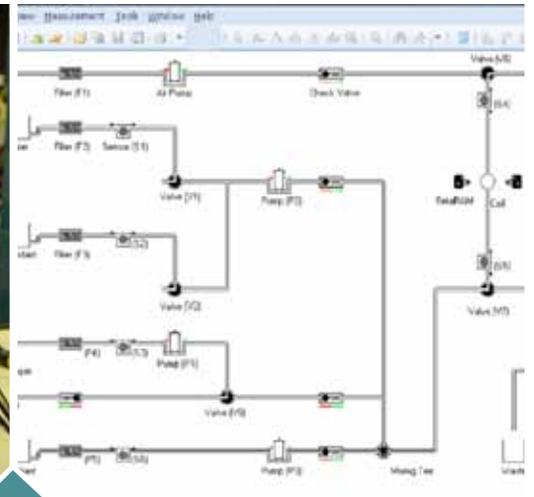
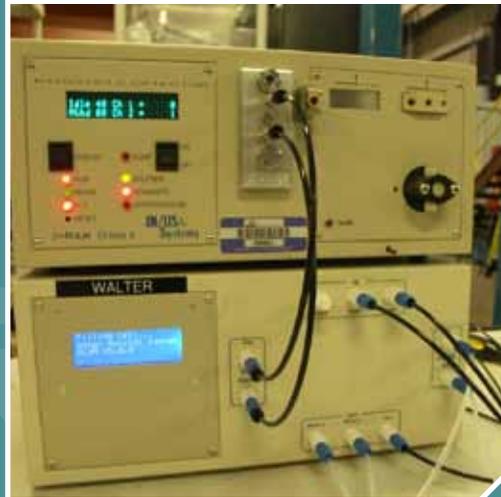


# LabLogic Radiation Detection Online Water Quality Monitoring System



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National Homeland Security Research Center  
Office of Research and Development  
U.S. Environmental Protection Agency  
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## **Disclaimer**

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## Abbreviations/Acronyms

β-RAM	Beta RAM - Online Water Quality Radiation Monitor
Bq	Bequerel
°C	degrees Celsius
cm	centimeters
cpm	counts per minute
DHS	U.S. Department of Homeland Security
dpm	disintegrations per minute
EPA	U.S. Environmental Protection Agency
FoM	Figure of Merit
HPLC	High Performance Liquid Chromatography
keV	kilo electron volts
mL	Milliliter
L	Liter
LAN	Local Area Network
LLS	LabLogic Systems, Inc.
MCA	Multi-Channel Analyzer
Modbus <sup>®</sup>	A serial communications protocol originally published by Modicon (now Schneider Electric)
PAG	Protective Action Guide
PC	Personal Computer
pCi	picocuries
PMT	Photomultiplier Tube
NAREL	National Analytical Radiation Environmental Laboratory
NHSRC	National Homeland Security Research Center
O&M	Operations and Maintenance
OAR	Office of Air and Radiation
ORD	Office of Research and Development
QAPP	Quality Assurance Project Plan
mrem	Milli-Roentgen Equivalent Man (units of radiation dose)
TA	Technical Associates, Inc.
T&E	Test and Evaluation
USB	Universal Serial Bus

## Radionuclides

Am-241	Americium-241
Cs-137	Cesium-137
H-3 or <sup>3</sup> H	Tritium
Sr-90	Strontium-90
Y-90	Yittrium-90

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## Executive Summary

Detecting the presence of radiological substance in drinking water is important both from a consumer safety and a national security perspective. The EPA's Office of Research and Development /National Homeland Security Research Center (NHSRC) routinely acquires and evaluates commercially available radiation detection systems for water. This report summarizes the information collected by EPA NHSRC during the testing and evaluation of the  $\beta$ -RAM scintillation-counting system manufactured by LabLogic Systems, Inc. (LLS).

The  $\beta$ -RAM Model 4 scintillation-counting system (a.k.a.  $\beta$ -RAM) was procured by EPA NHSRC and first operationally evaluated at the EPA Test and Evaluation (T&E) Facility in Cincinnati, Ohio. Subsequently, the unit was tested with radioactive materials at the EPA National Analytical Radiation Environmental Laboratory (NAREL) Facility in Montgomery, Alabama.

The initial operational evaluation at the EPA T&E Facility indicated that the system (after some initial modifications) can be operated and maintained by a typical water treatment plant technician. The system was able to function in a somewhat-rugged test environment that is representative of a typical drinking water monitoring site where the unit can be potentially deployed. The key focus of the testing at EPA NAREL was to determine the system's ability to detect radiation activity in water at levels near the Department of Homeland Security's Protective Action Guide (PAG) level, which is based on a drinking water interdiction of

500 mrem/year. This 500 mrem/year dose rate converts to the following PAG levels for the isotopes tested: (1) Tritium H-3 - 4,540,000 pCi/L, (2) Strontium-90 (Sr-90) - 6,730 pCi/L, (3) Cesium-137 (Cs-137) - 13,800 pCi/L, (4) Americium-241 (Am-241) - 908 pCi/L. The actual injected levels were all well below PAG levels with the exception of Am-241, which was slightly above PAG level. The actual injected activity levels are as follows: Sr-90 (1,120 pCi/L), Cs-137 (1,100 pCi/L), Am-241 (2,480 pCi/L), Am-241 (1,310 pCi/L), H-3 (740 pCi/L), and H-3 (74 pCi/L). Testing at the NAREL Facility indicated that the  $\beta$ -RAM system can be used as an online real-time radiation monitor (response time < 30-minutes). In its current state of development, the  $\beta$ -RAM with the accompanying Wilma software system demonstrated the ability to: (1) detect contamination at the injected levels (as stated above), (2) discharge a sample for retention, and (3) provide an alarm to the user.

The results of the testing also indicate that it may be possible to detect lower-levels of radioactivity in water than those used in these tests. Section 2.0 of this document presents an overview of the technology and describes the operational evaluation and testing. The detailed NAREL radioactivity testing results are presented in Section 3.0. Section 4.0 provides a summary of conclusions and a listing of recommended system enhancements for future testing.

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## 1.0 Introduction

Detecting the presence of radiological substance in drinking water is important both from a consumer safety and national security perspective. In some parts of the U.S., groundwater naturally contains elevated levels of radionuclides. For example, radium-226 occurs at an elevated level in the groundwater of the North-central states (Zapecza and Szabo 1987, EPA 2000). Similarly, uranium is found at elevated levels in the groundwaters of the Colorado plateau, the Western-central platform, the Rocky mountain system, and the Pacific mountain system (Zapecza and Szabo 1987, EPA 2000). From a national security perspective, pursuant to the Fukushima nuclear plant disaster in Japan in March 2011, the U.S. Environmental Protection Agency (EPA) temporarily stepped up routine radiological monitoring of the drinking water. The results (released on April, 2011) from the monitoring of drinking water indicated that only two of the monitored locations detected radiation in drinking water samples, but the values were well below the levels of public-health concern. Another potential security incident occurred in December 2013, when a vehicle containing radioactive cobalt was stolen, but recovered by Mexican authorities (Romo et al., 2013). Looking at these scenarios collectively, one can say that the ability to measure the presence of radiological substance in drinking water in near real-time is important for safeguarding public health and national security. While a variety of equipment is available to detect the presence of radiation in air, the options for water are somewhat limited. To fill the gaps in technologies for keeping drinking water safe, the EPA's Office of Research and Development (ORD)/National Homeland

Security Research Center (NHSRC) routinely acquires and evaluates such commercially available radiological detection systems.

In 2008, the EPA, Office of Air and Radiation (OAR), National Analytical Radiation Environmental Laboratory (NAREL) committed to work with the EPA NHSRC to evaluate the performance of selected radiation detection systems, purchased by NHSRC, relative to manufacturer-supplied performance criteria. The testing described in this report focuses on the ability of the system to accurately monitor and detect radioactive materials in drinking water. After development and acceptance of a proposal to execute the desired tests, a Quality Assurance Project Plan (QAPP) was developed and approved in April 2009 to perform the tests with well defined objectives and goals (EPA 2009, Amended EPA 2012). The  $\beta$ -RAM scintillation-counting system is manufactured by LabLogic Systems, Inc. (LLS) located in Brandon, Florida. The  $\beta$ -RAM Model 4 scintillation-counting system was procured by EPA and first operationally evaluated at the EPA Test and Evaluation (T&E) Facility in Cincinnati, Ohio. Subsequently it was tested with radioactive substances at the EPA NAREL Facility in Montgomery, Alabama. The tests to evaluate operational suitability at the T&E Facility were conducted in an environment that is representative of a typical water distribution system. Following several months of operational evaluation at the T&E Facility, the  $\beta$ -RAM system was delivered to NAREL. The radiological testing facilities at NAREL allowed for testing the radiation detection capabilities of the system by

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introduction of radioactive material at designated activity levels. The reported activity level by the  $\beta$ -RAM system and its accuracy were monitored and evaluated. This document describes the tests performed at both testing facilities and provides some additional information regarding the radio-analytical theory and functional requirements helpful for review and understanding of the test results.

### 1.1 Background

Initially, LLS held discussions with EPA NHSRC regarding the system specifications and desired operational capabilities. Subsequently, representatives from LLS and EPA NHSRC arrived at NAREL with LLS's existing commercially available  $\beta$ -RAM scintillation-counting system. This original  $\beta$ -RAM system is a bench-top offline flow cell scintillation detection system which is normally coupled with a High Performance Liquid Chromatography (HPLC) unit, as employed by radiopharmaceutical companies and oncology facilities to detect basic tracer isotopes such as tritium (H-3), carbon-14, and phosphorus-32.

The LLS personnel demonstrated the standard configuration and operation of their original  $\beta$ -RAM system and requested EPA's input for necessary modifications required to convert this system into an online radiation detection system suitable for deployment in a water distribution system. EPA suggested that the system meet the following criteria:

- 1) The system should be able to be operated by typical water treatment plant operators and technicians.
- 2) Maintenance of the system should be simple and allow for routine replenishment of supplies or checking of instrument responses

- 3) The system should be able to endure the rugged environment of a typical pump station facility. This includes temperature variation and humidity levels that are often elevated.
- 4) A multi-channel analyzer (MCA) should be incorporated into the system with appropriate software. The MCA should be capable of providing information to qualified individuals regarding the nature of the ionizing radiation: alpha decay or beta decay. The MCA should be available as an optional add-on device or upgrade.
- 5) A means of calibrating and checking the operational readiness of the instrument should be established, allowing typical plant operators the ability to check operational readiness of the system.
- 6) The system should be capable of displaying the detected level of radiation and the time stamp; and should provide an alarm at user-defined activity levels to a remote location.
- 7) The alarm set point should be adjustable to an activity level determined by the user based on a site-specific background count rate.
- 8) The system should have the ability to automate the collection of the water sample that created the alarming condition for verification analysis.

- 
- 9) The system should accommodate user-selected and adjustable count times with a maximum polling interval of 30 minutes.
  - 10) The system should have the ability to be operated continuously online as a flow-through sensor without human intervention except for alarm response, reagent replacement, or normal calibration and for Operations and Maintenance (O&M) requirements.

NAREL and NHSRC both agreed that NHSRC would purchase and test the system after LLS completed the system

modifications that would be required to meet the aforementioned criteria. The testing would follow the procedures previously established for the Technical Associates, Inc. (TA) Model SSS-33-5FT water monitoring system (EPA 2009, EPA 2011). A QAPP addendum for testing of the  $\beta$ -RAM system was prepared and approved prior to commencing the tests (EPA 2012). In August of 2011, EPA received the modified  $\beta$ -RAM system. Prior to delivery of the system to EPA, LLS had tested the system for tritium detection. These results are included in Appendix A, *Dilute  $^3\text{H}$  [ $\text{H-3}$ ] Measurements Using a 5 mL Wilma Cell and ScintLogic LB*.

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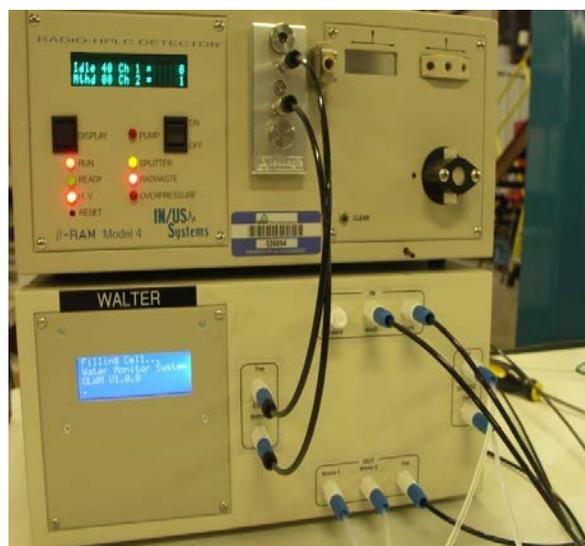
## 2.0 Technology and Testing Description

During decay, radioactive compounds emit ionizing radiation in the form of gamma rays and/or alpha or beta, particles. One type of technology often used for detecting radioactive compounds in liquid samples is scintillation. A scintillator is a material that exhibits scintillation (luminescence) when excited by ionizing radiation. These materials, when struck by ionizing radiation, absorb its energy and scintillate (i.e., re-emit the absorbed energy in the form of light). The emitted light is often detected by using an electronic sensor such as a photomultiplier tube (PMT). The  $\beta$ -RAM device described in this report uses a

proprietary scintillation liquid cocktail and a PMT to detect radioactive compounds present in water. The modified  $\beta$ -RAM device (as tested by EPA) employs a flow-through cell with a radio HPLC detector combination. The features and performance of the testing related to the  $\beta$ -RAM system is described further in this report.

### 2.1 The Modified $\beta$ -RAM Scintillation-Counting System

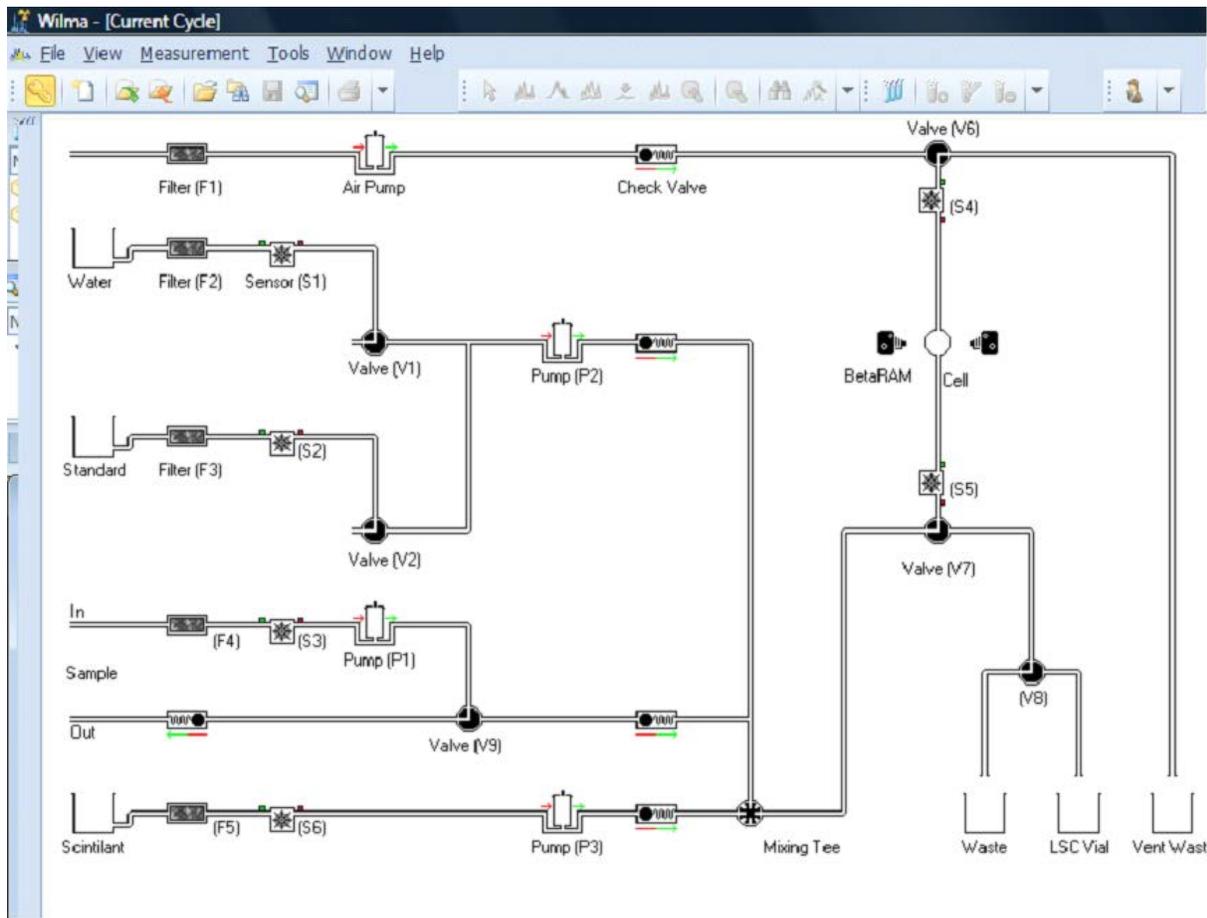
Figure 1 shows a picture of the modified  $\beta$ -RAM scintillation-counting system as tested by EPA.



**Figure 1. Model 4  $\beta$ -RAM scintillation-counting system and Walter fluid handling/distribution unit.**

This system employs three separate modules: (1) Walter – a fluid handling and distribution unit (shown at the bottom of Figure 1), (2)  $\beta$ -RAM – a scintillation-counting system (shown at the top of Figure 1), and (3) a Lynx<sup>®</sup> multi-channel analyzer (MCA) from the Canberra Corporation, Meriden, Connecticut (not shown in Figure

1). The entire system is controlled by Wilma software, a computer program that controls pump operation and valve switching. The sample, scintillant, and wash fluid flow paths set in the Walter module are visualized through Wilma software and illustrated in Figure 2.



**Figure 2. Flow paths in Walter unit and the  $\beta$ -RAM system.**

There are several preset operating cycles included with the Wilma software. Each operating cycle consists of a timed-sequence of events that the instrument performs to complete the measurement cycle. These cycles are incorporated into methods. The methods are essentially electronic files containing the prescribed sequence of instrument operation placed in file folders on the computer hosting the Wilma software for sets of common analyses. Both NHSRC and NAREL methods were set by LLS for

the testing conducted at each facility. Though different time cycles were employed at each facility, they are all saved under the respective facility-specific methods on the PC hosting the Wilma software. A typical cycle on the Wilma software is shown in Figure 3. This cycle chart appears on the PC screen during each cycle operation and provides the user with a direct indication of the current state of the cycle, the time remaining in that portion of the cycle, and the total elapsed time in the cycle.

Complete Cycle (Scint Based)

**Event List**  
Configure the list of events

Event List

Name	Time	Action	Device	Status	Value	Description
Scint Pump	00:00:00.000	Set Device Status	Pump 3	Run	7.000 ml/min	
.	00:02:45.000	Set Device Status	Pump 3	Stop		
.	00:00:00.000	Set Device Status	Valve 9	Close		
Trigger	00:00:00.000	On Threshold Run	Threshold Exc		if > 10000.000 cp	
Display Text	00:00:00.000	Display on LCD (line 4)				Wait for IT!
.	00:00:00.000	Prepare Chromatograph				
Wait	00:07:00.000	Wait				
Display Text	00:00:00.000	Display on LCD (line 4)				.
Display Text	00:00:00.000	Display on LCD (line 1)				Counting Sample...
Count	00:00:00.000	Start Chromatograph				
Stop Count	00:10:00.000	Finish Chromatograph				
Display Text	00:00:00.000	Display on LCD (line 1)				Emptying Cell...
Empty	00:00:00.000	Set Device Status	Valve 6	Open		
.	00:00:00.000	Set Device Status	Valve 7	Open		
.	00:00:00.000	Set Device Status	Pump 4	Run	7.000 ml/min	
.	00:03:30.000	Set Device Status	Pump 4	Stop		
Close Vial Valve	00:00:00.000	Set Device Status	Valve 8	Close		
.	00:00:00.000	Set Device Status	Valve 6	Close		
.	00:00:00.000	Set Device Status	Valve 7	Close		
Text	00:00:00.000	Display on LCD (line 1)				Washing cell...
Wash	00:00:00.000	Set Device Status	Valve 1	Open		
.	00:00:00.000	Set Device Status	Pump 2	Run	7.000 ml/min	
.	00:01:05.000	Set Device Status	Pump 2	Stop		
.	00:00:00.000	Set Device Status	Valve 1	Close		
Empty again	00:00:00.000	Set Device Status	Valve 6	Open		
.	00:00:00.000	Set Device Status	Valve 7	Open		
.	00:00:00.000	Set Device Status	Pump 4	Run	7.000 ml/min	
.	00:01:30.000	Set Device Status	Pump 4	Stop		
.	00:00:00.000	Set Device Status	Valve 6	Close		
.	00:00:00.000	Set Device Status	Valve 7	Close		
Wash Again	00:00:00.000	Set Device Status	Valve 1	Open		
.	00:00:00.000	Set Device Status	Pump 2	Run	7.000 ml/min	
.	00:01:05.000	Set Device Status	Pump 2	Stop		
.	00:00:00.000	Set Device Status	Valve 1	Close		
Final Empty	00:00:00.000	Set Device Status	Valve 6	Open		
.	00:00:00.000	Set Device Status	Valve 7	Open		
.	00:00:00.000	Set Device Status	Pump 4	Run	7.000 ml/min	
.	00:02:30.000	Set Device Status	Pump 4	Stop		
.	00:00:00.000	Set Device Status	Valve 6	Close		
.	00:00:00.000	Set Device Status	Pump 1	Stop		
.	00:00:00.000	Set Device Status	Valve 7	Close		
Text	00:00:00.000	Display on LCD (line 1)				Waiting...

Click Next to continue.

Figure 3. Typical Wilma software operating cycle.

## 2.2 Installation and Testing at EPA NHSRC - Cincinnati

In September 2011, NHSRC received the Model 4  $\beta$ -RAM system at the EPA T&E Facility in Cincinnati. On September 14, 2011, the LLS representatives arrived at the T&E Facility to set up the system and provide operational instructions to the NHSRC and Shaw Environmental & Infrastructure, Inc. project staff.

Operational set-up was fairly straightforward. Both Walter and the  $\beta$ -RAM modules require a Universal Serial Bus (USB) connection to the PC on which the Wilma software resides. The Canberra Lynx MCA module requires either a USB or Local Area Network (LAN) connection to the PC. In addition, there are other interconnecting cables that connect these devices. A number of small tubes are used as fluid lines in the Walter module to draw water from the sample source, the wash fluid source (50% mix of water and methanol), the scintillation fluid source, and associated sample capture/drain lines. The pumps and valves located in the Walter module perform all the operations to deliver and discharge fluid from the  $\beta$ -RAM module. The  $\beta$ -RAM module has only one set of fluid lines connecting to and from the flow cell chamber used for measuring the radiation activity. As mentioned previously, the radiation activity is measured via a scintillation counter, which is an instrument for detecting light/photon counts. The Scintillator generates photons of light in response to incident radiation; these photon counts are cascaded using a PMT. The measured cascaded photon counts are referred to as “raw counts.”

At the T&E Facility, the three components of the radiation detection system, i.e., the Model 4  $\beta$ -RAM scintillation-counting system unit, the Walter fluid handling and

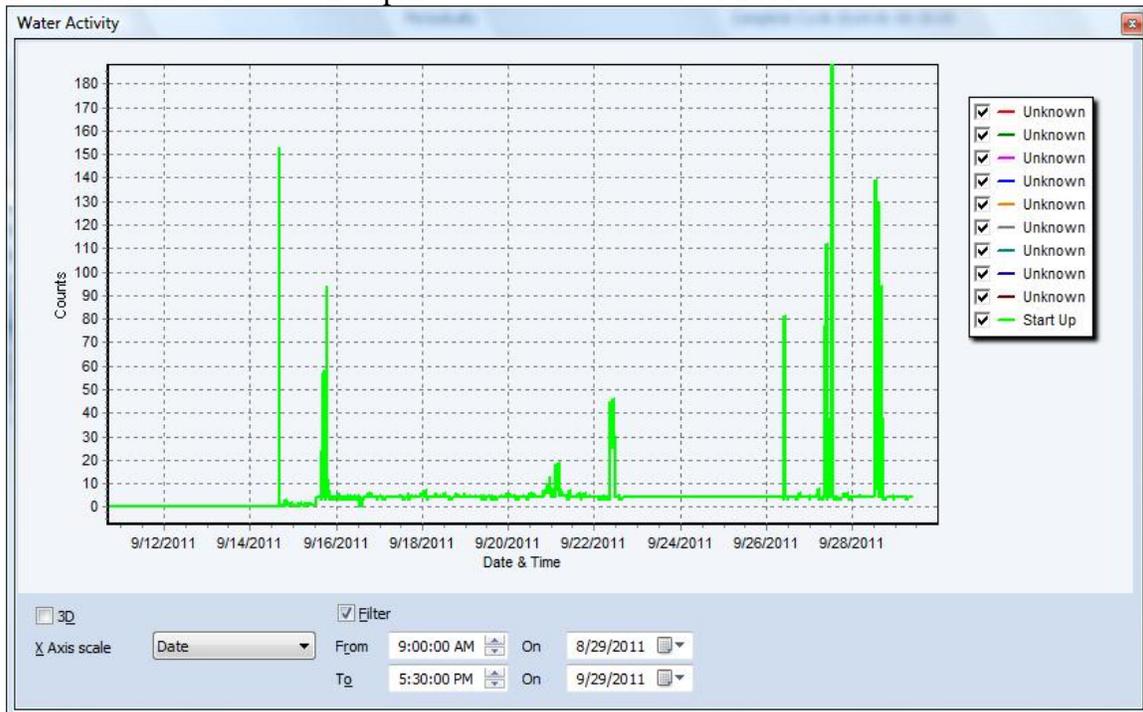
distribution unit, and the Wilma software, were operational. Although the Canberra Lynx MCA hardware was provided by LLS, the software license for utilizing the MCA was initially not provided to EPA. During the initial discussions, the LLS representatives informed EPA that they were in the process of developing their own MCA and wanted to avoid the cost related to acquiring the Canberra software until necessary. As no radiological testing was proposed to be conducted at the EPA T&E Facility, LLS agreed that either the Canberra software or the alternative LLS product (if developed and ready) would be provided when the unit was ready for shipment to the EPA NAREL laboratory for radiological testing. Meanwhile, a dummy MCA graphic spectrum was provided as a place-holder in the software setting. At the completion of the initial setup in Cincinnati, the only available interpretable water quality output from the system was the raw counts.

After the installation of the system was deemed complete, EPA proceeded to evaluate the first three criteria previously identified as Operational Parameters 1 through 3 in Section 1.1 of this report. The potential option for evaluating Operational Parameter 6 (i.e., displaying the alarm and other sample information to a remote location) was also discussed during the initial meeting. The specific options discussed to facilitate Operational Parameter 6 included: (1) file transfer via the existing LAN, and (2) using a standard online instrument data transfer protocol such as Modbus<sup>®</sup>. (The Modbus<sup>®</sup> protocol is a serial communications protocol originally published by Modicon, which is now owned by Schneider Electric, Palatine, Illinois.) The LLS representative advised that, under normal operations, the LAN card should be connected to the Canberra Lynx MCA. Any future data transfer option would either

require a serial Modbus protocol or additional LAN card to be installed on the Wilma-PC for interfacing with the Lynx MCA. As the current LAN port was not utilized for Lynx connectivity, to potentially test for simple file data transfer, the PC (loaded with Wilma software) was enabled to utilize the LAN for network access so that data could be transferred to a remote location.

While the initial evaluation was underway at the T&E Facility, it was noted during September that there were several days when the instrument raw counts spiked

intermittently from the background levels between 0 and 10 to over 100 ( Figure 4). LLS representatives were contacted via email about this problem. After some troubleshooting efforts, LLS determined that these peaks were the result of light leaks and suggested covering the tubes connecting Walter unit and the  $\beta$ -RAM scintillation-counting system with black electrical tape. Light leaks increase the background photon counts, which is unrelated to the tested radiation activity present in the sample. The black electrical tape was applied and the system was continually monitored during October 2011.



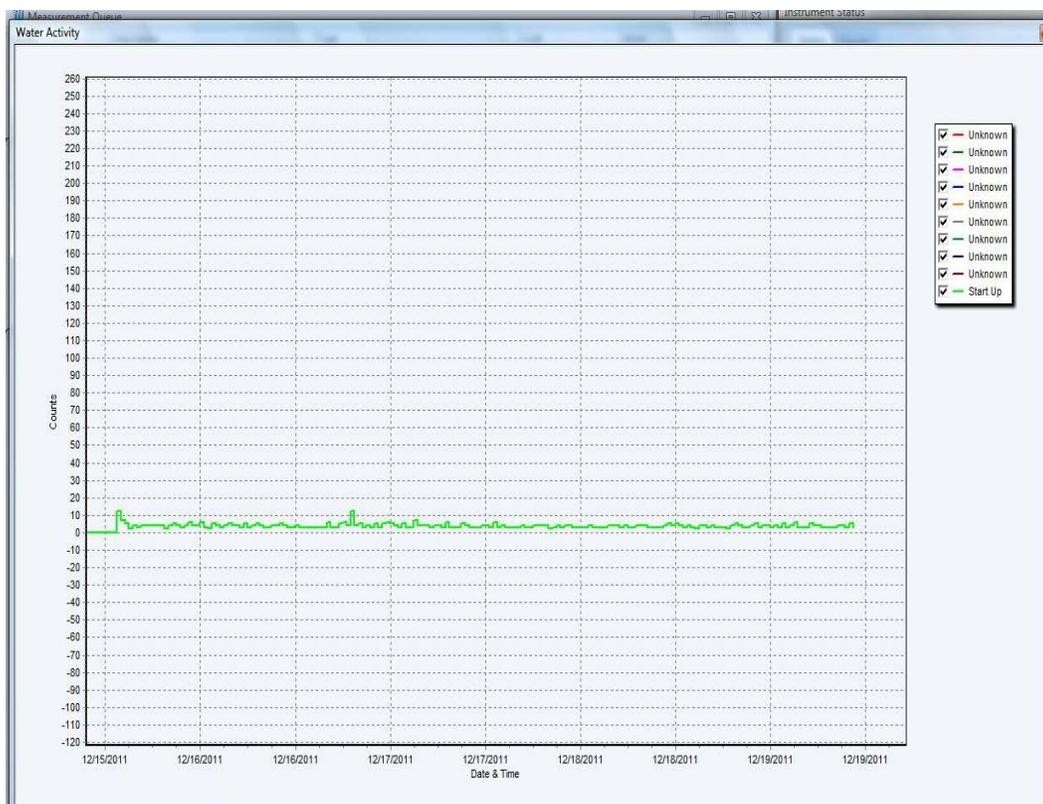
**Figure 4.  $\beta$ -RAM system initial (raw count) data.**

In November 2011, EPA informed LLS that the  $\beta$ -RAM module was not communicating with the Wilma software and the module display was malfunctioning. Subsequently, LLS serviced the system by installing and upgrading the software. The signal processor was re-seated which corrected the display malfunction. In addition, LLS provided

documentation for serial Modbus interface to collect data remotely. Implementing the Modbus serial interface on the existing data logger at the T&E Facility required some upgrades; hence, the Modbus implementation was temporarily delayed. An additional system malfunction (bad display) was observed in December 2011.

In response, LLS shipped a replacement module. However, the periodic spikes continued to be observed in the data. LLS provided EPA with new black Teflon-coated tubing (see Figure 1) to eliminate the light leaks, which significantly improved the data

quality as shown in Figure 5. As a final/permanent resolution, LLS redesigned the unit with stainless steel interconnecting tubes to eliminate the light leaks. The stainless steel interconnecting tubes were received and installed in January 2012.



**Figure 5.  $\beta$ -RAM system data (raw count) after replacement of the tubing.**

The design modification of using stainless steel interconnecting tubes resulted in the elimination of periodic light leaks. As shown in Figure 5, the normal background counts were reduced close to single digits and remained relatively flat without any background spikes in the raw data when compared to the previously shown Figure 4. The reagent/fluid use was monitored and replaced as needed. After over a month of stable background raw count observation and trouble-free operation, it was determined now the system was in a form

that could be operated and maintained by a typical water treatment plant technician. Subsequently, the unit was prepared for shipment to the NAREL laboratory for radiological testing.

### 2.3 $\beta$ -RAM Operating Cost Summary

Besides the nominal electric use of the system and the associated PC (which were not monitored), the main operational costs were the scintillant reagent and the methanol

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for preparing the wash water. The unit, as tested at the EPA T&E Facility, was configured to use these fluids at ~ 5 ml/min and the cycle time/use varied with the overall sampling rate. The unit was programmed to sample once every half hour and it used approximately one gallon per week of lab grade methanol (i.e., one case of methanol per month at a cost of ~ \$361/month) and one gallon of scintillant reagent per month (~ \$195/month). The scintillant (Flowlogic U from LLS, Brandon, Florida) used for this testing is considered non hazardous per the MSDS sheets. The total monthly cost (\$556) is based on the 5 ml/min and ½ hour cycle times as tested in Cincinnati at the EPA's T&E facility. Once the unit was fully operational, it did not require much oversight other than the weekly (methanol)/monthly (scintillant) replacement of the fluids. The fluid replacement frequency could be decreased by using fluid storage containers greater than 1 gallon. The exact consumption rate of the fluids would depend upon the operational input requirements. For example, to obtain cleaner signals, the wash cycle between measurements could be doubled leading to increase in methanol use and the monthly operational costs. The cost data presented above were based on the reagent prices quoted at the time of testing in the year 2011.

#### **2.4 Installation at EPA NAREL Laboratory - Montgomery, Alabama**

In May of 2012, NHSRC transferred the system to NAREL for radiological testing. LLS representatives assisted EPA NAREL in setting up and initializing the system for operation. Several challenges resulted from

the storage and transfer of the system from EPA Cincinnati operations to EPA NAREL, which are listed below:

1. The instrument had to be cleaned out. Several weeks of non-operation had resulted in bio-fouling of the instrument lines.
2. The instrument sample pump had failed as a result of bio-fouling, sealing the micro pump passages. The pump was replaced with a larger pump.

During the enabling process of the MCA, it was observed that the MCA spectrum signal was low compared to the  $\beta$ -RAM output. Subsequent troubleshooting activities discovered that the third-party MCA chosen by LLS (Canberra – Lynx model) required a pre-amplifier when used in conjunction with the instrument's PMT output. The configuration the manufacturer uses with their own brand of scintillation detectors incorporates a pre-amplifier into the output of the PMT. However, at the time this testing was conducted, LLS did not have a pre-amplifier in-line with the output signal to the MCA. Therefore, to improve the Lynx MCA output to match the  $\beta$ -RAM output, the gain setting of the MCA was changed from a "times 4 setting" to a "times 256 setting." By increasing the gain setting, a sufficient increase in pulse count was achieved to allow for a distinction between background and sample activity. LLS is incorporating a modification with the next generation system to place the MCA internally into the  $\beta$ -RAM and to have a pre-amplifier included. The radiological testing of the system at NAREL began in August 2012.

## 2.5 $\beta$ -RAM Calibration and Testing at EPA NAREL

EPA NAREL proceeded to test the system's capabilities previously identified as Operational Parameters 4 through 10 in Section 1.1 of this report. The key focus of the testing was to determine the system's ability to detect radiation activity in water at levels near the Department of Homeland Security's (DHS) Protective Action Guide (PAG) level, which is based on a drinking water interdiction of 500 mrem/year (DHS 2008). This 500 mrem/year dose rate converts to the following PAG levels for the isotopes tested: (1) Tritium H-3 - 4,540,000 pCi/L, (2) Strontium-90 (Sr-90) - 6,730 pCi/L, (3) Cesium-137 (Cs-137) - 13,800 pCi/L, (4) Americium-241 (Am-241) - 908 pCi/L.

Testing for radiological response consisted of the following steps: 1) ensuring that background values are stable, 2) determining the response to a calibration source material (Sr-90), and 3) introducing prepared radioactive solutions of known activities and determining the response relative to the calibration and measured background level. The solutions of the following four radioisotopes were employed: H-3, Sr-90, Cs-137, and Am-241. Counts were repeated a minimum of 20 times to determine statistical variation in the counts. For the purposes of this testing, a response is considered to be above background level if it is at least three standard deviations above the background level (EPA and DHS, 2008).

NAREL began using the  $\beta$ -RAM to determine the total background counts in clean water. The background sample counts of 25 runs resulted in a mean count rate of  $18.5 \pm 5.7$  cpm (counts per minute). The reported error range represents three standard deviations (see Section 3.0 for raw data and further discussion). During the

next 25 runs, a prepared solution (with known activity level) of Sr-90 was introduced into the system. With decay correction, the solution had a stated value of 413.76 Bq/L or 11,180 pCi/L of Sr-90. Sr-90 decays to Y-90 with a half-life of 64 hours. The Sr-90 solution was produced in 2007 and therefore is at full equilibrium with Y-90. The Sr-90 has an average beta decay energy of 200 keV, while Y-90 has an average beta decay energy of 931 keV. These energy levels bracket many of the typical beta energies measured in environmental samples. The maximum beta energy of Y-90 is 2,245 keV, which is at the lower end of the energy emitted by potential alpha decays. This peak enables a qualified individual to identify potential cross-over measurements of high energy betas being detected as low energy alphas.

It should be noted that the MCA provides a measure of pulse height and does not record the pulse timing. The alpha decay time is often 150 to 200 nanoseconds delayed from a beta/gamma peak, which is not differentiated by this device. Therefore, it is possible to erroneously count a delayed alpha light peak in the MCA spectrum. Pulse height will not show this difference. In order to properly calibrate for alpha beta analysis, an alpha source (Am-241 solution) was also prepared and used. Calibrations of this manner should be performed by an individual appropriately trained in radio analytical measurements beyond that required of a typical operator of the system. Calibration of the instrument was performed by pumping the prepared solutions through the sample lines and waiting for a repetitive stable response. It is feasible that this calibration could be performed in the field by simply swapping the existing sample cell with a static cell containing the calibration source and cocktail solution.

### 3.0 Experimental Results

As with the calibration/background level determination procedures described in Section 2.5, solutions of known activity levels were prepared and introduced into the system and analyzed between 20 and 25 times each to ensure stable readings. The following solutions were prepared by EPA NAREL onsite personnel using NIST traceable source materials and verified at the listed activity levels:

- Sr-90 at 1,129 pCi/L
- Cs-137 at 1,100 pCi/L
- Am-241 at 2,480 pCi/L and 1,310 pCi/L
- H-3 at 740 pCi/L and 74 pCi/L

The results of these tests are described in this section. The  $\beta$ -RAM counts are measured/detected using two sensors: Radio 1 and Radio 2. The instrument outputs provide both values. The Radio 1 output is a processed signal, which uses proprietary chemi-luminescence and background subtraction algorithms. The Radio 2 output is simply the raw counts without any data processing. The results presented include instrument output from Radio 1 and Radio 2.

However, only the processed signal values from Radio 1 have been interpreted in this section as recommended by LLS. The  $\beta$ -RAM system was setup to count for 10 minutes, to get results in near real-time as will be required for a typical drinking water contamination warning system. The tabulated 10-minute counts of Radio 1 are converted to counts per minute (cpm) to evaluate the system performance.

The remaining solutions of Sr-90, Cs-137, Am-241, and H-3 not used for this testing remain under the control and ownership of NAREL at the Gunter Air Force Base in Montgomery, Alabama per the Interagency Agreement used to perform this work. The solutions, methanol, and scintillant used during this testing were disposed in accordance with the applicable permits for the NAREL Facility.

#### 3.1 Background Determination

Table 1 summarizes the raw results from the 25 background runs (112 through 136).

**Table 1.  $\beta$ -RAM System Background Output (Counts) Water Analysis Summary**

Run#	Radio-1	Radio-2	Run#	Radio-1	Radio-2
112	194	373	125	160	414
113	218	364	126	178	390
114	159	346	127	156	369
115	201	387	128	186	394
116	178	374	129	185	363
117	157	342	130	207	384
118	204	355	131	196	373
119	157	365	132	190	346
120	186	351	133	180	340
121	162	377	134	179	364
122	184	371	135	195	394
123	196	340	136	209	406
124	216	326	Average	185.3	
			Std. Dev	18.9	

As shown in Table 1, the average Radio 1 reported values over the programmed 10-minute period was 185.3 raw counts with a standard deviation of 18.9 raw counts for the background runs. This value, divided by the 10-minute counting period, results in an average background level of 18.5 cpm with a standard deviation of 1.9 cpm. For the purposes of this report, an error range of three standard deviations from the mean was used as an indication of radiation detection.

Therefore, any detected level over 24.2 cpm ( $=18.5 + 3 \times 1.9$ ) in this operation mode (using 10-minute counts) would be considered as detectable.

### 3.2 Sr-90 Test Run Results

Table 2 summarizes the raw results from the 23 Sr-90 test runs (89 through 111) at an activity level 1,120 pCi/L.

**Table 2.  $\beta$ -RAM System Output (Counts) for Sr-90 (1,120 pCi/L) Water Analysis Summary**

Run#	Radio-1	Radio-2	Run#	Radio-1	Radio-2
89	715	2250	102	741	2435
90	738	2208	103	714	2303
91	714	2280	104	964	2412
92	705	2210	105	701	2289
93	735	2334	106	820	2371
94	688	2234	107	651	2387
95	686	2292	108	685	2367
96	717	2163	109	687	2350
97	766	2284	110	675	2379
98	666	2261	111	633	2345
99	679	2303	Average	714.8	
100	683	2282	Std. Dev	67.2	
101	677	2310			

As shown in Table 2, the average Radio 1 reported values over the programmed 10-minute period was 714.8 raw counts with a standard deviation of 67.2 raw counts for the Sr-90 runs. This value, divided by the 10-minute counting period, results in an average level of 71.5 cpm. This mean value is well above the established detection level

of background plus three-sigma (24.2 cpm) at the tested activity level.

### 3.3 Cs-137 Test Run Results

Table 3 summarizes the raw results from the 23 Cs-137 test runs (137 through 159) at an activity level 1,100 pCi/L.

**Table 3.  $\beta$ -RAM System Output (Counts) for Cs-137 (1,110 pCi/L) Water Analysis Summary**

Run#	Radio-1	Radio-2	Run#	Radio-1	Radio-2
137	487	782	150	510	833
138	503	855	151	511	715
139	537	796	152	540	747
140	543	871	153	479	774
141	550	831	154	538	765
142	484	837	155	522	802
143	513	794	156	532	861
144	486	723	157	537	776
145	529	819	158	532	799
146	525	821	159	501	849
147	532	747	Average	518.9	
148	546	757	Std. Dev	21.9	
149	498	786			

As shown in Table 3, the average Radio 1 reported values over the programmed 10-minute period was 518.9 raw counts with a standard deviation of 21.9 raw counts for the Cs-137 runs. This value, divided by the 10-minute counting period, results in an average level of 51.9 cpm. This mean value is well above the established detection level

of background plus three-sigma (24.2 cpm) at the tested activity level.

### 3.4 Am-241 Test Run Results

Table 4 summarizes the raw results from the 23 Am-241 test runs (160 through 182) at an activity level 2,480 pCi/L.

**Table 4.  $\beta$ -RAM System Output (Counts) for Am-241 (2,480 pCi/L) Water Analysis Summary**

Run#	Radio-1	Radio-2	Run#	Radio-1	Radio-2
160	290	1302	173	360	1303
161	276	1284	174	281	1232
162	291	1266	175	307	1265
163	263	1231	176	237	1292
164	248	1388	177	259	1334
165	291	1342	178	296	1249
166	314	1257	179	352	1256
167	290	1332	180	219	1282
168	255	1252	181	317	1259
169	265	1251	182	277	1321
170	269	1323	Average	297.1	
171	574	1249	Std. Dev	68.8	
172	302	1252			

As shown in Table 4, the average Radio 1 reported values over the programmed 10-minute period was 297.1 raw counts with a standard deviation of 68.8 raw counts for the Am-241 runs at 2,480 pCi/L level. The standard deviation is high because Test Run 171 reported a high count of 574. This value, divided by the 10-minute counting period, results in an average level of 29.7 cpm. This mean value is above the

established detection level of background plus three-sigma (24.2 cpm) at the tested activity level.

Am-241 was also tested at an activity level of 1,310 pCi/L, which is closer to the PAG level. Table 5 summarizes the raw results from these 20 Am-241 test runs (183 through 202).

**Table 5.  $\beta$ -RAM System Output (Counts) for Am-241 (1,130 pCi/L) Water Analysis Summary**

Run#	Radio-1	Radio-2	Run#	Radio-1	Radio-2
183	356	1061	196	410	1021
184	319	1012	197	424	1015
185	376	981	198	378	1000
186	422	981	199	291	1004
187	337	1063	200	384	990
188	376	999	201	356	999
189	316	1069	202	382	1017
190	308	973	Average	362.6	
191	376	942	Std. Dev	39.5	
192	383	980			
193	301	960			
194	399	1018			
195	358	970			

As shown in Table 5, the average Radio 1 reported values over the programmed 10-minute period was 362.6 raw counts with a standard deviation of 39.5 raw counts for the Am-241 runs at a 1,130 pCi/L level. The overall response is counter intuitive when compared to the other Am-241 test runs. Those tests were conducted using higher Am-241 activity level stock, but they reported lower overall counts. However, counting efficiencies of scintillation detection methods can vary for many reasons, such as differences in sample and scintillation cocktail compositions. Poor counting efficiency may result from lower

energy to light conversion rate, referenced as scintillation efficiency. Regardless, both sets of values are above the established detection level of background plus three-sigma (24.2 cpm), and therefore Am-241 would be considered as detectable at both of these activity levels.

### 3.5 H-3 Test Run Results

Table 6 summarizes the raw results from the 23 test runs (203 through 225) of H-3 at an activity level 740 pCi/L.

**Table 6.  $\beta$ -RAM System Output (Counts) for H-3 (740 pCi/L) Water Analysis Summary**

Run#	Radio-1	Radio-2	Run#	Radio-1	Radio-2
203	477	667	216	385	732
204	459	717	217	442	690
205	487	701	218	478	701
206	466	734	219	449	686
207	497	692	220	590	798
208	452	695	221	662	837
209	375	682	222	711	872
210	406	689	223	708	933
211	437	732	224	614	846
212	405	656	225	661	869
213	400	736	Average	499.6	
214	483	707	Std. Dev	103.8	
215	447	740			

As shown in Table 6, the average Radio 1 reported values over the programmed 10-minute period was 499.6 raw counts with a standard deviation of 103.8 raw counts for the H-3 runs at 740 pCi/L level. This value, divided by the 10-minute counting period, results in an average level of 50 cpm. This mean value is above the established

detection level of background plus three-sigma (24.2 cpm) at the tested activity level.

H-3 was also tested at an activity level of 74 pCi/L. Table 7 summarizes the raw results from the 23 test runs (227 through 250) for H-3 at this level.

**Table 7.  $\beta$ -RAM System Output (Counts) for H-3 (74 pCi/L) Water Analysis Summary**

Run#	Radio-1	Radio-2	Run#	Radio-1	Radio-2
227	246	677	240	342	627
228	260	649	241	317	651
229	303	652	242	292	667
230	263	632	243	339	606
231	323	668	244	328	622
232	337	671	246	337	620
233	278	670	247	359	670
234	291	631	248	369	689
235	286	612	248	354	673
236	313	665	250	315	650
237	273	638	Average	308.7	
238	290	660	Std. Dev	33.9	
239	284	600			

As shown in Table 7, the average Radio 1 reported values over the programmed 10-minute period was 308.7 raw counts with a standard deviation of 33.9 raw counts for the H-3 runs at 74 pCi/L level. This value, divided by the 10-minute counting period, results in an average level of 30.9 cpm. This mean value is above the established detection level of background plus three-sigma (24.2 cpm) at the tested activity level.

### **3.6 Online Sample Cross Contamination Check**

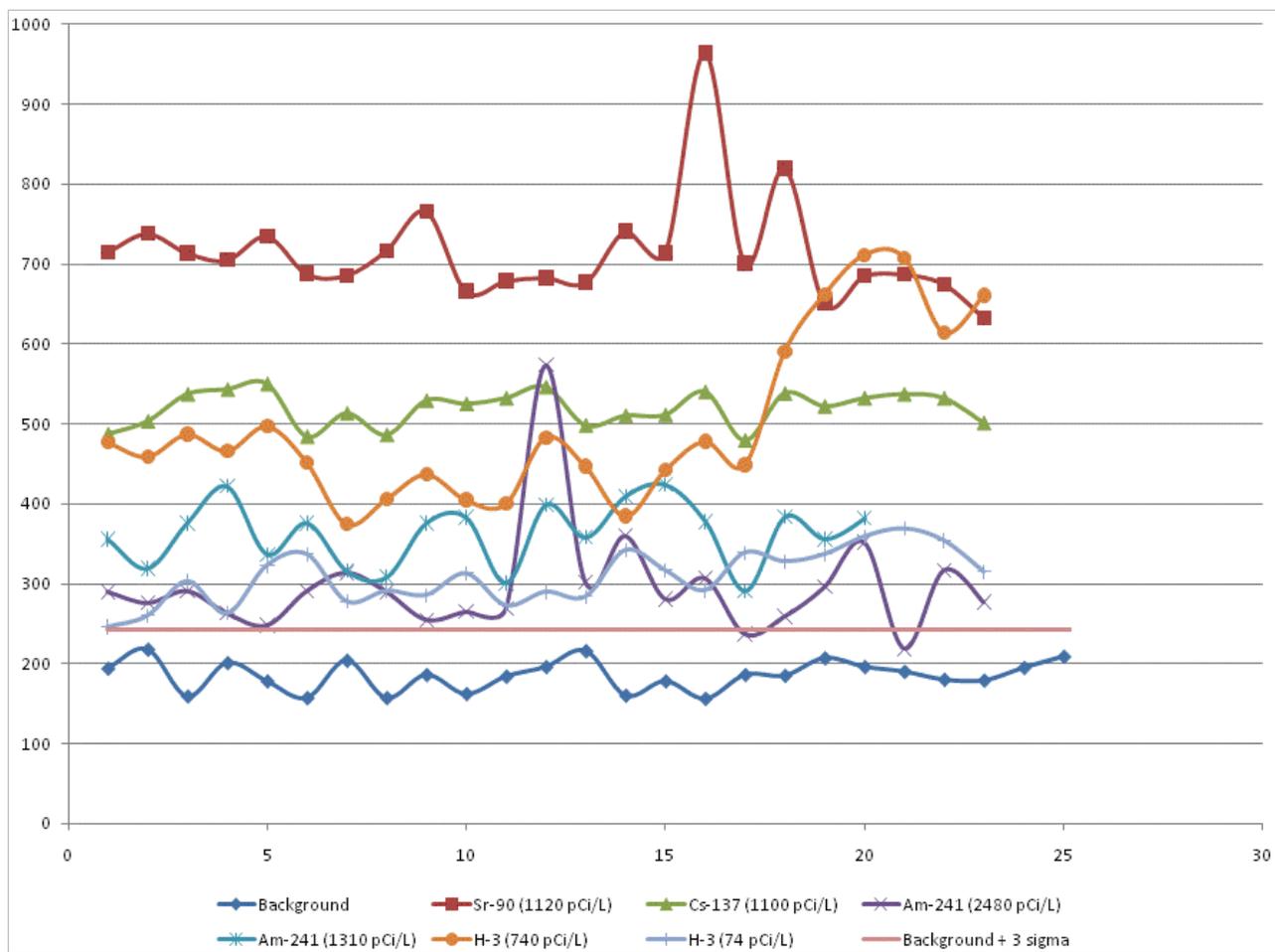
Tests were conducted to determine how long the contamination remained in the system after analysis, during flushing, and subsequent measurements. Under normal operation, the system has a continuous sample flow running through the sample system pump up to the sample divergence valve (previously illustrated in the list of events in Figure 3). However, during the tests conducted at NAREL, this normally continuously running sample flow was terminated to preserve the sample. Subsequently, it provided an opportunity to observe how the system responded to changing contamination levels. The dead volume was noted to pass through the detector with a return to baseline after the third sampling of normal water. There was no indication of retained contamination in the plumbing or the sample cell. It would be expected that, if the sample contamination is cleared out during analysis as the sample is flushed through the sample line, the only retained contamination would be that contained downstream of the divergence valve, a volume less than that of the cell. Monitored activity levels would then drop in the first count after sample analysis.

### **3.7 Event-based Automated Sampling**

A test of the automated event-based water sampling system was performed. The  $\beta$ -RAM system allows the user to set a level of activity in cpm at which the sample, complete with cocktail solution, will be discharged automatically to an awaiting container for further analysis. The system is still under design with respect to the type of container to be used for collection of the sample. As the sample is essentially a scintillation sample, a scintillation cell or container is considered the likely candidate. Multiple cells could be placed into a carousel that could rotate the cells after filling to allow for a flush prior to filling the next cell. One concern noted with the sample collection procedure is the lack of flushing of the line after saving the sample. The line contains microliters of sample media that could be inadvertently added to the next sample, causing some cross-contamination. This effect should not greatly change the contaminant alarm capability of the device, but would likely affect the accuracy of the sample count.

### **3.8 Overall Results Summary**

The results show that all isotopes measured would be detectable near the PAG levels. The overall response of activity in raw 10-minute counts (Radio 1 values), as reported by the instrument (as shown in Tables 1 through 7), are illustrated in Figure 6. It should be noted that these are simple tests of the system; a more extensive testing of the next generation  $\beta$ -RAM system is recommended to determine the lowest detection limits for each compound.



**Figure 6. Overall Summary of  $\beta$ -RAM system testing. Counts (y-axis) versus test runs**

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## 4.0 Conclusions and Recommendations

The test results indicate that the  $\beta$ -RAM system can be used as an online real-time (response less than 30-minutes) radiation monitor. As shown previously in Figure 6, the  $\beta$ -RAM system consistently reported raw count values above the background level plus three-sigma for selected isotopes near or below the PAG activity levels. Based on the system improvement performed during the initial operational evaluation in Cincinnati indicates that the system can be operated and maintained by a typical water treatment plant technician. The system was able to function in a somewhat-rugged test environment.

The LLS  $\beta$ -RAM with the Wilma operating system operated satisfactorily, as designed/modified by the vendor. The system provided an indication of the presence of radioactive material at the measured background level plus three-sigma. The level of detection will be dependent upon the background radiation level of the drinking water supply being tested. In its current state of development, the system clearly demonstrated the ability to (1) detect contamination at the injected levels, (2) discharge the sample for retention, and (3) provide an alarm to the user. The results of the testing also indicate that it may be possible to detect lower levels of activity than those used in this test.

Based on the testing and evaluation data collected during the study, it appears that the instrument is well suited as an online water quality monitor for detecting the presence of radiological contamination at the tested levels. From a field deployment perspective, it is a substantial improvement and can be considered as the “state of the art” for an online radiological water quality monitor at the time when the testing was performed.

However, several system enhancements are recommended to make the device more readily acceptable to the market.

### 4.1 Recommended System Enhancements and Future Testing

The following is a listing of recommended system improvements:

- The MCA module needs further development and integration prior to additional testing. LLS is continuing to refine the integration of the MCA module.
- The industrial robustness of the fluidics needs to be further improved to fit the capabilities of a typical user (or a water technician).
- Vendor must continue to find ways to lower the count times and level of detections from the current tested levels.
- A radiation source and radiation expertise is necessary to calibrate the  $\beta$ -RAM system. These types of facilities (e.g., water utilities) may not be permitted to store or use radioactive calibration sources. Therefore, a “factory calibration” procedure or factory service option may need to be provided along with the sale of the device.
- Although the system provides the capability for programming a set alarm level, further improvements are necessary to fully automate the sample collection system.
- A redesign of the device with “industrial use” focus for the nuclear industry might further expand the market for the device.
- Additional long-term testing needs to be performed for use as a

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contamination warning device; especially, in areas with high background levels of radioactivity in water.

This list of recommendations is not meant to be exhaustive, but more to illustrate the general areas of improvements to further the

use of this technology. Based upon the encouraging test results of this technology presented in this report, the  $\beta$ -RAM technology should continue to be tested and commercialized for wide-spread field applications.

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## 5.0 References

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## Appendix A - Dilute <sup>3</sup>H Measurements Using a 5 mL Wilma Cell and ScintLogic LB - March 2012 (Reprinted with Permission – LabLogic Systems, Inc.)

### Samples

Five <sup>3</sup>H-glycerol samples were prepared by Unilever Research and the activity rates were determined at the point of production using static

counting (10:1 cocktail:sample ratio). Three 1 mL aliquots of each sample were measured over a period of 20 minutes to determine the dpm (counting efficiency ca. 40%). The sample properties are summarized in Table 1:

**Table 1. <sup>3</sup>H Sample Activities**

Sample Ref	Target Activity (dpm/mL)	Actual Activity (dpm/mL)
3H_Dil_1	<50	42.5±0.2
3H_Dil_2	100	121.9±24.2
3H_Dil_3	350	372.0±29.2
3H_Dil_4	600	622.8±13.9
3H_Dil_5	1000	1003.5±7.6

[dpm, disintegrations per minute]

### Sample Preparation

Prior to measurement, each of the five samples was prepared as follows: 3 mL of ScintLogic LB was dispensed into a 7 mL vial using a 2.5 mL disposable needle syringe. 3 mL of sample was then added to the cocktail and the two mixed together vigorously for 60 s. The sample/cocktail mixture was then left to stand for 15 min to allow any air bubbles to diffuse out. When ready for measurement, 5 mL of the sample was syringed into the 5 mL Wilma cell, the cell then being placed into the β-RAM used for all measurements. Each sample was then left to dark adapt in the instrument for a minimum of three hours to ensure that any unwanted luminescent effects were kept to a minimum.

Between each sample, the measurement cell was cleaned thoroughly using a water-diluted methanol solution. The syringes were also changed for each step involving a new sample in order to prevent contamination.

### Measurements of Counting Efficiency

Prior to the measurement of the <sup>3</sup>H samples, the background characteristics of the ScintLogic LB cocktail was measured using a 1:1 cocktail:H<sub>2</sub>O sample over a period of 15 minutes. Five replicates were measured for two different background

samples. The background rate was found to be **8.9±0.4 cpm** for Channel 1 and **25.2±0.5 cpm** for Channel 2.

Each sample was then measured statically in the β-RAM within the recommended counting window of 5-70 in order to determine the average count rate. Both counting channels were used in order to determine the effect of the cross-talk subtraction firmware algorithm. For each sample, three 15 min runs were made, followed by one 30 min and 60 min in order to confirm that the measured count rate did not vary significantly with the counting time.

The counting efficiency for each sample was then determined using the average count rate value obtained from the three 15 min measurements and using the relationship:

$$\text{Counting Efficiency (Eff\%)} = (\text{cpm/dpm})/100$$

This calculation was made for both channels and with/without background subtraction (BS). Using the calculated value of the efficiency, the Figure of Merit (FoM),  $E^2/B$  was also determined. The results are summarized in Table 2, the values quoted with their associated errors.

**Table 2. Counting Efficiency and FoM Values for the Five Samples**

Sample	Average Count Rate (cpm)	Expected Activity Rate in 2.5mL (dpm)	Eff. (%)	E <sup>2</sup> /B
3H_Dil_1 Ch.1; No BS	12.4±0.4	106.3±0.5	<b>11.7±0.4</b>	14.9±0.8
3H_Dil_1 Ch.2; No BS	28.4±0.7	106.3±0.5	<b>26.7±0.4</b>	21±0.5
3H_Dil_1 Ch.1; BS	3.2±0.02	106.3±0.5	<b>3.0±0.02</b>	0.97±0.04
3H_Dil_1 Ch.2; BS	3.2±0.08	106.3±0.5	<b>3.0±0.08</b>	0.35±0.01
3H_Dil_2 Ch.1; No BS	42.5±2.1	304.7±60.5	<b>13.9±2.9</b>	21±4.4
3H_Dil_2 Ch.2; No BS	54.2±3.1	304.7±60.5	<b>17.8±3.7</b>	12.6±2.6
3H_Dil_2 Ch.1; BS	33.3±1.7	304.7±60.5	<b>10.9±2.2</b>	12.9±2.7
3H_Dil_2 Ch.2; BS	29.0±1.7	304.7±60.5	<b>9.5±2.0</b>	3.6±0.8
3H_Dil_3 Ch.1; No BS	66.4±0.8	929.9±73.0	<b>7.1±0.6</b>	5.5±0.5
3H_Dil_3 Ch.2; No BS	72.7±1.4	929.9±73.0	<b>7.8±0.6</b>	2.4±0.2
3H_Dil_3 Ch.1; BS	57.2±0.7	929.9±73.0	<b>6.2±0.5</b>	4.2±0.4
3H_Dil_3 Ch.2; BS	47.5±0.9	929.9±73.0	<b>5.1±0.4</b>	1.0±0.1
3H_Dil_4 Ch.1; No BS	99.0±2.4	1557.0±34.8	<b>6.4±0.2</b>	4.5±0.3
3H_Dil_4 Ch.2; No BS	100.9±2.2	1557.0±34.8	<b>6.5±0.2</b>	1.7±0.1
3H_Dil_4 Ch.1; BS	89.8±2.2	1557.0±34.8	<b>5.8±0.2</b>	3.7±0.2
3H_Dil_4 Ch.2; BS	75.7±1.7	1557.0±34.8	<b>4.9±0.2</b>	1.0±0.03
3H_Dil_5 Ch.1; No BS	154.8±4.2	2508.8±19.0	<b>6.2±0.2</b>	4.2±0.2
3H_Dil_5 Ch.2; No BS	155.7±4.1	2508.8±19.0	<b>6.2±0.2</b>	1.5±0.05
3H_Dil_5 Ch.1; BS	145.6±4.0	2508.8±19.0	<b>5.8±0.2</b>	3.7±0.2
3H_Dil_5 Ch.2; BS	130.5±3.4	2508.8±19.0	<b>5.3±0.2</b>	1.1±0.04

[cpm, counts per minute; dpm, disintegrations per minute]

### Discussion

The measurements have shown that the Wilma cell can be used to measure <sup>3</sup>H samples with levels of activity below 50 dpm/mL and the results are reproducible. Channel 1 shows a greater impact in terms of count subtraction for low-level samples; the effect is less evident above around 350 dpm/mL.

The counting efficiencies and hence figures of merit are rather low, being less than

10% in general for the former and less than 10 for the latter.

This may be due to a number of factors that need to be optimized such as the sample:cocktail ratio and also the width of the counting window. The latter needs to be investigated further in order to maximize the SNR [signal-to-noise ratio].

Dr. Tom Deakin, March 2012

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