Water and Food Analytical Laboratory (WAFAL)

- WAFAL maintains a comprehensive analytical laboratory dedicated to analyzing spaceflight water and food samples and providing data that forms the basis for critical operational decision making regarding water quality and crew health.

- WAFAL scientists and engineers support in-flight operations of the environmental health systems (EHS) and environmental control and life support systems (ECLSS) on Shuttle and ISS.

- WAFAL staff develop and evaluate technologies for in-flight water quality monitoring.

ICP/MS analysis of trace metals and minerals

ISS Total Organic Carbon Analyzer (TOCA)

Colorimetric Water Quality Monitoring Kit (CWQMk)
Why Monitor the Spacecraft Environment

Ensure crew safety!

- Monitor performance of regenerative ECLSS
- Atmospheric revitalization
  - Removal of CO₂ and contaminants
  - Electrolytic generation of O₂
- Water Recovery
  - Condensate
  - Urine distillate

Resupply opportunities exist in low earth orbit, however, as mission objectives become more ambitious resupply will not be feasible. Creates a need for regenerative ECLSS systems that can be sustained long term with no ground support.

Utilize ISS to evaluate new technologies

Types of Water on the ISS

Ground Supplied/Stored Water
- Water prepared on ground (or in-flight)
- Delivered on resupply vehicles (Shuttle, Progress, ATV, HTV)
- Transferred into storage tanks and containers
- Supplements recovered water, may be stored up to a year prior to use

Recovered Water
- Condensing heat exchangers remove moisture from the atmosphere, condensate is processed into potable water (US and Russian segments of ISS)
- Preserved urine is distilled, distillate is recovered and processed into potable water (US segment only)
- Primary potable water source, used as it is generated
Maintaining Water Quality

Environmental Contaminants

Tank/Storage Container Materials

Byproducts of Processing

Pollutants in Source Water

Biocides and Preservatives

Current Approach to Water Quality Monitoring

• Limited in-flight analysis capability
• Relys heavily on archive samples
  • Collected monthly
  • Stored on the ISS until return on Shuttle or Soyuz vehicle

Challenges with Current Approach
• Time lapse between collection and analysis
• Irregular return frequency
• Limited return volume
• Degradation during storage
Drivers for Change

• Looming Shuttle retirement
  • Significant reduction in launch and return payload capacity
    • Difficult to resupply consumables for environmental monitoring
    • 1L archive water bag weighs ~ 2 pounds!
  • Samples returned less frequently

• Uncertain future of Constellation Program
  • Rely on Soyuz vehicles for sample return
    • Reduction in archive sample volume
    • Added complication of returning samples to US after landing

• 6 crew habitation on ISS
  • ECLSS systems needed to support a 6 person crew have up and downmass needs
  • Environmental sampling hardware must compete for limited payload capacity with other critical resupply items (such as food) needed to support a 6 person crew

Opportunity

Situation highlights the need to develop systems capable of monitoring the spacecraft environment in-flight.

Limited up-mass, space constraints, power requirements, safety concerns preclude deployment of most analytical instrumentation on-orbit.

- Small
- Rugged
- Reliable
- Provide direct readout of results
- Contain no hazardous materials
- Function in zero gravity

- Lightweight
- Simple to operate
- Sensitive
- Minimize waste generation
- Minimal power consumption
- Address multiple needs

Utilize ISS to evaluate new technologies
Color in Analytical Chemistry

Qualitative Analysis

• Identification of compounds based on appearance
• “Qual Scheme”
• Flame Test
• Chromatography

Quantitative Analysis

• Test strips/colorimetric indicators
• Modern spectroscopic instruments
CSPE Technology

1. Impregnate membranes with colorimetric reagent
2. Cut membrane into 13 mm disks and load into filter holder
3. Withdraw sample using syringe
4. Pass sample through analysis cartridge loaded with membrane disk
5. Acquire reflectance spectrum of disk with portable spectrometer
Analyte Quantification

Relative reflectance ($R$) is related to concentration by the Kubelka-Munk function

$$F(R) = (1-R)^2/(2R) = \varepsilon C / s$$

$\varepsilon$ is the molar absorptivity, $C$ is concentration, and $s$ is a scattering coefficient.

Assuming $\varepsilon$ and $s$ are constant, $F(R)$ is directly proportional to analyte concentration.

Technology Demonstration

SDTO experiment #15012-U, “Near Real-time Water Quality Monitoring Demonstration for ISS Biocides Using Colorimetric Solid-Phase Extraction (CSPE)” was delivered to ISS in August 2009 aboard STS-128/17A.

- Colorimetric Water Quality Monitoring Kit (CWQMK)
- Planned duration 6 months
  - Extended to 21 months
- Evaluate the acceptability of CSPE technology for routine in-flight water quality monitoring on ISS
  - Monitoring biocides in water supplies on the ISS
    - Ionic silver ($\text{Ag}^+$)
    - Molecular iodine ($\text{I}_2$)
Hardware Description

- Spectrophotometer (2)
- Sample bags
- Sampling adapters
- Waste bags
- Silver/Iodine analysis syringes
- Silver/Iodine analysis cartridges
- Calibration standards
- Data cables
- Wipes
- Note cards
- Standard solutions

Need for Biocide Monitoring

- 5,000 ppb
  Health concern short-term exposures

- 500 ppb
  Health concern long-term exposures

- 100-150 ppb
  Minimum for anti-microbial efficacy

- ~5,000 ppb
  Health concern short-term exposure, risk depends on individual

- 1,000 ppb
  Minimum for anti-microbial efficacy

- 200 ppb
  Health concern long-term exposures

- ≤100 ppb
  Output of ACTEX/US iodine removal hardware
Health Risks Associated with Elevated Biocide Concentrations

Ingesting high concentrations of silver can cause Argyria, an irreversible blue-gray discoloration of the skin.

Excessive iodine in drinking water causes aesthetic issues (taste, odor) that may lead to decreased consumption and can cause hypothyroidism.

Silver Analysis

Silver(I) Determination: **1.0 mL** sample volume

Ag⁺ reacts with 5-(p-dimethylaminobenzylidene)rhodanine*

![DMABR](image)

**Dynamic range:** 0.100 to 1.0 mg/L
Iodine Analysis

**Iodine Determination: 10.0 mL sample volume**

I$_2$ reacts with polyvinylpyrrolidone

![Graph showing dynamic range: 0.200 to 5.0 mg/L](attachment:iodine_analysis.png)

LOD: 0.05 ppm

Reagent Stability

**Graph showing Percent Recovery vs. Sampling Date**

- **Iodine Analysis Cartridges**
- **Silver Analysis Cartridges**
### In-flight Results - SVO-ZV Samples

<table>
<thead>
<tr>
<th>Analysis Date</th>
<th>In-flight CSPE (mg/L)</th>
<th>N</th>
<th>Ground Result (mg/L)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>9/23/2009</td>
<td>&lt;0.100</td>
<td>1</td>
<td>0.023</td>
<td>Ground result from SVO-ZV sample collected 9/22/09 and returned on STS-129/ULF3.</td>
</tr>
<tr>
<td>10/20/2009</td>
<td>&lt;0.100</td>
<td>1</td>
<td>0.081</td>
<td>Ground result from SVO-ZV sample collected 10/20/09 and returned on STS-129/ULF3.</td>
</tr>
<tr>
<td>11/10/2009</td>
<td>&lt;0.100</td>
<td>3</td>
<td>0.018</td>
<td>Ground result from SVO-ZV sample collected 11/10/09 and returned on STS-129/ULF3.</td>
</tr>
<tr>
<td>1/7/2010</td>
<td>0.129/&lt;0.100</td>
<td>3</td>
<td>0.056</td>
<td>Ground result from SVO-ZV sample collected 1/6/10 and returned on STS-130/20A.</td>
</tr>
<tr>
<td>3/4/2010</td>
<td>0.197/&lt;0.100</td>
<td>3</td>
<td>0.069</td>
<td>Ground result from SVO-ZV sample collected 3/3/10 and returned on STS-130/20A.</td>
</tr>
<tr>
<td>4/1/2010</td>
<td>0.100</td>
<td>1</td>
<td>0.090</td>
<td>Ground result from SVO-ZV sample collected 3/31/10 and returned on STS-130/20A.</td>
</tr>
<tr>
<td>4/27/2010</td>
<td>0.241</td>
<td>3</td>
<td>0.086</td>
<td>Ground result from SVO-ZV sample collected 4/26/10 and returned on STS-132/ULF4.</td>
</tr>
<tr>
<td>5/20/2010</td>
<td>&lt;0.100</td>
<td>1</td>
<td>0.125</td>
<td>Ground result from SVO-ZV sample collected 5/18/10 and returned on STS-132/ULF4.</td>
</tr>
<tr>
<td>6/16/2010</td>
<td>&lt;0.100</td>
<td>3</td>
<td>N/A</td>
<td>No samples returned yet.</td>
</tr>
<tr>
<td>7/15/2010</td>
<td>&lt;0.100</td>
<td>3</td>
<td>N/A</td>
<td>No samples returned yet.</td>
</tr>
<tr>
<td>8/30/2010</td>
<td>&lt;0.100</td>
<td>3</td>
<td>N/A</td>
<td>No samples returned yet.</td>
</tr>
<tr>
<td>9/22/2010</td>
<td>&lt;0.100</td>
<td>3</td>
<td>N/A</td>
<td>No samples returned yet.</td>
</tr>
<tr>
<td>12/2/2010</td>
<td>&lt;0.100</td>
<td>3</td>
<td>N/A</td>
<td>No samples returned yet.</td>
</tr>
</tbody>
</table>

### In-flight Results - PWD Dispensing Needle Samples

<table>
<thead>
<tr>
<th>Analysis Date</th>
<th>In-flight CSPE (mg/L)</th>
<th>N</th>
<th>Ground Result (mg/L)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>9/23/2009</td>
<td>&lt; 0.200</td>
<td>1</td>
<td>&lt;0.050</td>
<td>Ground result from PWD sample collected 9/22/09 and returned on STS-129/ULF4.</td>
</tr>
<tr>
<td>10/20/2009</td>
<td>&lt;0.200</td>
<td>3</td>
<td>&lt;0.050</td>
<td>Ground result from PWD sample collected 10/20/09 and returned on STS-129/ULF4.</td>
</tr>
<tr>
<td>11/10/2009</td>
<td>&lt;0.200</td>
<td>2</td>
<td>&lt;0.050</td>
<td>Ground result from PWD sample collected 11/10/09 and returned on STS-129/ULF4.</td>
</tr>
<tr>
<td>4/27/2010</td>
<td>0.973</td>
<td>3</td>
<td>&lt;0.050</td>
<td>Ground result from PWD sample collected 4/26/10 and returned on STS-132/ULF4.</td>
</tr>
<tr>
<td>5/20/2010</td>
<td>&lt;0.200</td>
<td>1</td>
<td>&lt;0.050</td>
<td>Ground result from PWD sample collected 5/18/10 and returned on STS-132/ULF4.</td>
</tr>
<tr>
<td>7/15/2010</td>
<td>&lt;0.200</td>
<td>2</td>
<td>&lt;0.50</td>
<td>Ground result from PWD sample collected 7/14/2010 and returned on Soyuz 22.</td>
</tr>
<tr>
<td>9/22/2010</td>
<td>&lt;0.200</td>
<td>3</td>
<td>&lt;0.050</td>
<td>Ground result from PWD sample collected on 9/15/2010 and returned on Soyuz 22.</td>
</tr>
<tr>
<td>12/2/2010</td>
<td>&lt;0.200</td>
<td>3</td>
<td>N/A</td>
<td>No samples returned yet.</td>
</tr>
</tbody>
</table>

- Samples collected downstream of iodine removal hardware (ACTEX)
- Certified to reduce iodine concentrations to <0.100 mg/L
- MORD requirement for iodine at points of consumption is <0.200 mg/L
In-flight Results - PWD Aux. Port Samples

<table>
<thead>
<tr>
<th>Analysis Date</th>
<th>In-flight CSPE (mg/L)</th>
<th>N</th>
<th>Ground Result (mg/L)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/7/2010</td>
<td>0.769</td>
<td>3</td>
<td>N/A</td>
<td>No archive sample returned</td>
</tr>
<tr>
<td>3/4/2010</td>
<td>0.590</td>
<td>3</td>
<td>1.11</td>
<td>Ground result from PWD sample collected 2/26/10 and returned on STS-131/19A</td>
</tr>
<tr>
<td>4/1/2010</td>
<td>0.380</td>
<td>1</td>
<td>&lt;0.050</td>
<td>Ground result from CWQMK archive sample collected 3/31/10</td>
</tr>
<tr>
<td>6/16/2010</td>
<td>0.972</td>
<td>3</td>
<td>N/A</td>
<td>No samples returned yet</td>
</tr>
<tr>
<td>8/30/2010</td>
<td>1.65</td>
<td>3</td>
<td>2.05</td>
<td>Ground results from WPA RIP sample collected 7.29/10 and returned on Soyuz 22</td>
</tr>
</tbody>
</table>

- Samples collected upstream of iodine removal hardware (ACTEX)
- MORD requirement for residual biocide is 1-4 mg/L I₂

Summary

Results demonstrate the capability to monitor biocide concentrations in-flight with CSPE
- Samples from PWD ambient leg contain no detectable iodine
- Iodine detected in samples from PWD Aux. port
- Results from SVO-ZV samples are consistent with archival samples
Forward Work

- Transition to operational hardware
  - Consumables delivered on 43 Progress (June 2011)
  - Add capability to monitor total iodine
    - Sum of iodine, iodide, and triiodide

Expand the capabilities of the CWQMK
- Urinary calcium
- o-phthalaldehyde, ammonia, pH in coolant fluids
- Biological monitoring
- Air quality monitoring

Explore terrestrial applications of CSPE technology
Acknowledgements

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