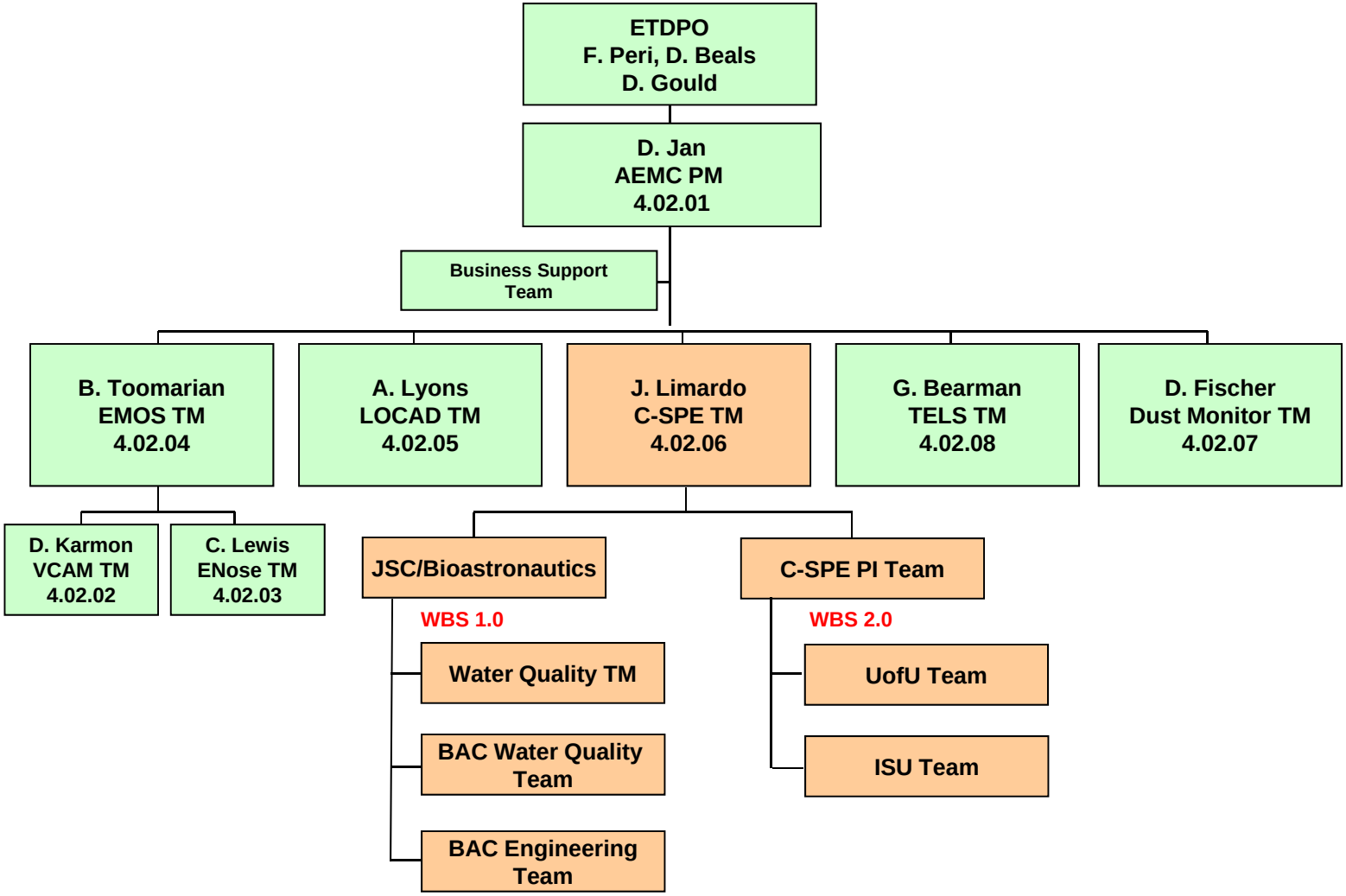
II. AEMC Project Technology Tasks

Colorimetric Solid Phase Extraction (C-SPE)

C-9 Flight: October 24, 2007

Team & Partnerships

AEMC Project WBS Structure



Work Agreements

AEMC

- NASA's Exploration Systems Mission Directorate (ESMD) and Space Operations Mission Directorate (SOMD) have signed a Memorandum of Agreement (MOA) at the NASA Associate Administrator level;
  - This cooperative agreement between ESMD, SOMD would facilitate activities associated with development, launch and cooperative utilization of the C-SPE aboard the International Space Station (ISS).

C-SPE

- Station Development Test Objectives (SDTO) Plan
  - This plan provides the ability to interface with the SDTO Working Group in order to facilitate manifestation on an ISS increment to test and evaluate the CSPE hardware, software, and procedures on-orbit .
  - ISS as test bed for testing, evaluation and demonstration

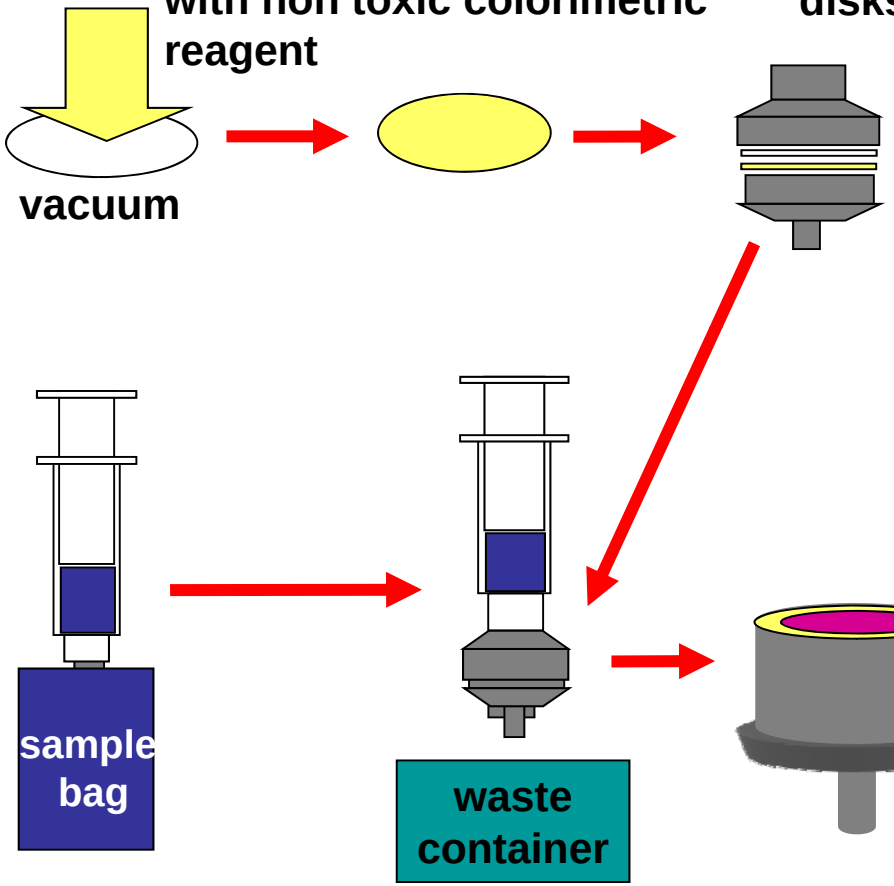
AEMC: Colorimetric Solid Phase Extraction

History

- **AEMC competitive grant support through two three-year cycles to move C-SPE from TRL 1 to 4.**
- **Joint ISS/AEMC Trade Study for ISS in FY03-FY04.**
- **Expert external panel evaluated RFI proposals/presentations Oct 2004.**
- **C-SPE top technique for biocides and inorganics in water.**
- **Development continued with support from AEMC Rapid Technology Development and Environmental Monitors on Station (EMOS) funding**
- **Method development has focused mainly on biocide monitoring.**

Illustration of C-SPE Method

1. Impregnate membranes with non toxic colorimetric reagent
2. Cut membrane into 13-mm disks and load in filter holder

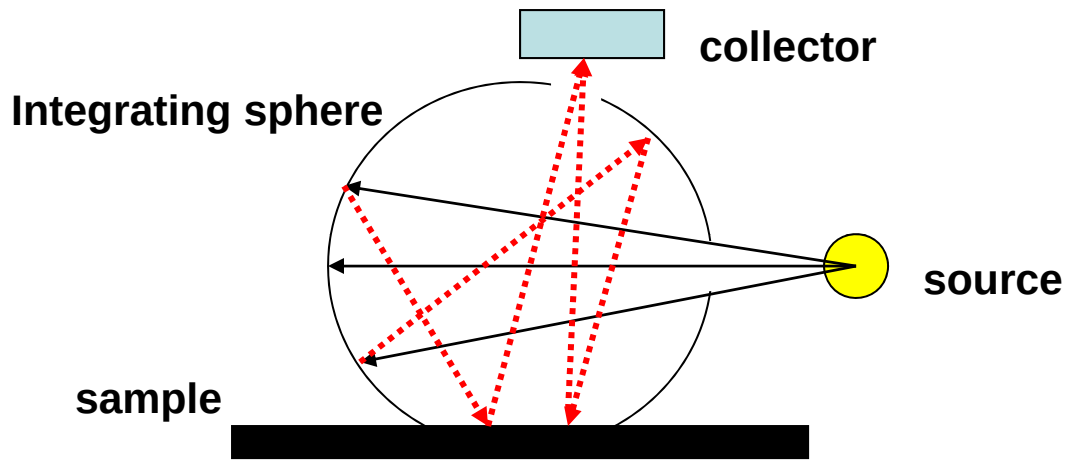


3. Withdraw sample using syringe
4. Pass sample through disk
5. Acquire spectrum with portable reflectance spectrometer

Total analysis (steps 3-5) under 2 min

Quantification of Analytes by C-SPE

Diffuse Reflection - reflection from a surface with a roughness comparable to the wavelength of incident radiation



Reflectance (*R*) related to concentration by the Kubelka Munk function

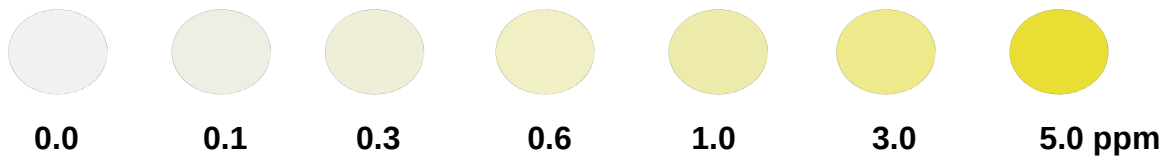
$$f(R) = (1-R)^2/(2R) = \varepsilon c/s$$

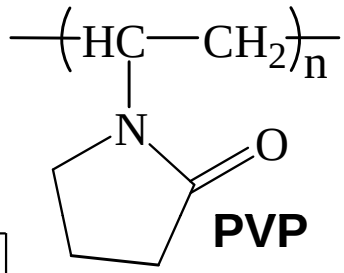
ε is molar absorptivity, *c* is concentration, and *s* is scattering coefficient

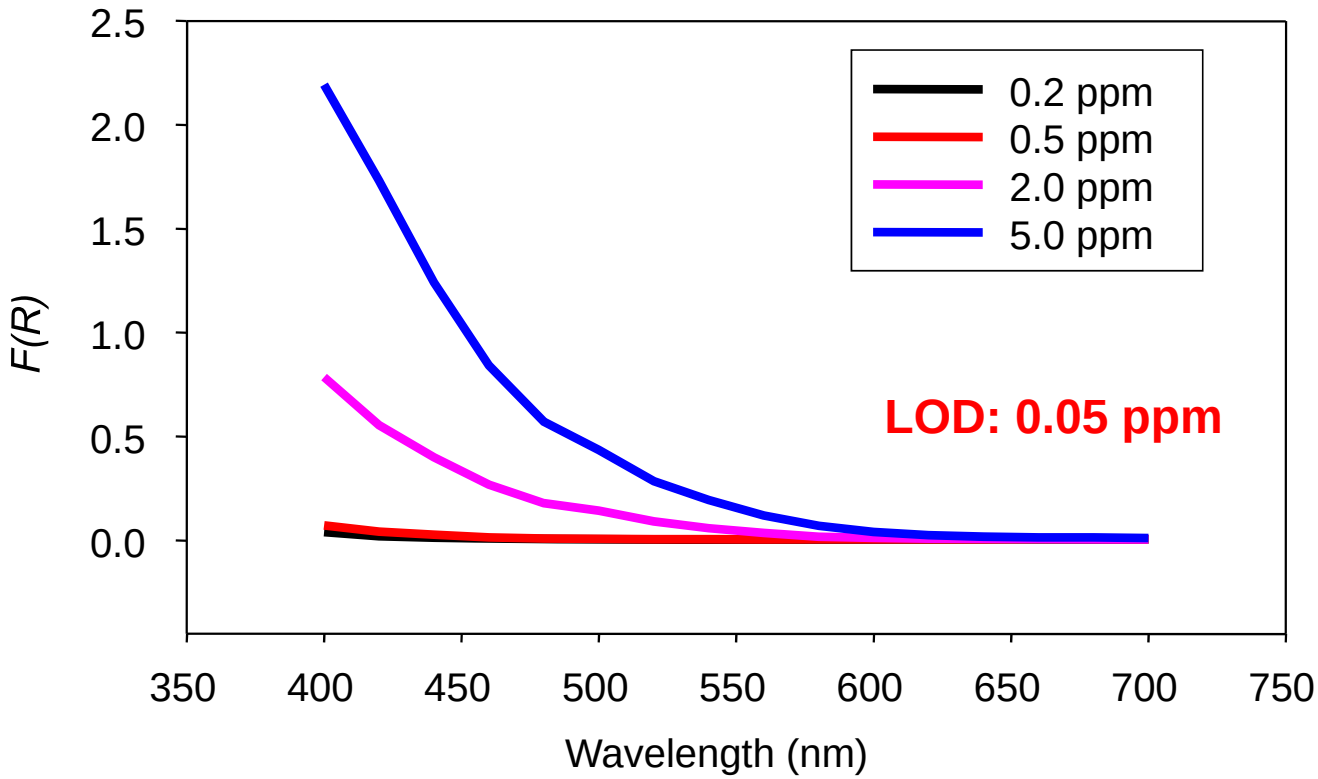
f(*R*) is directly proportional to analyte concentration

Iodine Determination: 10.0 mL sample volume

I_2 reacts with polyvinylpyrrolidone



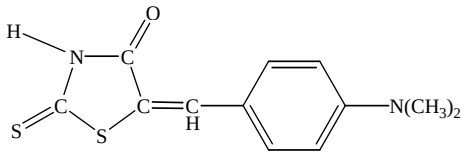




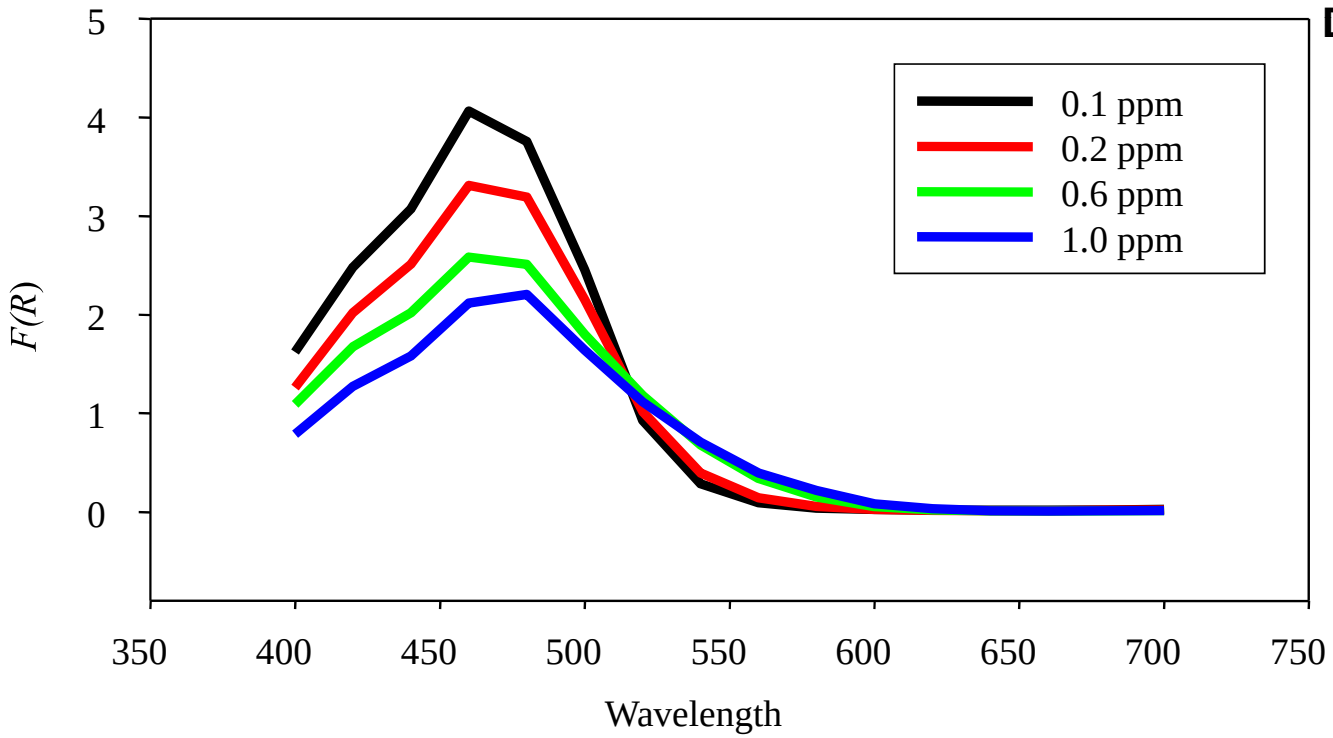
Silver(I) Determination: 1.0 mL sample volume

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

0.0 0.1 0.3 0.6 1.0 ppm



DMABR



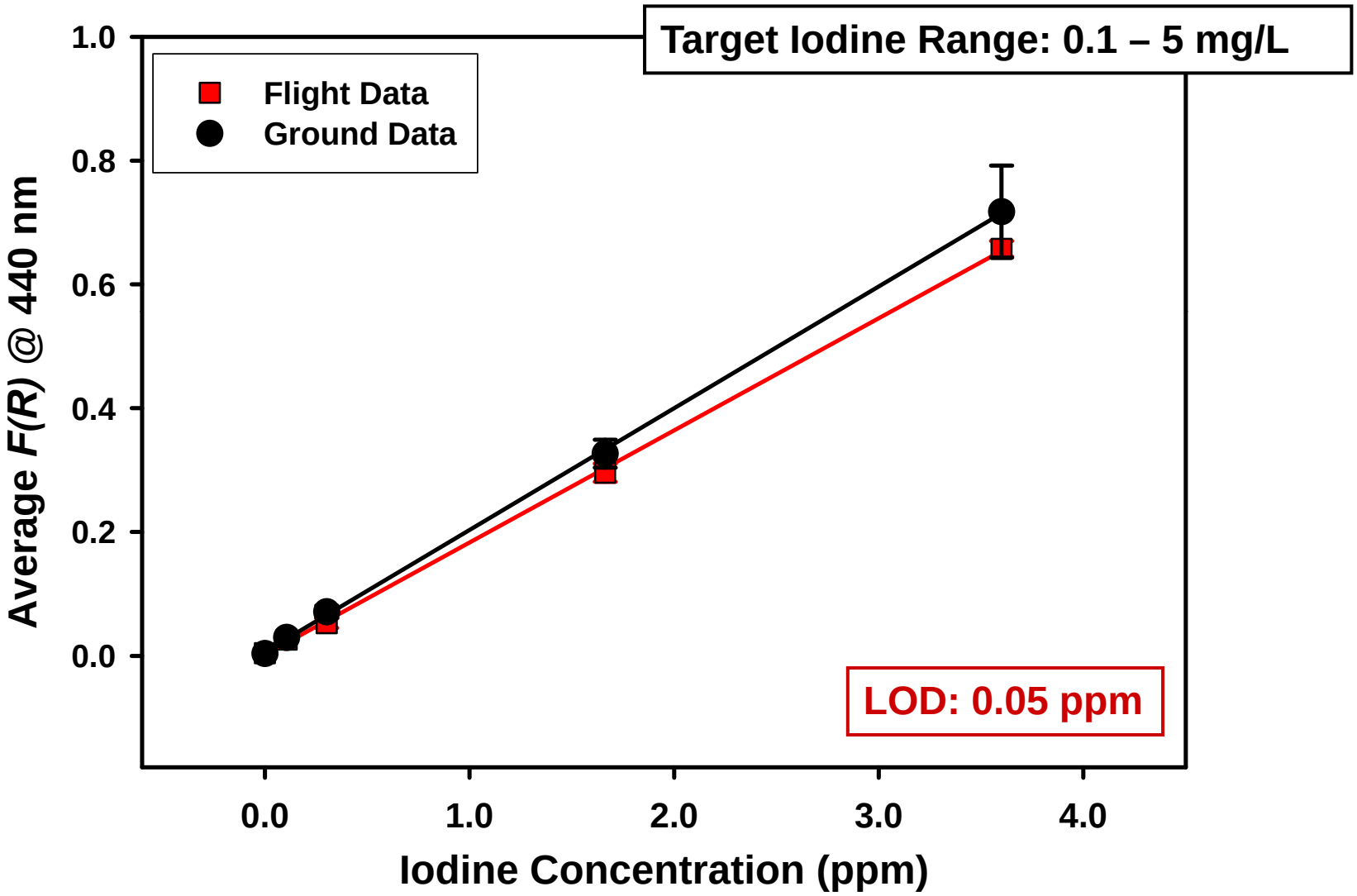
dynamic range: 5 to 1000 ppb

LOD: ~5 ppb

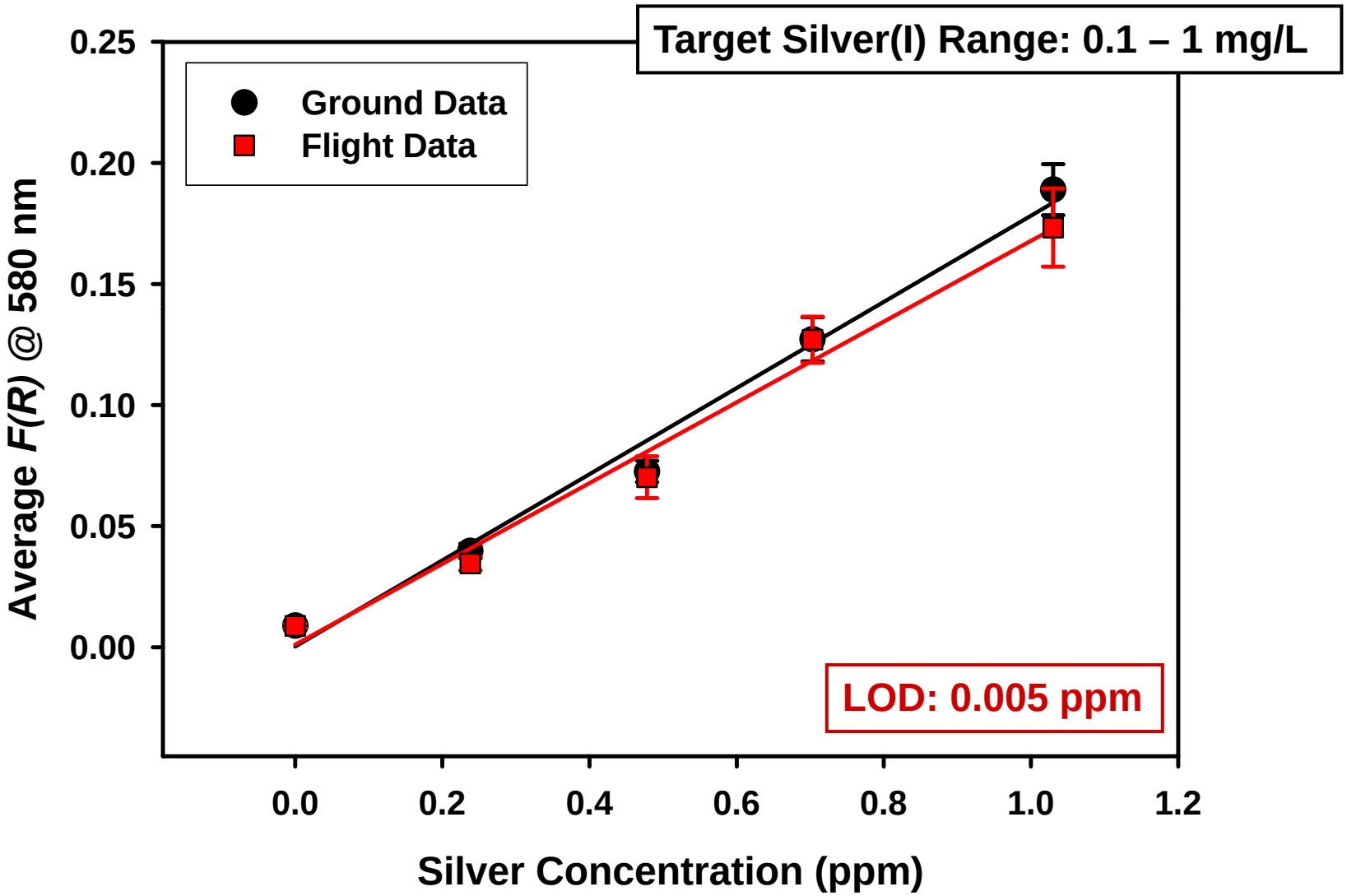
Challenge: Bubbles in microgravity complicate metering of an accurate sample volume through C-SPE disk

Manual manipulation of water sample bags is simple and effective for air/water separation.

C-9 Iodine Analysis by C-SPE

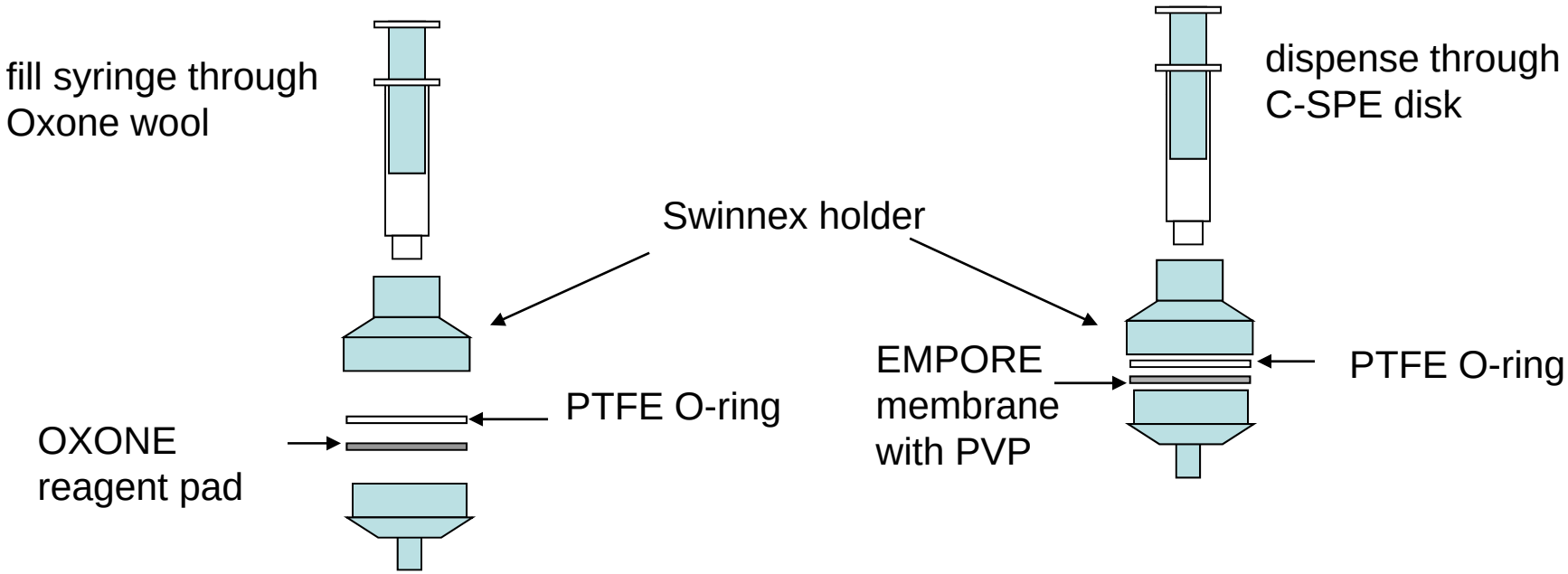


C-9 Silver Analysis by C-SPE



Total Iodine Determination:

Iodide (I^-) and triiodide (I_3^-) are readily oxidized to iodine (I_2) using Oxone ($2KHSO_5 \bullet KHSO_4 \bullet K_2SO_4$)



By utilizing solid-phase reagents and in-line reagent introduction the iodine C-SPE method is capable of measuring total iodine in water samples

$$[I]_{tot}=[I_2]+[I^-]+[I_3^-]$$

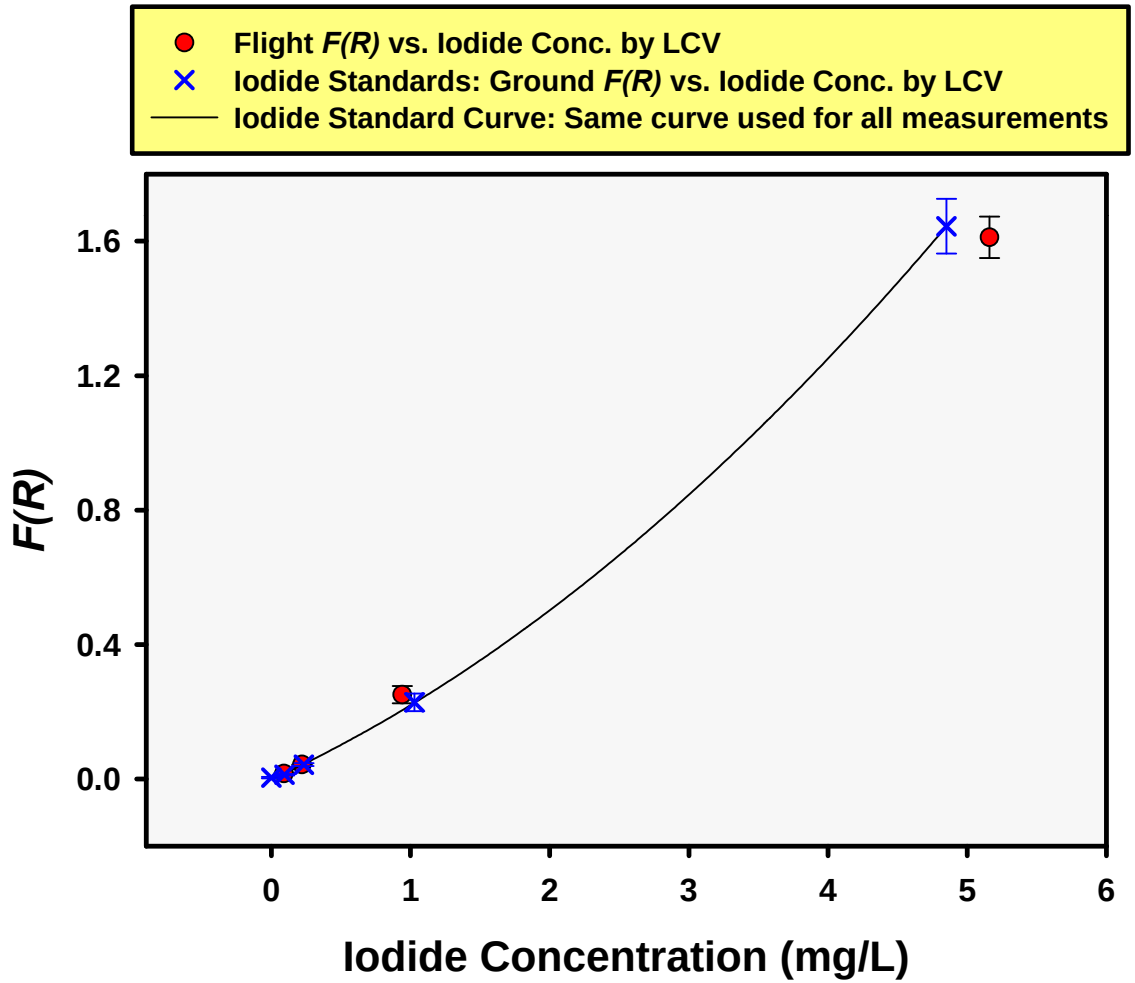
October 2007 C-9 Results

Objective

- Test ability to measure total I in microgravity

Procedures

- Fill syringes through Oxone cartridges to convert iodide to iodine
- Remove air from syringe to accurately meter 10.0-mL of sample
- Pass sample through indicator cartridge and measure *F(R)*
- Use standard curve measured on the ground to determine flight concentrations
- All concentrations verified by Wyle Laboratories using leuco crystal violet (LCV) method
- Concentrations spot-checked by ICP-MS



AEMC: Colorimetric Solid Phase Extraction

Technology Maturity

- **Methods for silver, iodine, and total iodine have been successfully demonstrated during C-9 microgravity flights.**
- **Fluid handling and readout are identical for all analytes; additional methods only require development of new reagent/membrane cartridges.**
- **Methods for total silver, nickel, cadmium, pH, and formaldehyde developed, but not tested on C-9 microgravity flights.**
- **COTS reflectance spectrophotometer requires laptop for data analysis and has flash memory that may be susceptible to radiation damage.**
- **Subcontract to modify COTS reflectance spectrophotometer to simplify data handling and mitigate radiation concerns recently established.**

AEMC: Colorimetric-Solid Phase Extraction

Benchmarks & Proposed Experiment

- **The performance of C-SPE methods are being measured against standard methods of water analysis available from APHA/AWWA/WPCF, U.S. EPA, or ASTM**
  - **Iodine: Leuco Crystal Violet colorimetric method**
  - **Total Iodine: Leuco Crystal Violet or Inductively Coupled Plasma/Mass Spectroscopy (ICP/MS)**
  - **Ionic Silver: Filtration followed by ICP/MS**

- **During proposed flight experiment, performance of C-SPE methods will be evaluated by measuring check standards on-orbit, returning samples analyzed on-orbit for post-flight analysis, and conducting parallel ground tests**

Schedule

- **The C-SPE SDTO technology demonstration flight experiment hardware (TRL-6) is scheduled to be delivered by August 2009**
  - The earliest Shuttle flight available is scheduled for November 2009 (STS-130, 19A)
  - The original scheduled delivery date was set to November 2008 prior to the Water Processor Assembly (WPA) becoming operational and providing iodinated water to the six-member crew (delayed due to lack of available funding)
- **The requirement for in-flight water quality monitoring for iodine onboard ISS is currently unmet***
  - On-orbit water quality is critical to the health of the crewmembers
  - In-flight monitoring is necessary to get timely results and to evaluate critical parameters that may not be stable in archival samples

***International Space Station Medical Operations Requirements Document (ISS MORD)**

AEMC: Colorimetric-Solid Phase Extraction
Schedule - Milestones

Key Milestones/Activities	Estimated Completion Date
Complete System Requirements Review (SRR)	February 2008
Complete Prototype Spectrophotometers & Kit	April 2008
Complete Extended Shelf-Life Studies	June 2008
Complete Prototype Ground & C-9 Testing	September 2008
Complete Phase I/II Safety Review	October 2008
Complete System Design Review (SDR)	November 2008
Complete Qual Unit Fabrication (1)	January 2009
Complete Qualification Testing	March 2009
Complete Flight Unit Fabrication (4)	April 2009
Complete Acceptance Testing	May 2009
Complete Phase III Safety Review	June 2009
Complete System Acceptance Review & Delivery	August 2009

AEMC: Colorimetric-Solid Phase Extraction

Resource Requirements

Funding Requirements ($K)

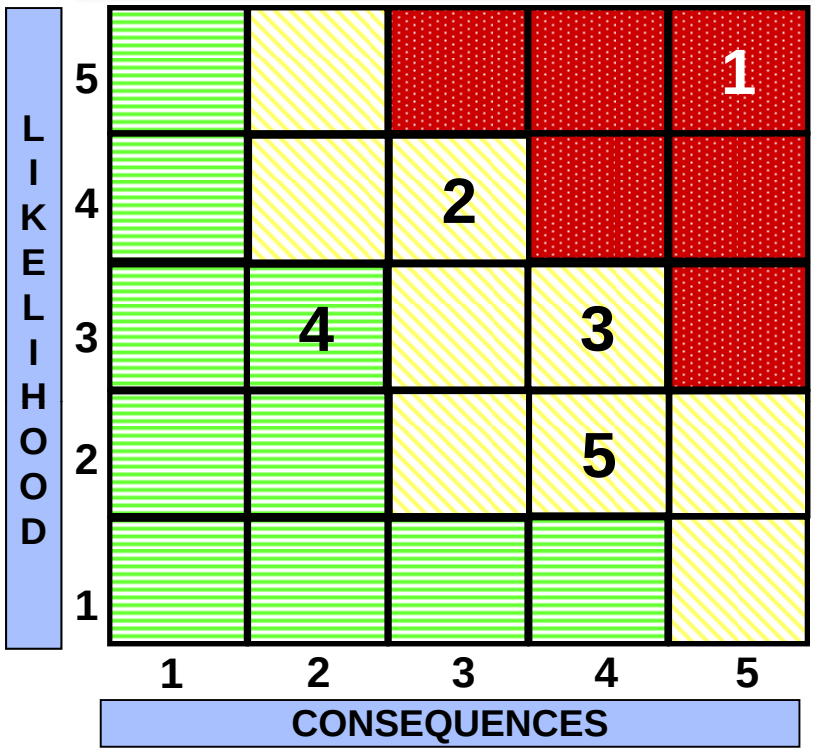
WBS	FY08	FY09	FY10	FY11
1.0 JSC Team	766	1039	TBD	TBD
2.0 PI Team	512	456	TBD	TBD
Total	1239	1461	1500	300

Workforce Requirements (WYE)

WBS	FY08	FY09	FY10	FY11
1.0 JSC Team	2.95	4.40	TBD	TBD
2.0 PI Team	3.40	3.40	TBD	TBD

AEMC: Colorimetric-Solid Phase Extraction

Technology Task Risk Mitigation



Criticality

- High
- Med
- Low

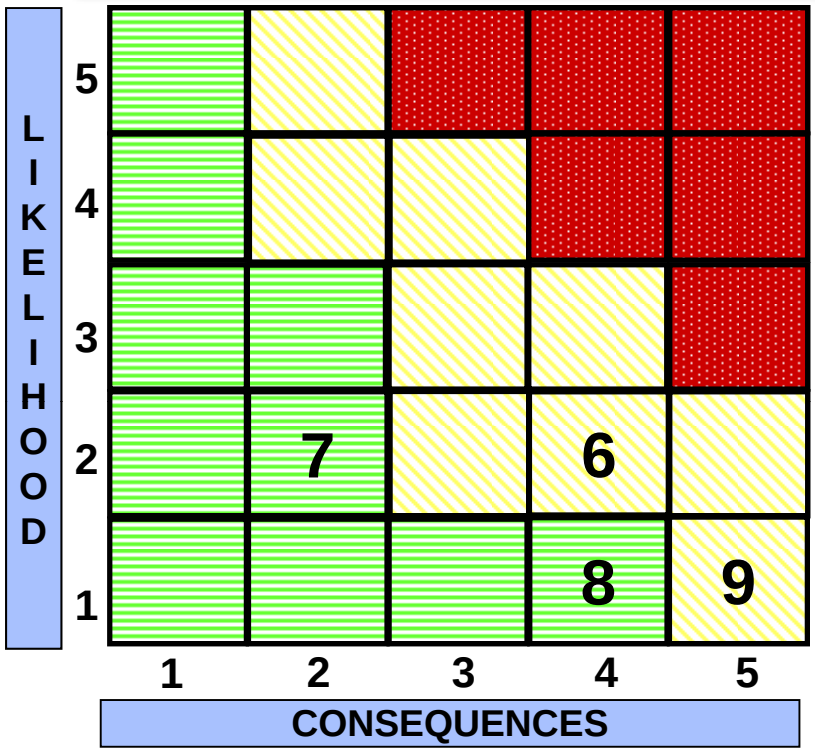
L x C Trend

- ⬇ Decreasing (Improving)
- ⬆ Increasing (Worsening)
- ➡ Unchanged
- * New since last month

Rank ID Trend	Description	Risk Management Approach
1 CSPE1 ➡	Inconsistent funding from AEMC Program throughout the course of the project at both WBS. PI Team has not received FY07 funding.	Mitigate: Work with program office and further support by stakeholders
2 CSPE2 ➡	Flash memory and COTS spectrophotometer may be susceptible to radiation damage. Is not cost effective for SDTO to replace flash memory.	Mitigate: SDTO kit contains (2) spectrophotometers. Assess approaches to store the kit for protection from ionizing event. Software will also be modified to store the code and data in multiple locations.
3 CSPE3 ➡	Long-term shelf-life of reagent cartridges has not been tested.	Mitigate: Vacuum pack reagent cartridges. Conduct shelf-life studies. Modify chemistries as needed.
4 CSPE4 ➡	Water passed through cartridges has not been reviewed by safety or toxicology.	Mitigate: Obtain early toxicology review of reagent cartridges and waste water.
5 CSPE5 ➡	Safety, materials, offgassing, and electrical reviews of COTS spectrophotometer have not been completed.	Mitigate: Early delivery of prototype unit for C-9 testing. Pre-prototype review of hardware and software.

AEMC: Colorimetric-Solid Phase Extraction

Technology Task Risk Mitigation, cont.



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Rank ID Trend	Description	Risk Management Approach
6 CSPE6 ⬇	Unknown contaminants in water samples may interfere with the biocide detection chemistries resulting either erroneously low or high results.	Mitigate: Continue interference testing with ISS and Shuttle archival samples and spacecraft simulants.
7 CSPE7 ⬇	Method requires the water volume passed through the reagent cartridges to be known. Air bubbles in water can yield uncertainly in water volume.	Mitigate: Continue advancing sample manipulation procedures via C-9 microgravity flights.
8 CSPE8 ⬇	Delays in project funding increase risk that spectrophotometer vendor may decline to continue participation in project.	Mitigate: Continue to develop relationship with the spectrophotometer vendor.
9 CSPE9 ➡	Delay in delivery of ISS Water Processor will delay availability of iodinated water to be tested.	Watch