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A Spectrophotometric Analyzer for Aqueous Samples in Microgravity

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ABSTRACT

The development of a spectrophotometric analyzer for use on water samples in microgravity environments is discussed. The instrument is constructed around a commercial spectrophotometer, the Hewlett-Packard HP8453, with a separate turbidimetric analyzer, here a modified Hach 2100P ratio turbidimeter. Flow-through sample cells were constructed for each instrument to support microgravity use and sample deaeration. Spectrophotometric analyses on aqueous samples on orbit are sensitive to the presence of undissolved gases in the samples. In a micro-g environment, free gas in samples can and does remain suspended, clouding the mixture and interfering with spectral optical density measurements. This paper discusses the design of a spectrophotometric analyzer, with particular emphasis on the merits of two approaches to eliminating free gas interferences in on-orbit water analyses: hyperbaric gas redissolution and deaeration across a hydrophobic membrane. Both techniques mitigate gas loads effectively – membrane removal uses a minimum of sample and time, hyperbaric redissolution takes more time, but provides capability to handle bubbles should gas get introduced into or otherwise evolve in the analytical system.

INTRODUCTION

On-orbit analyses of International Space Station water supplies are important in assessing recycled water potability. Against this mission, analytical instruments compatible with microgravity applications have been under development. While the station provides a shirtsleeve environment that approximates a terrestrial laboratory, there remain differences that provide challenges for the on-orbit use of lab instrumentation. The principal challenges for solution spectroscopic measurements are fluid handling - simply keeping a sample in the spectrophotometer - and the presence of free gas distributed in liquid samples, particularly microfine bubbles that make samples appear turbid. The presence of even small quantities of free gas is problematical, as spectrophotometric measurements are sensitive to sub-part-per-thousand changes in sample optical throughput.

The determination of biocide load in potable water supplies provides an example of the spectroscopic requirements for analysis and the need for gas-free samples. In neutral, aqueous samples, iodine disproportionates to form hypodiodous acid, hypoiodate, iodate and iodide ion, and the free iodide ion in turn equilibrates with the remaining iodine to form triiodide ion (equations 1-4)[1,2,3]. Iodine and triodide ion concentrations can be determined with spectroscopic measurements on solutions, and the concentrations of the remaining species estimated using the equilibria defined below. However, accurate deconvolution of iodine speciation and determination of total iodine load requires the measurement of solution absorbance to 0.0001 absorbance unit on a 10cm path cell. In an instrument with a 1 cm diameter optical beam, the presence of a 100 micron diameter bubble can affect the accuracy of the measurement.

$I_2 + H_2O \rightarrow HOI + I^- + H^+$	$K_{eq} = 3 \times 10^{-13}$	(1)
$HOI \rightarrow H^+ + OI^-$	$K_a = 4.5 \times 10^{-13}$	(2)
$I_2 + I^- \rightarrow I_3^-$	$K_{eq} = 1.4 \times 10^{-3}$	(3)
$3OI^- \rightarrow IO_3^- + 2I^-$	$K_{eq} = 1 \times 10^{20}$	(4)

Free gas in water supplies on orbit is an ubiquitous problem. From hydrogen-loaded fuel cell water to oxygen-loaded water in the Apollo Command Module water tank, potable water supplies have presented challenges with bubbles in water, particularly at the point of use and when that point is heated.[4] By itself, the presence of a little free gas in a water supply is not critical, and steps have been made to minimize entrained gas[5]. However, samples submitted for spectroscopic analyses cannot accommodate even small quantities of gas, and mitigation of free gas originating in samples or introduced as part of the sampling process is necessary for the precise determination of dissolved species, such as iodine or triiodide.

Sample handling provides a separate challenge in microgravity, requiring careful control of potentially hazardous solutions from the point of sampling, through analysis, and ultimately into a waste receptacle. Open sample cuvettes are not an option. Flow-through sample cells, available for both spectrophotometric and turbidimetric analyzers, can be used with syringe injection to provide a sealed sample path. However, the lack of a pressure gradient forcing bubble removal and the restricted access to the sample cell volumes makes the use of flow cells problematical on orbit. Sample deaeration immediately prior to injection helps prevent the introduction or formation of bubbles in the sample cuvette, but once formed, the bubbles must be dealt with *in situ* since they become difficult to flush out. In enclosed systems, the maintenance of a backpressure or preload can be used to prevent bubble formation or redissolve bubbles that are introduced. However, dissolution kinetics can be painfully slow for large bubbles in the absence of mixing[6], and remarkably fast for small (critical-diameter or smaller) bubbles[7], so a mechanism for agitating or cavitating samples is necessary for relatively quick experiments.

Deaeration can be coupled effectively with the sampling process, minimizing design requirements for dealing with free gas in the analytical sample cells. For this purpose, either active (ie, centripetal acceleration) or 'passive' (membrane-based phase separation) may be used. Variants of both systems have been used extensively in space[8], with pumping systems dominating mixed fluid/gas streams (for example, the separator technology used in the MIR urine processor to handle the urine/air input stream[9], and a separator design for handling shower water/air mixtures[10]) and membrane technology dominating phase separations where a fluid contains or generates a low flux of another phase (eg. electrochemical cell gas generation, hydrogen gas removal from fuel cell water[11], and heat exhanger condensate removal from recirculating air[12]). A simple hydrophobic membrane-based deaeration unit is sufficient to remove entrained gases at the one to five percent loads expected in analytical samples[13].

Two additional challenges posed by flow-through sample cells are sample volume and chemical reactivity. Sample volume is directly related to analytical accuracy; larger samples are required to rinse-down a flow-through spectrophotometer cell than are required in replicate fill-and-decant rinses of an open cuvette. Sample line dead volumes and sample cell volumes must be kept to a minimum (order 5 milliliters) for accurate analyses on samples of 50 milliliters or less. However, minimizing sample loop volumes increases the surface area to volume ratio, increasing the reactivity of iodine[14,15], an important analyte for on-orbit analyses. Careful selection of sample loop materials is necessary to optimize sample volumes while preserving the integrity of samples.

This paper presents the development of a spectrophotometric analyzer and sample system for on-orbit use. Sample loop design for minimizing sample volume while controlling iodine reactivity and mitigating free gas interference is discussed, with data illustrating the degree to which these problems have been handled. A simple membrane-based deaerator/sampler, optimized for efficient extraction of free and dissolved gas in small samples, is described.

SYSTEM ARCHITECTURE

The prototype analyzer was built around commercial UV-Visible spectrophotometer and turbidimeter instrumentation. The UV-visible spectrometer was an unmodified Hewlett-Packard HP-8453a, single-beam instrument, retrofitted with a custom pressure cell. Specifications for this instrument include a spectroscopic range from 190 to 1100 nm, with 1 nm resolution and a 0 to 3.3 absorbance dynamic range. Turbidity was measured using a Hach 2100P portable turbidimeter, modified to include an RS232-compatible interface. A low-volume sample cell was fabricated to resemble the standard flow-through cell. Figure 1 illustrates the mounting scheme for the two instruments and supporting hardware in the prototype analyzer.

Instrument control, data collection, and data analysis are performed via laptop computer using custom software commanding the instruments over an IEEE-488 compatible interface (National Instruments PCMCIA-GPIB). An IOTech Serial/488A interface module is included to allow remote control over the turbidimeter module using the one IEEE-488 interface bus.

A membrane-based deaerator was developed for use in conjunction with a water sampler (i.e., a syringe) to allow for the degassing of samples prior to introduction in the analyzer. The deaerator employs a large number of hollow-fiber membranes (408 Hoechst-Celanese microporous polypropylene lumens, each 5 inches in length, 400 microns internal diameter, and with a 30 micron wall) bundled into a polycarbonate shell that can be evacuated prior to use. The lumens were potted into the assembly using epoxy resin and hardener. As liquid

is pulled through the membrane bundle, bubbles that contact the membranes are drawn across the polypropylene pores into the evacuated shell. The deaerator and sample syringe is shown in Figure 1 and schematically illustrated in Figure 2. The valves illustrated on either end of the unit allow the evacuation of the polycarbonate shell with the syringe immediately prior to sampling.

borosilicate (Pyrex) cylinder, and top and bottom caps with holes for fluid entry and exit. The caps are coated to a flat black finish. The assembly is held together by four posts which mount into the top and bottom caps.

Both cells are modified to accept piezoelectric transducers, allowing ultrasonic excitation of samples prior to spectrophotometric data collection. The ultrasonic input was required to agitate the solutions,

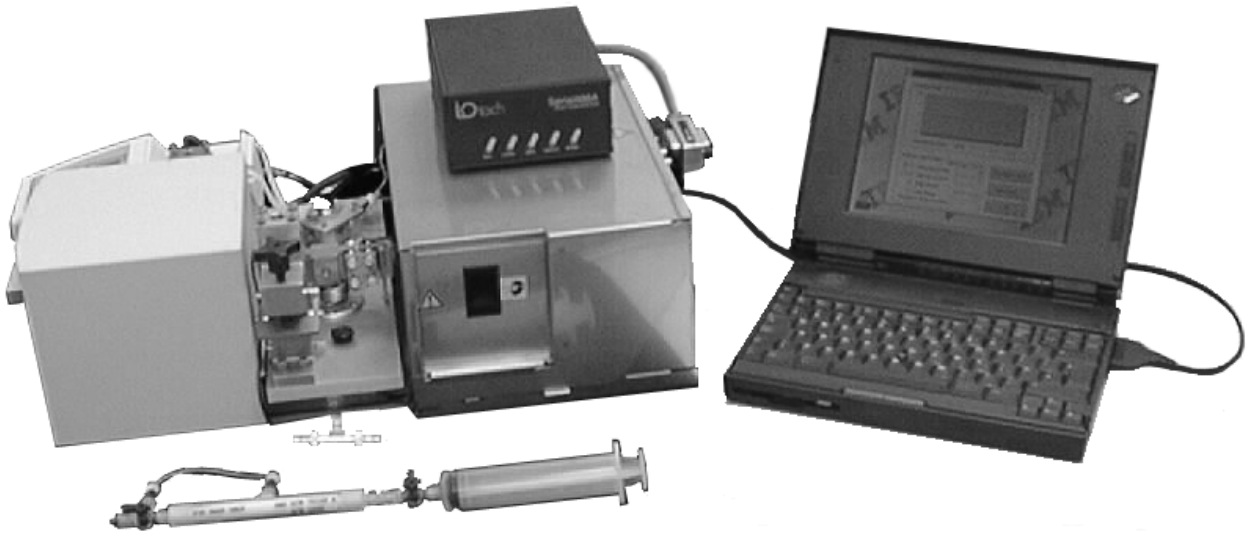

Figure 1, Prototype Spectrophotometric Analyzer, Consisting of a HP8453a Ultraviolet-Visible-NearIR Spectrophotometer and a Hach 2100P Ratio Turbidimeter. A sample syringe and membrane-based deaerator are shown in front of the instrument, next to a control processor (laptop computer) that operates the instrument via an IEEE 488 interface.

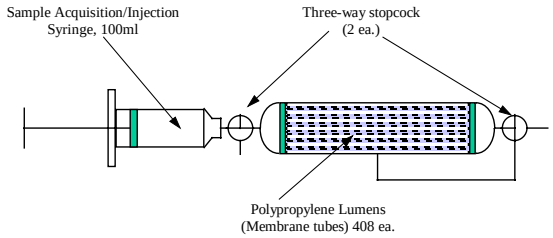

Figure 2, Sample Withdrawal/Injection Device (SWID), a Two-Pass Flow-Through Membrane Deaerator.

The spectrophotometer cell is constructed from 316 stainless steel, with a 0.55 cm diameter by 10 cm long sample volume. Quartz windows are mounted at the distal ends of the sample cell, and two additional windows are mounted at the sides of the cell to provide a 1 cm path-length option for viewing darker samples. Viton® o-rings captured between the quartz windows and flanges on the cell body seal unit against operating sample pressures up to 150 psi. The cell pivots about its center to one of two indented sample positions to allow measurements from both long and short cell paths.

The turbidimeter sample cell is a custom flow-through design that is plumbed in series with the spectro-photometer cell in the pressurized sample loop. The flow-through cell consists of the a thick-walled

facilitating dissolution of bubbles in the cells.

A schematic of the instrument is presented in Figure 3. Samples are introduced into the cell through 1/16th inch tubing via a luer-lock interface to a sample syringe or membrane deaerating unit. In series with the spectrophotometer cell is a turbidimeter cell, which in turn is followed by a metal bellows preload assembly. As samples are introduced, the prior contents of the loop and excess sample fill a waste bag at the sample output port.

Applying a static pressure to the fluid with a spring-loaded mechanism acting on the metal bellows reservoir effected deaeration of fluids in the sample loop. Following the injection of a 75 milliliter sample (the bulk of which rinses through the sample cells, leaving those cells with representative samples for analysis), a 4-port HPLC valve is actuated to isolate the sample loop from the inlet and exhaust ports. A preload spring is then applied to a metal bellows reservoir, compressing the reservoir to a fixed pressure between 75 and 150 psig (user selectable). Ultrasonic agitation is applied to the samples in the analytical cells as the pressure is applied, moving any free gas in the cell volumes to facilitate dissolution under the static load.

The analyzer and sampler were separately evaluated for

analytical performance and chemical stability on standard iodine/iodide ion solutions, comparing iodine and triiodide concentrations as computed by UV spectrophotometric measurements[16] and Leuco Crystal Violet (LCV) colorimetry[17] on input and exhaust solutions. In situ monitoring of iodine concentrations in the spectrophotometer sample cell were performed over the periods associated with bubble removal. The sampler performance on turbid samples was evaluated for throughput using Styrene Divinyl Benzene (SDVB) micro-sphere turbidity standards, with no loss of turbidity or fouling of lumens observed. Analyzer compatibility for turbidity standards was evaluated for accuracy, precision, sample carryover, and potential fouling of the flowthrough cells, using formazin standards between 1 and 25 NTU in concentration. For color determinations, platinum/cobalt colorimetric standards were prepared according to standard EPA, method 110.2 specifications, and evaluated spectrophotometrically at 455nm (referenced to 631nm).

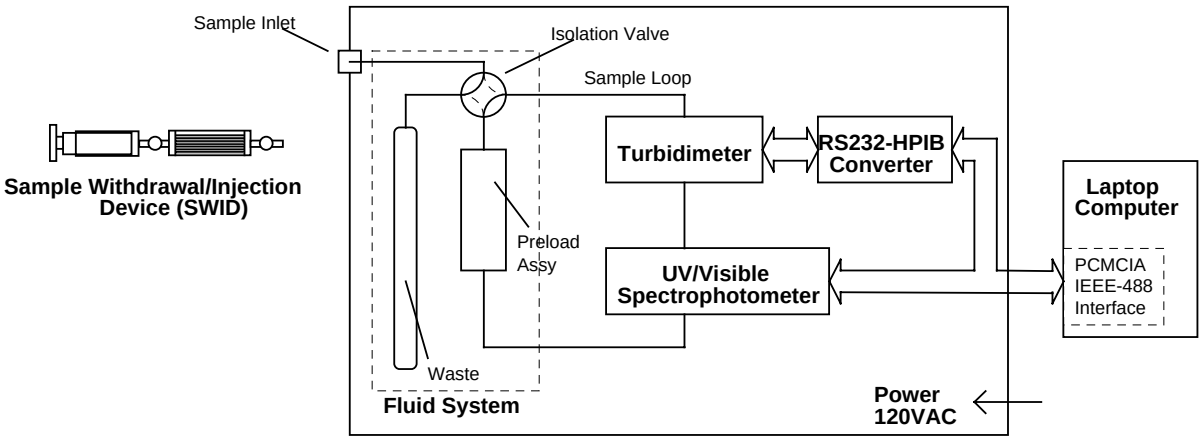

Figure 3, Spectrophotometer System Schematic, Illustrating the Sampler (SWID), Fluid Loop, and Communication Bus to an External Laptop Computer

RESULTS

ANALYTICAL PERFORMANCE

Analytical performance was evaluated against limits of detection and accuracy over analyte concentration range for sample turbidity, color, and iodine species concentrations, with the results listed in Table I. Iodine speciation and color parameters were computed using solution absorbances determined for the 10 cm pathlength flowthrough sample cell in the HP8453 spectrometer; turbidity was evaluated using the separate ratio turbidimeter.

Table I, Precision and Accuracy Results for Injected Water Samples in the Prototype Analyzer.

Parameter	Range	MDL	Accuracy
Turbidity	0 - 40 NTU	0.06 NTU	+/- 5% of reading
Color (Pt/Co)	0 - 100 Pt/Co	0.4 Pt/Co	+/- 1 Pt/Co
Iodine	0 - 5.0 mg/L	0.02 mg/L	0.1 mg/L
Triiodide Ion	0 - 0.5 mg/L	0.002 mg/L	0.03 mg/L

GAS REMOVAL WITH SWID

Membrane-Based Deaeration: A polypropylene membrane-based deaerator design illustrated in Figure 2 was selected from preliminary studies on the efficiency of polypropylene, rayon, and microporous PTFE membranes in scale model tests. Test articles were prepared using five-inch lumens potted in polycarbonate shells. These test articles were evaluated for deaeration efficiency and compatibility with turbidity standards and iodine solutions.

Efficiency of air removal was determined as function of pressure drop across the membrane, flow rate through the lumens, and sampler orientation. While the efficiency of a membrane-based deaerators in a microgravity environment is difficult to predict based on one-g tests, operation of this assembly was characterized under worst case orientations (in this case to draw and expel samples vertically (up) through the lumens, allowing buoyancy to minimize bubble/membrane contact). For these tests, 2.5 ml aliquots of air were injected into 50 ml water samples as those samples were drawn into a sample syringe. These tests demonstrated efficient degassing of 50 ml samples when the sampling flow was controlled to a rate of 50 ml/minute as long as the initial shell vacuum was greater than 480 mbar below atmospheric. SWID operation with a 100 ml sample syringe consistently provided a vacuum of 600 mbar or greater with respect to ambient.

The SWID was not only effective at eliminating free gas interferences, but it substantially reduced dissolved gas concentrations to levels consistent with the deaeration membrane back side pressure. Levels of saturation for input and output sample streams were evaluated by measuring evolved gas as these liquids were heated to

80°C. Solutions saturated at room temperature were deaerated and degassed to approximately 50% saturation (evolving less than 0.1 ml air per liter sample at 80°C) after transfer through lumens biased with a 480 to 640 mbar vacuum. The degassing of samples to less than 50% saturation provides significant protection

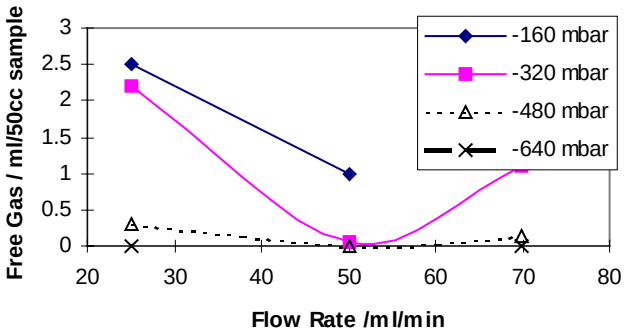

Figure 4, Residual Free Gas vs. Flow Rate and Applied Vacuum

against the formation of bubbles on sample cell windows following introduction of samples into the instrument.

The SWID design performed well with respect to sample deaeration and compatibility with turbid samples. However, iodine reactivity with the epoxy potting compound provided more of a challenge. Initial tests with standard iodine/iodide ion solutions showed a significant fraction (up to 30%) of the iodine sampled was reduced to iodide ion upon contact with the sampler. Discoloration was limited to the epoxy potting compound used to seal the polypropylene membrane lumens in the SWID outer tube; the lumens themselves showed no reactivity in assembled units or independent soak tests. Reactivity could be reduced with extended pretreatment of SWID units to solutions of iodine, but accuracy requirements could not be satisfied at nominal (5 ppm iodine) biocide concentrations. The stability of the lumen material in the iodine solutions prompted an investigation into the use of a polypropylene-based thermal set potting compound. The modified SWID improved iodine chemical compatibility, attenuating free iodine by no more than 0.1 mg/liter in solutions of 0.3 to 5 mg/liter. However, the thermal set adhesive failed to yield a leak-free seal of the lumens into the polycarbonate shell unless a barrier of epoxy was backfilled behind the polypropylene. Additional work is required to improve chemical compatibility while assuring leak-free bonding of the lumens in the potting compound.

GAS REDISSOLUTION WITH PRESSURE

Hyperbaric redissolution of free gas in liquid samples is an extremely simple and effective way to mitigate or prevent free gas in microgravity environments. Advantages of the method include that it is demonstrably independent of orientation and therefore verifiable in 1G tests, and that all gas in a system is removed eventually, irrespective of proximity to a degas module. When used with deaerated solutions, a hyperbaric sample loop prevents any subsequent formation of bubbles in the spectrometer sample cell as the sample warms. The principal disadvantage of hyperbaric degassing is speed – dissolution is slow in unmixed fluids, limited by diffusion of dissolved gas from a saturated boundary layer at the bubble/liquid interface. Dissolution speed is a function of overpressure, particularly when excess pressure can drive bubbles below the critical diameter, as determined by the fluid (low ionic strength water) and its temperature.

Table II identifies the minimum pressures required to dissolve excess gas in saturated solutions, for gases found in various Apollo program water supplies (nitrogen, oxygen, and hydrogen) or expected in humidity condensates (air and carbon dioxide). At a maximum 5% by volume free gas load with air as the entrained gas, a gauge pressure of 3 atmospheres is sufficient to dissolve all entrained gas, assuming that the gas is evenly distributed throughout the sample.

Table II, Gauge Pressures (in atmospheres) Required for Dissolution of Entrained Gas in 25°C Water Samples

Gas	Solubility[18] (mole fraction)	Free Gas Load, Percent by Volume				
		1%	2%	3%	4%	5%
Air		0.6 atm	1.1 atm	1.6 atm	2.2 atm	2.7 atm
O_2	2.29×10^{-5}	0.3	0.6	1.0	1.3	1.6
N_2	1.18×10^{-5}	0.6	1.3	1.9	2.5	3.1
H_2	1.41×10^{-5}	0.5	1.0	1.6	2.1	2.6
CO_2	6.15×10^{-4}	always less than 1 atm, gauge				

If 2.7 atmospheres is sufficient for entrained air, and 3.1 atmospheres is sufficient for the worst-case gas, preloads over 5 atmospheres should lead to the dissolution of all free gas as long as the gas load is below 5% by volume and that gas is evenly distributed in tiny bubbles. However, initial gas dissolution tests indicated that microliter-volume air bubbles would not dissolve in less than 10 minutes at preloads below 10 atmospheres and larger (5 microliter) bubbles would not dissolve in less than 70 minutes at this pressure. Should all free gas in a 5 milliliter sample coalesce into a single bubble, a 5% by volume gas inclusion provides for a 250 microliter bubble and an unacceptable dissolution rate. This result is consistent with a saturated interface between the liquid and gas bubble, with further dissolution limited by diffusion of dissolved gas from that interface. Higher pressures can accelerate free gas dissolution, and extrapolations to 500 psig suggest that large bubbles can be dissolved in less than 10 minutes at that pressure. However, a desire to keep preloads below 6 atmospheres led to an evaluation of methods to improve mixing.

Ultrasonic-assisted Hyperbaric Dissolution: Agitation of the samples in the turbidimeter and spectrophotometer cells was required to assure dissolution of moderate size gas bubbles for pressures below 10 atmospheres. The simplest approach to agitation was to bolt piezoelectric transducers to the cell walls and induce ultrasonic waves into the fluid samples. With ultrasonic mixing, gas dissolution for gas loads of 5% or less, may be dissolved in less than 10 minutes in a sample cell pressurized to 10

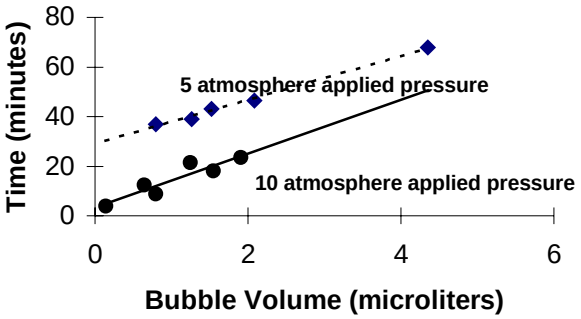

Figure 5, Single Bubble Dissolution Times in 5 Milliliter Sample Volumes as a Function of Time, Bubble Volume, and Applied Pressure.

atmospheres. However, efficient mixing of the sample speeds the known reactivity of iodine with the steel walls of the sample cell. Thus, while the small surface to volume ratio of the sample cell should lead to reactions slow enough for accurate iodine measurements, mixing raises the effective surface to volume ratio and results in losses up to 20% of the spiked iodine in the 10 minute agitation period. A vapor-deposited silica-on-steel coating applied to the inside of the sample cell decreased cell reactivity to Iodine to unmeasurable levels over 10 minute periods when the sample was left undisturbed. Optimization of this coating is required to completely eliminate iodine loss over a 10 minute period during ultrasonic mixing.

DISCUSSION

A brassboard analyzer was constructed to include: a Hewlett-Packard HP-8453 Diode Array UV-Visible spectrophotometer, Hach 2100P ratio turbidimeter, an IBM Thinkpad 750C-compatible computer, and a microgravity-compatible fluid loop. The analytical instrumentation was integrated to operate under control of the laptop computer, using an IEEE-488 data bus between the laptop and the analyzer, and converting the 488 bus to RS232 in the analyzer to control the turbidimeter.

The instrumentation sample cells were converted to minimum-volume, flow-through cells for compatibility with sample transfer in weightless environments. To handle the gas introduced with samples under microgravity conditions, three approaches to gas mitigation were evaluated: hyperbaric dissolution, ultrasonic-assisted hyperbaric dissolution, and membrane deaeration. The sample loop included a mechanism for pressurizing the sample to 10 atmospheres to eliminate free gas that may be introduced with the sample; ultrasonic transducers on the sample cells were ultimately included to externally agitate the fluid to assist gas dissolution at moderately low pressures. In parallel with the hyperbaric dissolution approaches to gas mitigation, a membrane-based deaerator was developed and evaluated. Membrane deaeration, performed on samples at the time of introduction, was demonstrated effective for gas loads up to 5% by volume.

The analyzer demonstrated capability to measure iodine and triiodide concentrations in a microgravity-compatible fluid loop at levels appropriate for potable water systems. Color, spectrophotometric, and turbidity measurements were demonstrated, as was compatibility with mass, envelope, power, and digital interface requirements for operation on both the Shuttle and Space Station platforms.

While the degas mechanisms were successful and the instrumentation and fluid loop met requirements for on-orbit operation, hyperbaric dissolution at 10 atmospheres proved too slow for this application. Ultrasonic-assisted hyperbaric dissolution and membrane deaeration are promising solutions. However, attention must be paid in materials selection and process optimization toward minimizing iodine reactivity.

CONTACT

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