu$ m ID, 3  $\mu$ m film, 100°C, FID at 250°C, Data Rate: 20 kHz.

\*Evaluation Data Courtesy G.Gross and R.Synovec, CPAC



# Separation Performance Model – OV-5

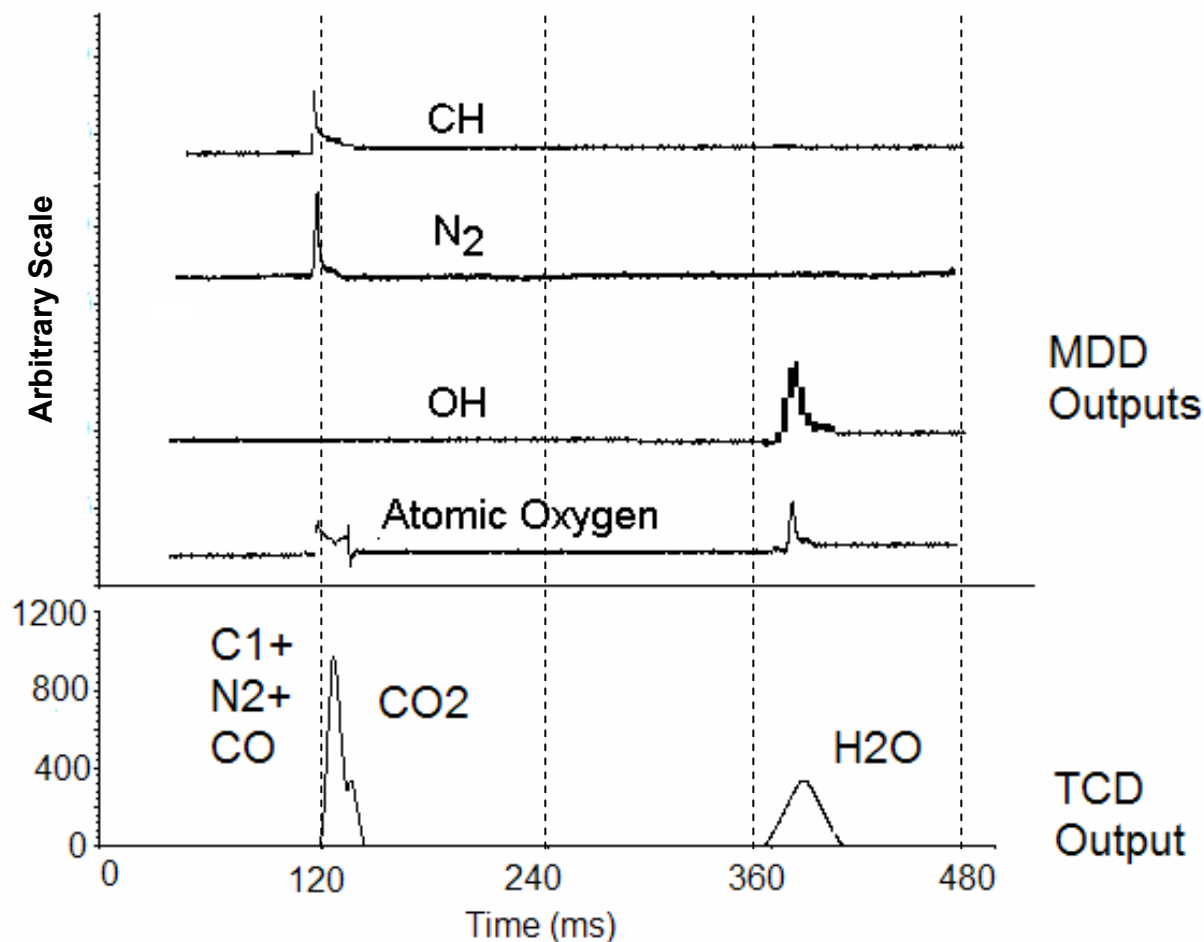
Hydrocarbon Application: Capillary: 25cm/100µm/400nm OV-5  
35 to 200 °C @200 °C/min; Linear Velocity: 100 cm/s



| #  | Component Name    | tR<br>(sec) | Width<br>(sec) | Rs    |
|----|-------------------|-------------|----------------|-------|
| 1. | Hydrogen Chloride | 0.28        | 0.0297         | 1.38  |
| 2. | C4 butane         | 0.33        | 0.0342         | 1.38  |
| 3. | C5 pentane        | 0.44        | 0.0460         | 3.11  |
| 4. | C6 hexane         | 0.77        | 0.0806         | 6.79  |
| 5. | C7 heptane        | 1.67        | 0.175          | 10.54 |
| 6. | C8 octane         | 3.88        | 0.376          | 12.11 |

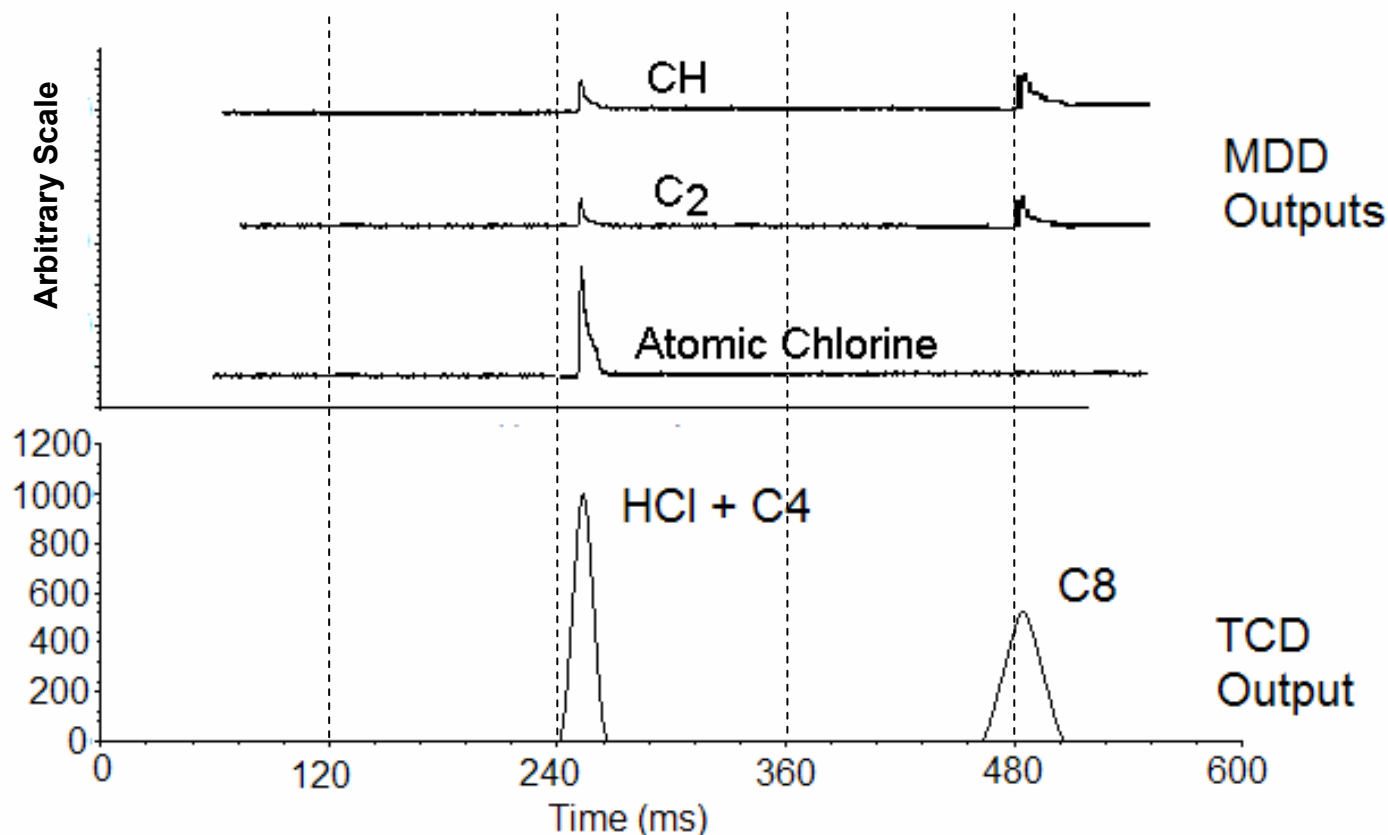
# Separation and MDD Output Model – Appl. 1

Air Products Application: Capillary: 25cm/100 $\mu$ m/100nm. OV-5  
30°C Linear Velocity 100 cm/sec



# Separation and MDD Output Model – Appl. 2

UOP Application: Capillary: 25cm/100 $\mu$ m/20nm OV-5  
30 °C Linear Velocity 100 cm/sec



# Barriers and Pathways

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## ■ PHASED:

- Presently available stationary phases (PDMS) were developed for coating capillary columns and not for MEMS processing and patterning for  $\mu$ GCs
- FID incompatible w/MEMS microminiaturization and portability

## ■ NeSSI:

- Communication protocol
- Availability of components and IS certifications

## ■ PHASED:

- Interim use of new, patternable nano-porous organo-silicate film; processing of PDMS now also possible, with better thickness control
- Alternatives such as TCD and MDD offer integratability, speed, sensitivity and greater orthogonal channel capacity (OCC)

## ■ NeSSI:

- USB to SAM, Ethernet to PC
- Use available, safe area components and limit test sites to safe areas

# Energy Savings

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- **How will energy be saved?** Industrial Process Plants benefit from in-situ sampling, more affordable and faster sensing, tighter control, fewer upsets, less product waste. We analyzed 8 specific applications to determine the total energy savings, resulting in:
  - **Tighter control of operational parameters: 78 TBtu/year**
  - **Upset avoidance: 3.7 TBtu/year**
- **Assumptions used in calc.:**
  - **Extrapolation based on ratio of energy used in example cases to energy used in the industry total**
- **How much energy will be saved?** The following slide shows the total for each example and the extrapolation
  - **Total savings for examples = 81.8 TBtu/year (62.0 w/o fired heaters); extrapolated total = 256 TBtu/year**



# Energy Savings Summary



Table 5. Summary of Energy Savings

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| Application                         | Ind.Group<br>(NAICS<br>Codes) | Type of Savings                   | Total Energy<br>Consumed<br>(MMBtu/yr) | Energy<br>Savings<br>(MMBtu/yr) | Energy<br>Savings<br>(%) | Total Energy<br>for Industry<br>Group<br>(MMBtu/yr) | Saving for<br>Industry<br>Group<br>(MMBtu/yr) | Energy<br>Savings<br>(%) | Sensors Required                                                                                                           |
|-------------------------------------|-------------------------------|-----------------------------------|----------------------------------------|---------------------------------|--------------------------|-----------------------------------------------------|-----------------------------------------------|--------------------------|----------------------------------------------------------------------------------------------------------------------------|
| Ethylene Prod.                      | 324110                        | Upset Avoid. &<br>Proc. Improv't. | 656,372,200                            | 3,692,094                       | 2.06%                    | 2,146,155,922                                       | 44,264,466                                    | 2.06%                    | Acetylene, CO, CO <sub>2</sub> , C <sub>1</sub> - C <sub>8</sub>                                                           |
| Metals Process.                     | 331, 332                      | Waste Avoidan.                    | 352,000,000                            | 35,200,000                      | 10.00%                   | 3,005,000,000                                       | 88,260,000                                    | 2.94%                    | H <sub>2</sub> O, CO, H <sub>2</sub> , CH <sub>4</sub> , O <sub>2</sub> , CO <sub>2</sub> , N <sub>2</sub> , THC           |
| H <sub>2</sub> Production           | 325                           | Proc. Improv't.                   | 283,202,150                            | 2,347,100                       | 0.83%                    | 6,064,000,000                                       | 50,256,731                                    | 0.83%                    | CO, CO <sub>2</sub> , H <sub>2</sub> , O <sub>2</sub> , N <sub>2</sub> , N <sub>2</sub> O, C <sub>1</sub> - C <sub>7</sub> |
| TiO <sub>2</sub> Drying             | 325                           | Proc. Improv't.                   | 888,462                                | 44,423                          | 5.00%                    | N/A                                                 | N/A                                           | N/A                      | H <sub>2</sub> O                                                                                                           |
| Fired Heaters                       | 324110                        | Proc. Improv't.                   | 1,524,240,000                          | 19,815,120                      | 1.30%                    | 4,983,844,078                                       | 64,789,973                                    | 1.30%                    | O <sub>2</sub>                                                                                                             |
| Al Production                       | 3313                          | Proc. Improv't.                   | 490,000,000                            | 7,350,000                       | 1.50%                    | N/A                                                 | N/A                                           | N/A                      | Al <sub>2</sub> O <sub>3</sub> :TC Ratio                                                                                   |
| Pulp Production                     | 322110                        | Proc. Improv't.                   | 83,904,000                             | 3,508,713                       | 4.18%                    | 198,000,000                                         | 8,280,000                                     | 4.18%                    | Inorganic Ions                                                                                                             |
| <b>TOTAL</b>                        |                               |                                   | <b>3,390,606,812</b>                   | <b>71,957,449</b>               | <b>2.12%</b>             | <b>16,397,000,000</b>                               | <b>255,851,170</b>                            | <b>1.56%</b>             |                                                                                                                            |
| <b>TOTAL INDUS.<br/>CONSUMPTION</b> |                               |                                   | <b>23,798,000,000</b>                  | <b>71,957,449</b>               | <b>0.30%</b>             |                                                     | <b>255,851,170</b>                            | <b>1.08%</b>             |                                                                                                                            |

# DoE Energy Savings Calculator Results



## Summary of DoE Energy Savings Calculator Results

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| Application                                  | Industry Group<br>(NAICS Codes) | Type of Savings                          | 2010 Savings<br>(TBtu) | 2015 Savings<br>(TBtu) | 2020 Savings<br>(TBtu) |
|----------------------------------------------|---------------------------------|------------------------------------------|------------------------|------------------------|------------------------|
| Ethylene Production                          | 324110                          | Upset Avoidance &<br>Process Improvement | 2.14                   | 10.26                  | 18.25                  |
| Metals Processing                            | 331, 332                        | Waste Avoidance                          | 2.45                   | 6.96                   | 16.20                  |
| Hydrogen Production                          | 325                             | Process Improvement                      | 0.52                   | 2.73                   | 5.35                   |
| TiO <sub>2</sub> Drying                      | 325                             | Process Improvement                      | 0.01                   | 0.04                   | 0.06                   |
| Fired Heaters                                | 324110                          | Process Improvement                      | 2.14                   | 10.26                  | 18.15                  |
| Aluminum Production                          | 3313                            | Process Improvement                      | 0.39                   | 1.12                   | 2.61                   |
| Pulp Production                              | 322110                          | Process Improvement                      | 0.45                   | 2.06                   | 3.49                   |
| Refrigerant Production                       | 325                             | Upset Avoidance                          | 0.00                   | 0.02                   | 0.04                   |
| <b>TOTAL</b>                                 |                                 |                                          | <b>8.11</b>            | <b>33.44</b>           | <b>64.14</b>           |
| Petroleum Refining                           | 324110                          | Upset Avoidance &<br>Process Improvement | 18.59                  | 86.48                  | 149.36                 |
| Metals                                       | 331, 332                        | Waste Avoidance                          | 4.41                   | 12.54                  | 29.20                  |
| Chemicals                                    | 325                             | Process Improvement                      | 7.95                   | 38.13                  | 67.83                  |
| Pulp Mills                                   | 322110                          | Process Improvement                      | 1.06                   | 4.84                   | 8.19                   |
| <b>TOTAL</b>                                 |                                 |                                          | <b>32.01</b>           | <b>141.99</b>          | <b>254.58</b>          |
| % of 1998 TOTAL<br>INDUSTRIAL<br>CONSUMPTION |                                 |                                          | <b>0.13%</b>           | <b>0.60%</b>           | <b>1.07%</b>           |

# Anticipated Economic Impact



- **For Helium and Hydrogen Production (one of two test sites)**
  - Total energy savings = 5.35TBtu in 2020 (**\$32M/year**)
  - Total emissions reductions in 10<sup>6</sup> lbs/y:  
193 - Carbon, 1.419 - NO<sub>x</sub>, 0.315 - CO, 0.031 - VOCs and  
0.014 - Particulates
- **For All Industries**
  - Energy Savings of = 254TBtu in 2020 (**\$1.5B/year**)
  - Total emissions reductions in 10<sup>6</sup> lbs/y:  
10233 - Carbon, 74.63 - NO<sub>x</sub>, 12.28 - CO, 1.663 – VOCs and  
1.885 - Particulates
- **For Honeywell, such data will be presented in the private session**

# Value Proposition for End User

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## PHASED $\mu$ GC on NeSSI-II provides:

- Availability, for the 1<sup>st</sup> time ever, of a GC **analyzer function in the form and cost of a sensor** (size, speed, simplicity, ruggedness, power, maintenance, environmental protection, cost)
- Greater affordability, speed and sensitivity, which equates to tighter process control, **energy savings** and **lower product cost**
- Wired or wireless communication with its dedicated SAM (Sensor-Actuator Manager, a local computer), which can have a **wireless interface** of **low installation cost** with the plant control room and with other plant sensors.

# Other Important Metrics

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of the **PHASED** mGC:

- **Compactness** ~ 12 in.3 ~ 250 cm<sup>3</sup> to fit on NeSSI
- **Speed:** Analysis time < 2 s, incl. pre-concentration
- **Sensitivity:** MDL < 0.1 ppm / PC Gain
- **No carrier gas**
- **No mechanical injection valve**
- **100-300x lower power (2-5 W) than desk-top GC**
- **10x lower cost**

of the **NeSSI** systems:

- **Standard modules of 1.5 x 1.5" foot-print (SP-76)**
- **4 SP-76 spaces for microanalytics**
- **Communication with SAM**



# Completed & Future Milestones

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- Preconcentration feasibility 3/31/04
- Separation feasibility 3/31/04
- Demonstrated 90% wafer yield 5/ 1/05
- NeSSI package design 4/30/06
- PHASED on SP-76 Demonstration 6/30/06
- PHASED on NeSSI lab test 7/31/06
- PHASED on NeSSI field test start 9/30/06
- PHASED on NeSSI field test compl. 4/30/07
- Final Report 6/30/07

# Project Status

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| Milestone                         | 2003      | 2004 | 2005     | 2006      | 2007      |
|-----------------------------------|-----------|------|----------|-----------|-----------|
| Project Start                     | $\Delta$  |      |          |           |           |
| Preconcentration feasibility      | $-\Delta$ |      |          |           |           |
| Separation feasibility            | $-\Delta$ |      |          |           |           |
| Development of microstructures    | —————     |      |          | $\Delta$  |           |
| Demonstrated 90% wafer yield      |           |      | $\Delta$ |           |           |
| NeSSI package design              |           |      | —————    | $\Delta$  |           |
| PHASED on SP-76 Demonstration     |           |      |          | $-\Delta$ |           |
| PHASED on NeSSI lab test          |           |      |          | $-\Delta$ |           |
| PHASED on NeSSI field test start  |           |      |          | $\Delta$  |           |
| PHASED on NeSSI field test compl. |           |      |          | —————     | $\Delta$  |
| Final Report                      |           |      |          |           | $-\Delta$ |

# Accomplishments to Date

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## ■ Prior Years

- Demonstrated high-speed  $\mu$ GC pre-concentrator & separation
- Demonstrated separation of H<sub>2</sub>O, CH<sub>4</sub>, C<sub>3</sub>H<sub>8</sub>, C<sub>4</sub>H<sub>10</sub>, CO<sub>2</sub>, ....., C<sub>9</sub>, C<sub>10</sub>
- Modeled feasibility of separation up to C<sub>18</sub>, w/temp. ramp
- Calculated adsorption enthalpies on dry & hydrated films
- Demonstrated PC gain of 63x for hexane, and ~100x for CO<sub>2</sub>; modeled PC gains >10,000 for high MW compounds
- Increased PHASED chip fabrication yield to >90%
- Determined that a complete PHASED  $\mu$ GC with electronics will fit into one NeSSI's SP-76 modules
- Agreed with Users to base NeSSI device communication on Wireless or FieldBus; *however, since NeSSI components to support this are not yet available the field test uses USB*

# Accomplishments to Date

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## ■ Current year

- Identified 2 test sites and applications; agreed with field test participants on gases to be pre-concentrated, separated and detected
- Completed the design and fabrication of 2 PHASED  $\mu$ GC packages for NeSSI
- Improved preconcentration with heated connections
- Improved separation with OV-5 thin film material
- Improved TCD with thermal isolation
- Integrated MDD into PHASED-on-NeSSI for 2-D detection

# Next Project Steps

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## ■ Current Year (Cont)

- Started development of a Chemical Impedance Detector as an alternative to the TCD
- Developed electronics for PHASED in NeSSI package
- Designed, started programming and debugging the PHASED- $\mu$ GC output software
- Designed, programmed and debugged the PHASED-NeSSI-SAM interface software
- Developed commercialization plan for all markets

## ■ Remaining

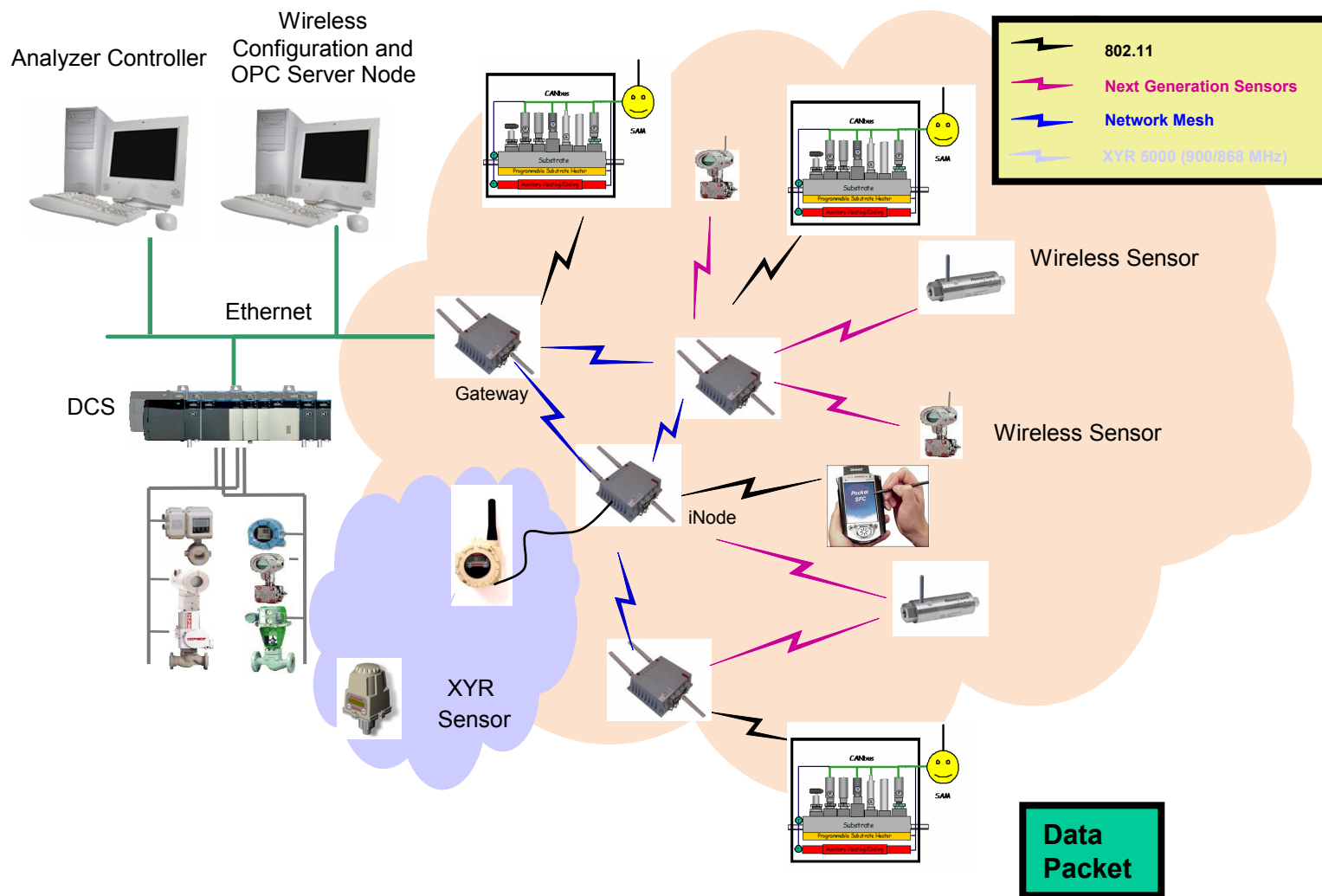
- Complete the assembly, testing and setup of 2 PHASED  $\mu$ GC packages for field test
- Start and complete field tests
- Final report

# Continuation after ITP-Sponsored Project

- The technology developed under this project will be commercialized, provided the **PHASED-on-NeSSI field tests** are completed successfully.
- Discussions with BUs about PHASED and NeSSI manufacturing, and marketing, distribution, sales and service have been held and internal Honeywell funding is being sought to deploy PHASED into a wide market area.



# PHASED MGA with Wireless Communications



# Technical Risks Remaining

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- **PHASED Applications:** Demonstrate the ability of PHASED to perform the needed separations, sensitivities and service life
  - Reaching a broad set of applications requires a selection of column phase materials. To date, we have successfully deposited two (OV-5 and Nanoglass). Some form of granular material such as a HayeSep variant, Molesieve, or a CarboPack material would be helpful, but experiments to date have not produced sufficiently smooth depositions.
  - For analysis of liquids, the development of a PHASED vaporization capability would be needed
- **Sampling via NeSSI:** A family of appropriate NeSSI components must exist. This is currently assumed but is developing very slowly – however, PHASED can be deployed without NeSSI.
- **Certifications:** Appropriate certifications must be met for each major market. An IS version of NeSSI may evolve; demonstrate an IS version of PHASED.

# Commercialization Plan

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- PHASED represents a new and significant commercial opportunity to Honeywell and its customers.
- A partnership with Parker-Hannifin is in place to **develop, integrate and commercialize NeSSI hardware** and its communication links (wired and wireless).
- Microfabrication of PHASED and its electronics will be transferred to one of **three in-house Si-foundries**
- Further development of PHASED as a commercial set of products will be transferred to **Business Units** that have strong positions in industrial, air quality, medical and defense applications.
- Both NeSSI and non-NeSSI products are envisioned from PHASED.
- A more detailed presentation is scheduled for the private session

# Competitive Comparison – Traditional Suppliers

Comparison between PHASED and Traditional Competitors

|                  | Units  | PHASED                | Baseline                | Emerson 700             | Siemens uSAM  | Yamatake                | ABB                     |
|------------------|--------|-----------------------|-------------------------|-------------------------|---------------|-------------------------|-------------------------|
| Approach         | -      | GC in Si              | Std. GC                 | Standard GC             | Standard GC   | BTU GC                  | Standard GC             |
| Integration      | -      | PCB                   | Portable/Bench          | EXP Box                 | EXP Box       | Exp Box                 | Purged                  |
| Preconcentration | Stages | 20-50-Stages          | None                    | None                    | None          | None                    | None                    |
| Injection Method | -      | 2-6-ms Thermal Desorb | Valve                   | Mechanical              | Valveless     | Mechanical              | Mechanical              |
| Volume           | nL     | < 50                  | 500?                    | 500+                    | 500+          | 500+                    | 500+                    |
| Carrier Gas      | Gas    | None/Air              |                         | H2, He or N2            | H2, He or N2  | He                      | H2, He or N2            |
| Size             | -      | 2 NeSSI Slots         | 18 X 16 X 9"            | 15"x7"x36"              | 10"x9"        | 3.9"x4.5"x9.6"          | 19.5"x13.4"x46.3"       |
| Analysis time    | sec    | 1 to 4                | 60-300                  | 60-300                  | 60-300        | 300                     | 60-300                  |
| Column Length    | cm     | 10 to 25              | Various                 | Various                 | Various       | Various                 | Various                 |
| Col./Channel ID  | µm     | 100                   | Std Capillary or Packed | Std Capillary or Packed | Std Capillary | Std Capillary or Packed | Std Capillary or Packed |
| Film Thickness   | µm     | <0.1                  | Various                 | Various                 | Various       | Various                 | Various                 |
| Stationary Phase | -      | OV-5                  | Many                    | Many                    | Many          | Several                 | Many                    |
| Column Temp.     | C      | 25 - 400              | 25-150                  | 25-100                  | ??            | ??                      | 25-289                  |
| Temp Program     |        | Yes                   | No                      | No                      | No            | No                      | Option                  |
| Detector         |        | TCD, Ion Trap, MDD    | PID, FID, TCD           | TCD, FID                | TCD           | TCD                     | TCD, FID, FPD           |
| Flow Rate        | sccm   | 0.5 - 1               | Appl Dep                | Appl Dep                | Appl Dep      | 50                      | Appl Dep                |
| Energy/Analysis  | J      | 1 to 10               | ?                       | 1980 Min                | 900 Min       | 7500 Typ                | 50,000 Min              |

# Competitive Comparison – Emerging Devices

Comparison between PHASED and Emerging Competitors

|                  | Units  | PHASED                   | SLS           | AFSI                         | Sandia                       | Graf Z-Nose             |
|------------------|--------|--------------------------|---------------|------------------------------|------------------------------|-------------------------|
| Approach         | -      | GC in Si                 | GC in Si      | GC in Si                     | GC in Si                     | Std. GC                 |
| Integration      | -      | PCB                      | PCB           | Portable                     | Portable                     | Portable/Bench          |
| Preconcentration | Stages | 20-50-Stages             | None          | Yes                          | Yes                          | Yes                     |
| Injection Method | -      | 2-6-ms Thermal<br>Desorb | Mechanical    | Pump, Thermal<br>Desorb      | Thermal Desorb               | Valve/Thermal<br>Desorb |
| Volume           | nL     | < 50                     | 500           | ?                            | ?                            | ?                       |
| Carrier Gas      | Gas    | None/Air                 | H2, He or N2  | H2, He or N2                 | Air                          | He                      |
| Size             | -      | 2 NeSSI Slots            | 3 x 5" PCB    | 16 X 9 X 5"                  | 8 X 4 X 2"                   | 12 X 10 X 7"            |
| Analysis time    | sec    | 1 to 4                   | < 60          | < 60                         | 30-60                        | < 60 Cycle              |
| Column Length    | cm     | 10 to 25                 | 86            | 500 - 2000                   | 100                          | ?                       |
| Col./Channel ID  | μm     | 100                      | ~100          | 100 - 200                    | Micro-machined,<br>50-100    | ?                       |
| Film Thickness   | μm     | <0.1                     | ?             | ?                            | Variable                     | ?                       |
| Stationary Phase | -      | OV-5                     | Several       | DB-1, DB-5,<br>DB-54, DB-Wax | Several, coated<br>or packed | ?                       |
| Column Temp.     | C      | 25 - 400                 | 25 - 250      | 25 - 350                     | 25 - ?                       | 25-125                  |
| Temp Program     |        | Yes                      | Yes           | Yes                          | Yes                          | Yes                     |
| Detector         |        | TCD, Ion Trap,<br>MDD    | FID           | Glow Discharge,<br>TCD       | SAW                          | SAW                     |
| Flow Rate        | sccm   | 0.5 - 1                  | 0.1 @ 30 psia | ?                            | ?                            | Appl Dep                |
| Energy/Analysis  | J      | 1 to 10                  | 480           | ?                            | ?                            | ?                       |

# Overview of Commercialization Plan

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- **The Commercialization Plan covers many aspects of getting PHASED from a government funded research project to a series of product development projects, and then into commercial production**
- **The areas covered are**
  - Market size estimates
  - Manufacturing approach
  - Industrial markets (refining, petrochemical and chemical; natural gas; mining, metals and minerals; pharmaceutical)
  - Air Quality markets (buildings, commercial aircraft and military platforms)
  - Handheld (chemical weapons detection and medical breath diagnosis)



# Commercial Risks Remaining

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- **New MicroAnalytics:** Despite past sales of industrial GCs, launching new microanalytics devices requires **investment** to establish marketing, distribution, sales, and service support, especially across the number of markets identified for PHASED
- **NeSSI:** Wide adoption of NeSSI in the conservative industrial marketplace is slow. According to a mini-VOC conducted by Honeywell, support for NeSSI at the plant level is still emerging; this Project could have helped to provide more of the needed field test data, before cutting back from 12 to 2 NeSSIs. Without a standard sampling systems such as NeSSI, deployment at many measurement points may require some additional packaging development.
- **Competition:** Competitors are continuing to develop products that could threaten PHASED. We are tracking a number of both new and established competitors. However, many customers demand multiple-source supply.

# Showcasing Opportunities

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- **Two field tests are being prepared and will be in place by October 2006**
  - Air Products, **Helium impurities**, in Manalapan, NJ
  - UOP (Honeywell), **HCl in alkanes**, in Des Plaines, IL

# Addendum – Discussion Items If Needed

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- **The following slides are provided as backup only**

# Electronics for PHASED on NeSSI

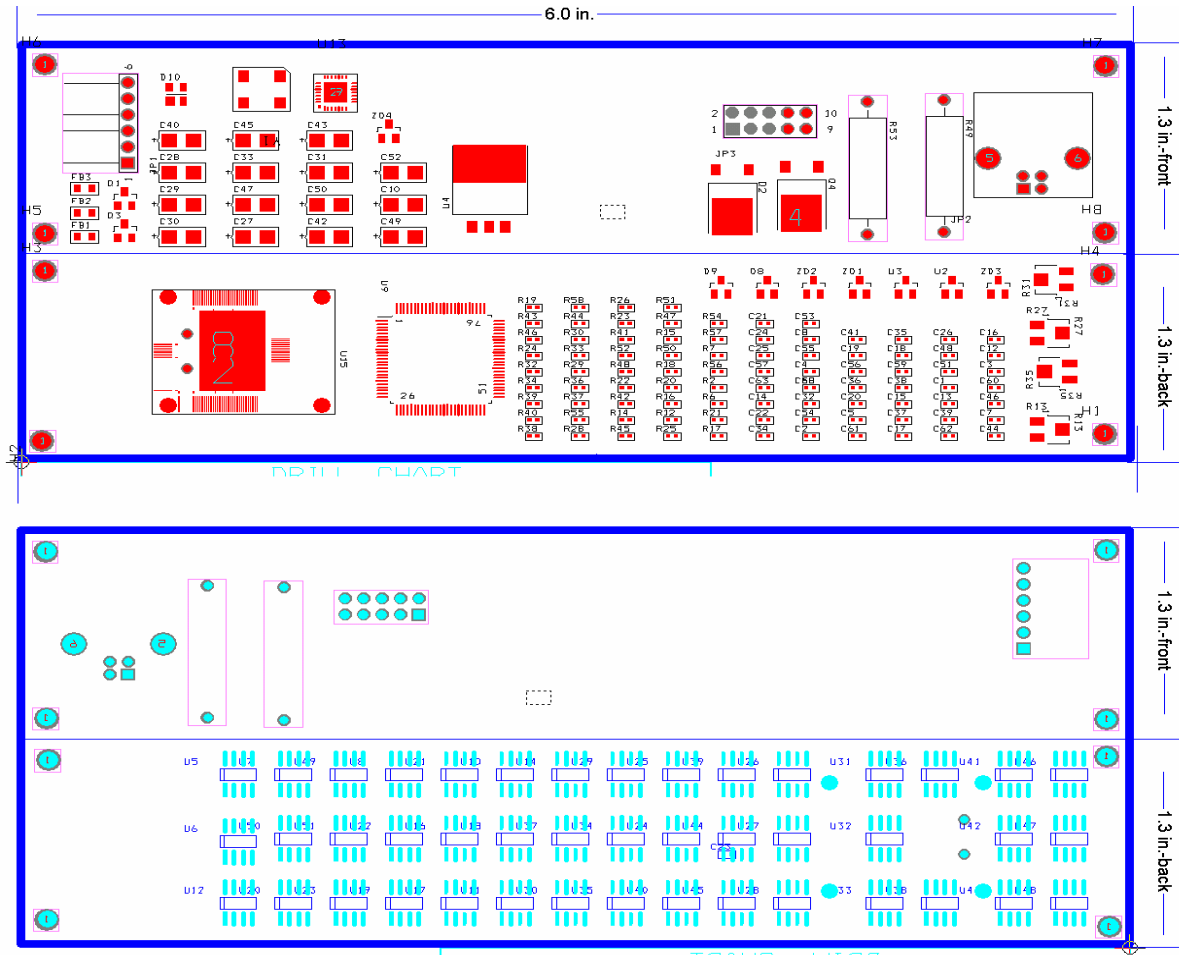
## Features:

- Individual control for concentrators, up to 30
- Integrated 12-bit A/D, up to 8 channels
- TCD, flow, CID, and temperature sensor circuits
- Dual power supply (+10V up to +30V/-5V down to -12V)
- USB/RS232/SPI
- Ramp up temperature of separators
- Variable voltage for separators and PCs
- All package components are standard, except for PHASED chip

## Issues:

- Coupling of capillary from PHASED chip to detectors and pump
- Circuits for MDD and MESO-Pump are not included (see next slide)

**2 (6X1.3 in) boards -1.5 cm apart**



Circuit by F.Nusseibeh

# Validation of Separation Model

Capillary Column: **100cm/100 $\mu$ m/0.4 $\mu$ m-DB-5, 125°C**  
**146 cm/s H<sub>2</sub>, 0.69 mL/min at 8.5 psi, W<sub>inj</sub>= 2 ms.**

