Examples of Advances in Measurement Science that are Enhanced by CPAC's New Sampling/Sensor Initiative (NeSSI™)

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Analytical Sampling for Online Applications
What is NeSSI?

• Industry-driven effort to define and promote a new standardized alternative to sample conditioning systems for analyzers and sensors
  • Standard fluidic interface for modular surface-mount components
    • ISA SP76
  • Standard wiring and communications interfaces
  • Standard platform for micro analytics
What does NeSSI Provide

- Simple “Lego-like” assembly
  - Easy to re-configure
  - No special tools or skills required
- Standardized flow components
  - “Mix-and-match” compatibility between vendors
  - Growing list of components
- Standardized electrical and comm. (Gen II)
  - “Plug-and-play” integration of multiple devices
  - Simplified interface for programmatic I/O and control
- Advanced analytics (Gen III)
  - Micro-analyzers
  - Integrated analysis or “smart” systems
Benefits of Being NeSSI

- Your condition space is constrained
  - Volumes, flow rates, pressures, and viscosities inherently bounded by architecture

- Your interfaces are defined
  - Electrical power, communication, and sample are all available in a standard way
  - Power budget could be main driver to miniaturize

- Your sample conditioning can be defined and controlled
  - Specify up-stream and down-stream NeSSI components (and verify)

- All this means your analytical can be more directed and focused
Where Does NeSSI Fit in the Lab

- **Instrument/Sensor Interfaces**
  - Design standards make development simpler
    - Reduced toolset to be mastered
    - Reduced sample variability to account for
  - Calibration/validation built-in
    - Consistent physical environment for measurement
    - Stream switching and/or mixing allow generation of standards to match analytical requirements

- **Reaction monitoring**
  - Microreactors and continuous flow reactors
  - Batch reactors (with fast loop)

- **Sample Preparation**
  - Gas handling (mixing, generation, delivery)
  - Liquid handling (mixing, dilution, conditioning, etc.)
NeSSI with an Array of Micro-Analytical Techniques will Impact Many Industries

- Process Control
- Process Optimization
- Product Development
NeSSI™: Enabler for MicroAnalytical

- (the “rail” concept)

**Standard Electrical (Digital) Interface “Rail”**

- Anyone’s Sensor
- Anyone’s Actuator

**Standard Mechanical Interface “Rail”**

- *Sensor/Actuator Manager

**Standard “connectivity”**

- SAM*

What technologies are available
Suitability for modular sampling systems
NeSSI Sensing Technologies

- Gas Chromatography
  - Thermal Desorption
- Dielectric
- Spectroscopies
  - IR
  - NIR
  - UV-Vis
  - Raman
  - Fluorescence
- Impedance
- Conductivity
- Refractive Index

- Particle Sizing
  - Light scattering (?)
- Turbidity
- pH
- RGA
- Mass Spectrometry
- LC, SEC, IC
- Terahertz (?)
C2V Fast Micro-GC

http://www.c2v.nl/
Interfacing NeSSI™ to Falcon μFast GC™

- Complete gas/vapor sensing test platform on the bench top
  - Gas delivery, vapor generation, and blending in NeSSI™
  - Real time verification of composition using GC and EP-IR
- Easily extended to include other analytical and sample treatments
At-Line GC’s with NeSSI Compatibility

ABB Natural GC

Siemens microSAM

Agilent 3000 Micro GC
Applied Analytics Inc. Diode Array

- OMA-300
- A Fiber-optics-diode-array process analyzer
- For on-line concentration monitoring
Applied Analytics Microspec IR

FEATURES

- Ideal for monitoring PPM level WATER in various solvents
- In stream quantitative measurements
- Contains no moving parts and
- Extremely robust allowing for installations in process stream environments
- Replaces analyzers such as process spectrometers in the process plant.
Sentelligence Current NIR Sensors

- NIR Sensors
- Removable Tip Version
Small Diameter ATR-IR-Fiber Probes
Fiber Photonics Inc.

Diamond ATR-IR-Fiber Probe
of 2.7mm diameter in NeSSI

www.fibrephotonics.com
H$_2$Scan Adaptation to NeSSI™ Platform

- Hydrogen specific: 0.5% H$_2$ to 100% H$_2$ v/v
- Response time (T90) < 30 sec
- In-line, real-time measurements in process gas streams up to 100°C
- Unique models for CO, H$_2$S, wet CL$_2$
- 4-20mA, RS422 or RS232 serial connectivity
- Stable results
- On-site verification and calibration
- Approved for hazardous locations
  - Intrinsically safe design
  - ATEX certificate granted; UL pending
- Cost effective to buy / install / maintain
- Field verification and calibration kit available

http://www.h2scan.com/
Astute Sampling System

Sampling + Sensors & micro-analyzers

Astute System with C₂V Micro GC and H₂Scan hydrogen analyzer

GAS TREATMENT COLUMN

www.eif-filters.com
Agilent NeSSI Dielectric Sensor

Cable to Agilent Network Analyzer

Swagelok 2-Port Valve Base

Dielectric Probe

Inner Body

O-ring (inside)

Outer Body

Exploded View

Close up of Coaxial Probe Tip

Close up of Coaxial Probe Tip
NeSSI™ IR Gas Cell

NDIRs. The Star NDIR has no moving parts.

- No Moving Parts or Special tools required for Easy Maintenance & Service
- No Critical Realignment
- Specific, Interference-Free CO₂ Detection
- Dual-Wavelength Ratioint Compensates for Drift
- Computer-Controlled for Accuracy
- Sapphire-Protected Optics
- Non-Corrosive, Non-Reflective Sample Cell
NeSSI Compatible Optical Cells

Axiom Analytical, Inc.

- Currently Available
  - FFV Series Transmission Cells (Near-IR, UV-Visible)
  - FNL-120 UV-Visible ATR Cell
- In Development
  - Raman Cells (Single- and Multi-pass)
- Possible Development
  - Diffuse Reflectance Cells
  - Mid-IR ATR Cells

Courtesy of Mike Doyle
Axiom Analytical, Inc.
Chevron NeSSI/Analytical Unit

Specifications

- ASI Microfast GC
- Parker NeSSI gas handling/sampling system
- Carrier gas
- Mobility
NeSSI based Analytical Developments at CPAC
NeSSI Ballprobe - Raman/NIR/UV

Matrix Solutions: www.ballprobe.com
Power loss = 2x Sampling error

Power out – 92%  Power out - ~100%

> 4%  <<1%

Sampling error

Power loss = 2x Sampling error

Raman Sampling Errors
Raman Ball Probe

- no moving parts
- sapphire spherical lens
- constant focal length and sample volume
- focus is at tangent of sphere
- probe is **ALWAYS** aligned when in contact with sample
- effective sampling of liquids, slurries, powders, pastes and solids
- high sampling precision allows it to be used effectively to monitor dynamic mixing systems (powders/slurries)
- particle size has minimal effect on optical performance (< 1μm – 5mm)
NeSSI Raman Sampling Block

- Parker Intraflow NeSSI substrate
- Sample conditioning to induce backpressure to reduce bubble formation and the heated substrate allows analysis at reactor conditions
NeSSI Gas/ Vapor System
NeSSI Calibration Gas Generation System

Features of NeSSI System:
- 4 Stage dilution, able to produce and maintain gas concentrations in ppb range
- Fully automated system, set and forget capability
Gas Mixing System LabView Control Program
Design of Small Fiber Optic Sensors

- Vapochromic chemistry
  - Optical response to analyte
- Simple design
- Reversible response
- Low power
- Inexpensive
- Fast response times
- High quantum efficiency
- Long term sensor stability
- Sensitive to a variety of analytes
- Wireless communication
- Battery powered

Oxygen
Moisture
Ammonia
Hydrogen
Common Solvents
  - Alcohols
  - Esters
  - Amines
Chlorinated Organics
Organic Hydrocarbons (BTEX)
Carbon Dioxide (in development)
Hydrogen Sulfide (in development)
Sensor Applications

Applications

1. Monitor Biological Processes (Fermentors, Proteomics)
2. Monitor Industrial Processes (Reaction Vessels, Corrosion)
3. Monitor Ocean Processes (Argo floats, SeaGliders, Moorings)
4. Area Monitors (airplanes, subs, hazardous works areas)
Vapochromic Humidity Sensor

- Measurement time – 100 ms
- 3 reps per concentration

- 2 PC calibration model
- Humidity range: 10 – 80%
- Ohmic feedback control

Humidity generator used for reference stds.

Elements: 6
Slope: 0.999824
Offset: 0.007585
Correlation: 0.999912
RMSEC: 0.342323
SEC: 0.374996
Bias: -1.272e-06

Measured Y vs Predicted Y
Vapochromic Benzene Sensor

Full spectrum response of the 0%, 10%, and 50% bubbler flow samples used to make the PLS model showing both the change in intensity and shift in peak maximum with changing benzene concentration.
Sensor response to $O_2$

20 replicates at each concentration
Concentration range: 0 - 100% Oxygen

$R^2 = 0.990, 3$ PC
RMSEC = 1.0744

![Graph showing sensor response to O2 with calculated O2% and predicted O2% on the y-axis, and calculated O2% on the x-axis. The graph includes data points and a trend line indicating a strong correlation between calculated and predicted O2% values.]
Liquid Chromatography for NeSSI™

Scott Gilbert, CPAC Visiting Scholar
Crystal Vision Microsystems LLC
Atofluidic Technologies, LLC

- Split flow approach to sampling
- \(\mu\)-fluidic LC Chip for On-line Sample Pretreatment
- Pulsed electrochemical detection (on-chip)
Development of a Micro-NMR System

RF Microcoil
MicroChannel Sample Chamber
Bonding Pads

M. McCarthy, UC Davis
Fringing Field Dielectric NeSSI Sensor

Alex Mamishev EE and Marquardt CPAC, UW
Calibrated Gas Generation

Full NeSSI Model

Analyte Side

Gas Phase Side
Application of Permeation Apparatus
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