



COMMONWEALTH OF  
PUERTO RICO  
ENVIRONMENTAL QUALITY BOARD

PROJECT NAME: QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR BEACH  
MONITORING AND PUBLIC NOTIFICATION PROGRAM  
ADDENDUM- SEPTEMBER 2015  
RESPONSIBLE AGENCY: PUERTO RICO ENVIRONMENTAL QUALITY BOARD (PREQB)

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P U E R T O R I C O  
ENVIRONMENTAL QUALITY BOARD

QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR  
BEACH MONITORING AND PUBLIC NOTIFICATION PROGRAM  
ADDENDUM - SEPTEMBER 2015

Prepared by:

Plans and Special Projects Division  
Water Quality Area



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## INTRODUCTION

This document and appendix serve to amend the Quality Assurance Project Plan (QAPP) for the Beach Monitoring and Public Notification Program (BMPNP), approved by United States Environmental Protection Agency (USEPA) on March, 2015. It is intended to be used with the original QAPP document. All procedures of the USEPA-approved QAPP for the sampling and analysis activities, as well as the ones related to the Quality Assurance and Quality Control data verification and quality assessment, are still applicable to the BMPNP. This addendum was prepared specifically to address the changes related to the public notification, which will be issued based on the Beach Action Value (BAV) for *Enterococcus* and the elimination of Playa Jauca-Santa Isabel from the BMPNP, due to safety issues.

## MODIFICATIONS

1. Starting on October 2015, the BMPNP will be implemented in 35 beaches of frequent use in Puerto Rico's Main Island (See Appendix 1). Playa Jauca in Santa Isabel was eliminated from the Program due to the fact that sampling activities in this beach could compromise the safety of the Water Sampling Officers. In every occasion that the QAPP document indicates 36 beaches, it should be replaced by 35 beaches.
2. In addition, beginning on October 2015, the public notification will be issued based on the exceedance of the Beach Action Value (BAV) for *Enterococcus* (70 colonies/mL). This means that, in every instance where the water quality standard is mentioned in the QAPP, it should be substituted by BAV.



## APPENDIX 1: BEACHES AND MONITORING STATIONS LOCATION

### Beaches and Monitoring Stations Location

Beach Name	Location	AU ID	Classification	Station ID and Location	Coordinates	
<b>Route 1: Dorado - Loíza</b>						
Balneario Manuel “Nolo” Morales or Sardinera	Road PR-693 Int. PR-697, Costas de Oro, Dorado	PRNC08	SB	RW-18	At the front of the administration building.	18°28'28.90" 66°16'51.21"
Balneario Punta Salinas	Road PR-165, Levittown, Toa Baja	PRNC09	SB	RW-19	At the front of the administration building.	18°28'17.97" 66°11'09.58"
Balneario El Escambrón	Muñoz Rivera Ave., Stop 8, Puerta de Tierra, San Juan	PREC12	SB	RW-20A	At the front of the showers and lifeguard stand.	18°28'02.05" 66°05'23.85"
Playa Sixto Escobar	Muñoz Rivera Ave., Stop 8, Puerta de Tierra, San Juan	PREC12	SB	RW-25A	At the center of the bathers area.	18°28'00.23" 66°05'12.00"
Playita del Condado	Ashford Ave., west to El Condado Plaza Hotel, San Juan	PRC13	SB	RW-26	At the center of the bathers area.	18°27'40.07" 66°04'56.67"
Ocean Park	General Patton Street, San Juan	PREC13	SB	RW-27	At the center of the bathers area.	18°27'10.84" 66°02'55.97"
Playa El Alambique	Isla Verde Ave., José M. Tartak Street, Carolina	PREC14	SB	RW-28	At the center of the bathers area.	18°26'38.73" 66°01'19.74"
Balneario de Carolina	Road PR-187, Boca de Cangrejos, Carolina	PREC14	SB	RW-21C	At the center of the bathers area.	18°26'45.56" 66°00'12.86"
Vacía Talega	Road PR-187, Loíza	PREC15	SB	RW-29	At the center of the bathers area.	18°26'52.29" 65°54'22.43"
<b>Route 2: Arroyo - Luquillo</b>						
Balneario Punta Guilarte	Road PR-3 Km 126, Arroyo	PRSC32	SB	RW-7	At the center of the bathers area	17°57'43.35" 66°02'24.00"
Balneario de Patillas	Road PR-3 Km 1.7, Los Bajos Ward, Patillas	PRSC32	SB	RW-6	At the center of the bathers area.	17°58'26.31" 65°59'20.33"
Playa Guayanés	El Ancla Beach Hotel, Yabucoa	PREC28C	SB	RW-30	At the center of the bathers area.	18°03'45.70" 65°49'09.10"
Balneario Punta Santiago	Road PR-3 Km 72.4, Humacao	PREC25	SB	RW-4	At the center of the bathers area.	18°09'30.29" 65°45'18.67"
Tropical Beach	Road PR-3 (confluence of Río Blanco and Río Santiago), Naguabo	PREC25	SB	RW-31	At the center of the bathers area.	18°11'12.94" 65°43'33.48"
Balneario Seven Seas	Road PR-195 Km 4.8 Las Croabas, Fajardo	PREC18	SB	RW-2	At the center of the bathers area	18°22'09.36" 65°38'09.86"



Beach Name	Location	AU ID	Classification	Station ID and Location		Coordinates	
Playa Azul	Luquillo Beach Boulevard (east of town), Luquillo	PREC18	SB	RW-32	At the center of the bathers area	18°22'54.72"	65°43'06.45"
Balneario La Monserrate	Road PR-3, Luquillo	PREC17	SB	RW-1A	In front of the administration building.	18°23'08.13"	65°43'46.1"
<b>Route 3: Lajas - Salinas</b>							
Playita Rosada	Camino de los Guayacanes, Lajas	PRSC41B2	SB	RW-33	In the pool.	17°58'18.18"	66°01'53.40"
Playa Santa	Road PR-325 Final, Providencia, Guánica	PRSC41B1	SB	RW-10	At the center of the bathers area, in front of AEELA building.	17°56'15.76"	66°57'18.71"
Caña Gorda	Road PR-333 Km 2.6 Caña Gorda Ward, Guánica	PRSC40	SB	RW-9	At the center of the bathers area.	17°57'09.11"	66°53'04.42"
Playa del Hilton	Ponce Hilton Hotel, Ponce	PRSC36B	SB	RW-34	In front of the hotels bathers area.	17°58'9.42"	66°36'9.82"
Balneario de Salinas	Road PR-1, Salinas	PRSC34	SB	RW-36	At the center of the bathers area.	17°58'39.32"	66°19'56.99"
<b>Route 4: Cabo Rojo</b>							
Playa El Combate	Road PR-3301 Final West Side, Cabo Rojo	PRSC43	SB	RW-12B	Near Los Salitrales	17°58'29.26"	67°12'46.46"
Playa Moja Casabe	Road PR-3301 Final, East Side, Cabo Rojo	PRWC43	SB	RW-14A	Alongside of where the office of the Department of Natural and Environmental Resources is located	17°58'57.49"	67°12'55.51"
Balneario de Boquerón	Road-101, Poblado Boquerón, Cabo Rojo	PRWC43	SB	RW-13	At the center of the bathers area.	18°01'09.99"	67°10'20.08"
Playa Buyé	Road PR-307 Km 3.8, Pederrales Ward, Cabo Rojo	PRWC44	SB	RW-8	At the center of the bathers area	18°02'55.94"	67°11'55.05"
Villa Lamela	Camino La Mela Final, Cabo Rojo	PRWC44	SB	RW-37	At the center of the bathers area.	18°03'52.32"	67°11'51.10"
<b>Route 5: Añasco - Aguadilla</b>							
Balneario de Añasco or Balneario Tres Hermanos	Road PR-115 Km 5, Hatillo Ward, Añasco	PRWC49	SB	RW-15	At the center of the bathers area.	18°17'16.79"	67°11'38.12"
Balneario de Rincón	Road PR-115 Int. Cabijas, Rincón	PRWC50	SB	RW-5	At the center of the bathers area.	18°20'27.33"	67°15'21.62"



Beach Name	Location	AU ID	Classification	Station ID and Location		Coordinates	
				Station ID	Location	Latitude	Longitude
Pico de Piedra	Road PR-115 Km 21, Aguada	PRWC51	SB	RW-22	At the center of the bathers area.	18°23'03.71"	67°12'46.76"
Balneario Crash Boat	Road PR-458 Final, Borinquen Ward, Aguadilla	PRWC52	SB	RW-16	At the center of the bathers area.	18°27'27.60"	67°09'49.60"
<b>Route: 6: Arcibo - Vega Alta</b>							
Muelle de Arcibo	Road PR-655, Arcibo	PRNC03	SB	RW-38	At the center of the bathers area.	18°28'45.33"	66°42'01.68"
Mar Chiquita	Road PR-648, Manatí	PRNC05	SB	RW-39	At the center of the bathers area.	18°28'22.50"	66°29'08.36"
Balneario de Puerto Nuevo	Road PR-692 Km 12, Vega Baja	PRNC06	SB	RW-23	At the center of the bathers area.	18°29'28.92"	66°23'56.56"
Balneario Cerro Gordo or Javier Calderón Nieves	Road PR-690, Cerro Gordo Ward, Vega Alta	PRNC07	SB	RW-17	At the center of the bathers area.	18°28'52.50"	66°20'26.36"
<p><b>AU</b> = Assessment Unit; <b>SB</b> = Coastal and estuarine waters designated for primary and secondary contact recreation, and propagation and preservation of desirable species including threatened or endangered species, as defined in the PRWQSR; <b>RW</b> = Recreational Waters</p>							



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Prepared by:

Plans and Special Projects Division  
Water Quality Area



TITLE AND APPROVAL PAGE

PROJECT NAME: QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR BEACH MONITORING AND PUBLIC NOTIFICATION PROGRAM

RESPONSIBLE AGENCY: PUERTO RICO ENVIRONMENTAL QUALITY BOARD (PREQB)

PREQB OVERALL PROJECT MANAGER:

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PROJECT QA OFFICER

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NAME: FRANCES M. SEGARRA ROMAN  
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USEPA PROJECT MANAGER

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## PROJECT NAME

Beach Monitoring and Public Notification Program (BMPNP)

## PROJECT REQUEST BY

Puerto Rico Environmental Quality Board (PREQB)

## DATE OF PROJECT INITIATION

November 2014

## OVERALL PROJECT MANAGER

Wanda García Hernández (PREQB)

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Frances M. Segarra Román (PREQB)

## DISTRIBUTION LIST

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- Wanda García Hernández, Acting Manager, Overall Project Manager
- Ángel Meléndez Aguilar, Chief of Plans and Special Projects Division (PSPD)
- Evelyn Figueroa Del Valle, Environmental Quality Specialist I, Project Officer

### PREQB Water Sampling Division (WSD)

- Vacant position, Chief of WSD. Nevertheless, the functions of this position are carried out by Mrs. Wanda García, WQA Manager.

### PREQB Environmental Research Laboratory of Puerto Rico (ERLPR)

- Janette Cambrelén, Manager, PREQB ERLPR

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PREQB Quality Assurance and Quality Control (QA/QC) Office

- Frances M. Segarra Román, QA/QC Office Acting Chief and Specialist Manager, QA Officer

United States Environmental Protection Agency (USEPA)

- Helen Grebe, USEPA Project Manager
- Donna Ringel, USEPA - Region II, QA Officer

## PROJECT ORGANIZATION AND RESPONSIBILITY

Below is a description of the key project personnel and their corresponding responsibilities. An organization chart is included in Figure 1 of Appendix 1.



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Date

Mrs. Wanda García Hernández is the Acting Manager of the WQA and the Overall Project Manager. She will be in charge of assessing compliance with the project objectives, and USEPA's administrative requirements. Also, she is in charge of coordinating field sampling activities with the WSD and sample transference to PREQB ERLPR.



Ángel Meléndez Aguilar  
 Chief of PSPD  
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Date

Mr. Ángel Meléndez Aguilar is the Chief of the PSPD. He will be the liaison between the Overall Project Manager and the Project Officer. Also, he will be in charge of supervising technical personnel, coordinating meetings, and ensuring compliance with BMPNP work plan.

*Evelyn Figueroa Del Valle*

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*March 30, 2015*

Date

Mrs. Evelyn Figueroa Del Valle is the Project Officer. She will be in charge of ensuring good management of the BMPNP data and information, including the database construction. Also, she is responsible of data evaluation with respect to compliance with the water quality standard and the storage in Excel Spreadsheets and Access database, as well as USEPA Water Quality Exchange (WQX).

*Frances M. Segarra Román*

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*March 30, 2015*

Date

Frances M. Segarra Román is the Quality Assurance and Quality Control (QA/QC) Office Acting Chief and QA Officer (QAO) for this Project. The QA Officer will be responsible for approving, as well as overseeing the implementation of the Quality Assurance and Quality Control (QA/QC) protocols and procedures established in this QAPP, as well as WSD and PREQB ERLPR Standard Operating Procedures (SOPs).

*Janette Cambrelén*

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*March 30, 2015*

Date

Mrs. Janette Cambrelén is the PREQB ERLPR Manager. She will be in charge of coordinating the water sample analyses and drafting of the reports, including the analytical results.

## PROJECT DESCRIPTION

### Project and Task Description

This project is implemented to achieve the goals of the Beaches Environmental Assessment and Coastal Health Act (Beach Act), as of reducing the risk of recreational beach users to the exposure to pathogens and diseases. PREQB BMPNP is implemented to collect bacteriological data along thirty six (36) beaches of frequent use in Puerto Rico's Main Island. The results are used to know the water quality by determining compliance with the Puerto Rico Water Quality Standards Regulation (PRWQSR); thereby, protecting the human health by achieving the primary contact recreation use (swimming).

The sampling, analyses, and assessment is focused on the Puerto Rico water quality indicator for direct contact, namely: *Enterococcus*. The water quality standard for this bacteriological parameter is: 35 colonies /100 mL. In addition, the monitoring network helps detect the sources of pollution that threaten or may impact the water quality of the beaches. Some of these sources of pollution identified are: urban runoff, sanitary sewer overflows, septic tanks overflows, and Confined Animal Feeding Operations (CAFOs).

Pathogen contamination in recreational waters represents for users a high level risk of contracting many diseases, such as: infections (respiratory, ear, eye, and skin), gastroenteritis, as well as meningitis and hepatitis (EPA-823-F-01-022, July 2001). For this reason, the immediate notification to the public of the exceedance of any water quality indicator is a mandatory element of this project. Figure 2 of Appendix 1 has a flow chart that represents the procedure implemented to accomplish the rapid public notification of water quality.

When the results of a routine sampling exceed the water quality standard for *Enterococcus*, PREQB notifies the beach administrators and USEPA via an electronic notification (e-mail). The general public are notified through PREQB's web page and a Public Release Notification that the beach is not suitable for primary contact recreation. Beach administrators are responsible for notifying the public when the water quality standard is exceeded. This notification could be done by updating the sign on the beach and hoisting the Yellow Flag when the water quality standard is exceeded.

Notification is still in effect until resampling is carried out and PSPD personnel compare analytical results to the water quality standard, to determine that it is no longer exceeded and the beach is suitable for primary contact recreation. When the water quality is no longer exceeded, PREQB notifies the beach administrators, USEPA and the general public that the beach is suitable for primary contact recreation.

This QAPP governs all the field and sampling activities that are performed for the BMPNP according to the following tasks:

### 1. Field Data Collection

WSD will perform the field sampling activities in order to obtain *in-situ* measurement of physical parameters (temperature and pH), and collect water samples for microbiological analysis across 36 beaches of frequent use in Puerto Rico. Sampling will be performed accordingly to WSD Standard Operating Procedures Surface Water Sampling (SOP-100). The field data will be obtained from the 36 stations previously identified, in a frequency of every two weeks throughout the year. As part of the Quality System Implementation of the WQA Quality Management Plan (QMP), the QA Officers from the QA/QC Office may perform on-site field technical audits of the field data collection processes. These are performed during the oversight of field sampling activities to determine whether all field sampling activities are conducted in compliance with the QA/QC established in this QAPP.

### 2. Sample and Data Analysis

PREQB ERLPR will analyze the microbiological parameter using the corresponding Defined Substrate Technology (Enterolert). In addition, they will provide the entire QA/QC documentation associated with the analysis. All physical parameters that must be evaluated immediately before sample collection will be done in the field by the technical personnel of the WSD. Data will be used to determine compliance with the water quality; thereby, protecting the human health by achieving the primary contact recreation use (swimming).

### 3. Data Verification and Quality Assessment

Sampling results along with all backup QA/QC documentation will be reviewed and evaluated by the QAO. The QAO will verify and assess the quality of the data in order to determine compliance with this QAPP. If deficiencies are

detected during evaluation, these will be reported in written to the WQA Manager so the appropriate corrective action, if necessary can be implemented.

#### 4. Data Usage

The data from the BMPNP will be used to verify the quality of the waters of 36 beaches along the coast of Puerto Rico, thereby protecting the human health by achieving the primary contact recreation use.

The data will be evaluated in terms of precision, accuracy, representativeness, comparability, completeness (PARCC), and sensitivity in order to support environmental decisions. In general, at the field and laboratory level all procedures must be followed as established by this QAPP, ensuring the quality of the field and analytical data obtained. This will include the data verification and data quality assessment by the QAO.

All data verified by the QAO and determined to have the quality required to be acceptable, precise, accurate, and scientifically valid for their intended purpose shall be available to the public to keep them informed on the water quality of the beaches through a public release notification (See Appendix 2), thus protecting the human health. In addition, it should be available for the use of the PSPD. This data will be used to supplement the information in the Storage and Retrieval (STORET) database. All field and bacteriological results will be stored in PREQB database (Excel and Access) and will be provided to USEPA-CDX program through a XLM format. These data will be useful to perform short and long term trends analyses of water quality indicator (*Enterococcus*) in beaches. Figure 3 of Appendix 1 illustrates the process of submitting monitoring and notification data to USEPA database.

## Monitoring Network Design and Rationale

BMPNP network will focus on continuous sampling activities, to provide essential beach assessment data. This project will only involves aqueous environmental samples. The network consists of monitoring stations located at 36 public beaches along the coast of Puerto Rico's Main Island (See Appendix 3).

To select the 36 beaches the following activities were carried out:

1. Evaluation of the Department of Natural and Environmental Resources bathing zones inventory (2007)<sup>1</sup>, which consists of coastal areas of Puerto Rico suitable for bathing and passive recreation that are classified as public beaches by the *Reglamento Número 4*<sup>2</sup> of the Puerto Rico Planning Board;
2. Inspection of the beaches to determine if they are suitable for recreation activities;
3. Selection of the beaches based on frequency of use by local bathers and tourists, number of users, public sanitation facilities, and location of pollution sources (point and non-point), as well as its accessibility and appropriateness for bathing activities.

In most of the beaches, the stations were located in the swimming designated area, according to the *Reglamento Número 4*. All the monitoring stations have been located strategically where bathers concentrate the most. The stations and locations are representative of the overall water quality of the beach.

At each monitoring station all parameters required for this project will be collected. Samples will be obtained directly from the surface of the water body by the WSD, according to the Standard Operating Procedure for Surface Water Sampling (SOP-100). PREQB ERLPR will perform the analysis of the water samples collected using Defined Substrate Technology (Enterolert), accordingly to Standard Operating Procedure Defined Substrate Technology (Enterolert), SOP 021.

## Monitoring Parameters and Frequency of Collection

The parameters to be monitored at each site are *Enterococcus*, pH, and temperature. The frequency of collection will be every two weeks, throughout the year, since in Puerto Rico, the season variability through the whole year is not significant and local bathers and tourists visit the beaches frequently. Samples will be collected Monday and Tuesday in the morning. However, in weeks where Monday is a holiday, samples will be collected on Tuesday and Wednesday.

When the result of a routine sample exceeds the water quality standard, a re-sampling is carried out one week from the day of exceedance (Monday or Tuesday). It should be pointed out that with the result of the routine sampling, PREQB notifies the beach administrators and USEPA via an electronic notification (e-mail). The

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<sup>1</sup> *Inventario de áreas para bañistas* (Marzo 2007)

<sup>2</sup> *Reglamento de Zonificación de Puerto Rico del 16 de septiembre de 1992*, as amended as *Reglamento de Calificación de Puerto Rico del 11 de enero de 2009*.

general public are notified through PREQB’s web page and a Public Release Notification that the beach is not suitable for primary contact recreation. Beach administrators are responsible for notifying the public when the water quality standard is exceeded. This notification could be done by updating the sign on the beach and hoisting the Yellow Flag when the water quality standard is exceeded.

If result of the re-sampling also exceeds the water quality standard, PREQB notifies the general public, beach administrators, and EPA as described in the paragraph above. Notification is still in effect until a new sampling is carried out and PSPD personnel compare analytical results to the water quality standard, to determine that it is no longer exceeded and the beach is suitable for primary contact recreation. When the water quality is no longer exceeded, PREQB notifies the beach administrators, USEPA and the general public that the beach is suitable for primary contact recreation.

A sample will be collected in all stations as described in Appendix 3. Table 1 describes the analytical methods, parameters, and other relevant information, to be done for each water sample.

Table 1: Parameters and Analytical Methods

Parameter	Sample Matrix	No. Samples*	Analytical Method	Container	Sample Preservative <sup>1</sup>	Holding Time
Physical	Measured at the field					
Temperature, °C (Field)	Sea Water	1,092	PREQB SOP No. 110	<i>In-situ.</i> If sample has to be collected, a 250 mL or more plastic or glass container will be used.	N/A	Analyze immediately in the field <sup>3</sup> .
pH (Field)	Sea Water	1,092	PREQB SOP No. 110	<i>In-situ.</i> If sample has to be collected, a 250 mL or more plastic or glass container will be used.	N/A	Analyze immediately in the field <sup>3</sup> .

Parameter	Sample Matrix	No. Samples*	Analytical Method	Container	Sample Preservative <sup>1</sup>	Holding Time
Microbiological	Measured at the laboratory					
<i>Enterococcus</i>	Sea Water	936	DST - Enterolert PREQB ERLPR SOP-021	500 mL PP bottle /sterile	Cool to ≤ 6°C	Must arrived at the laboratory within 6 hours of collection; Must be processed within 2 hours of arriving at the laboratory <sup>2</sup>

**Notes:**

PREQB SOP = Refers to the method number established in the Puerto Rico Environmental Quality Board Standard Operations Procedures.

DST = Defined Substrate Technology

PP = Polypropylene

\* Samples number calculation for physical parameters was computed using the following equation: (# beaches x # samples per sampling event x # weeks) + (# replicates per route x # weeks).

$$(36 \text{ beaches} \times 1 \text{ sample per sampling event} \times 26 \text{ weeks}) + ((1 \text{ replicates} \times 6 \text{ routes}) \times 26 \text{ weeks}) = 936 + 156 = 1,092 \text{ samples}$$

For microbiological parameter, sample number calculation was computed using the following equation: (# beaches x # samples per sampling event x # weeks), since duplicates are not collected.

$$(36 \text{ beaches} \times 1 \text{ sample per sampling event} \times 26 \text{ weeks}) = 936$$

1. Sample preservation should be performed immediately upon sample collection.
2. Sample should be analyzed within 2 hours after receiving at the laboratory, as established in the Standard Method. The times listed are the maximum that samples may be held before analysis and still be considered valid.
3. The term “analyzed immediately” usually means within 15 minutes or less of sample collection.

## SCHEDULE OF TASKS AND PRODUCTS

Samples will be collected every two weeks, throughout the whole year, starting on November 2014. The scheduled of the tasks to be performed are described in Table 2.

Table 2: Project Schedule

Tasks	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT
1. Sample collection	√	√	√	√	√	√	√	√	√	√	√	√	√
2. All laboratory analyses completed and submitted to the Project Officer	√	√	√	√	√	√	√	√	√	√	√	√	√
3. Data Verification and Quality Assessment Report	√	√	√	√	√	√	√	√	√	√	√	√	√
4. Data entry into Access Database	√	√	√	√	√	√	√	√	√	√	√	√	√
5. Final QA Report													√
6. Data entry into STORET through CDX				√					√				√

## DATA QUALITY REQUIREMENTS AND ASSESSMENTS

Establishing Data Quality Objectives (DQO's) is the most important part of any data collection activity, because it provides clear objectives and good planning. As a result of this process, PREQB will generate analytical results that will be used by the PREQB WQA to determine if the results are in agreement with the action levels established at the Puerto Rico Water Quality Standards Regulation PRWQSR (August, 2014); determine compliance with SB Class standards regarding *Enterococcus* (35 colonies/100 mL), pH (7.3-8.5 standard pH units) and temperature (Not to exceed 32.2 °C). The finality of this process is to protect the human health by achieving the primary contact recreation use; if not, release an immediate notification to public about exceedance of any quality indicator. In addition, this process will supplement USEPA Database (STORET).

### Representativeness

Representativeness expresses the degree to which accurate and precise data represents the characteristics of a population, the parameter variations at a sampling point, a process condition, or an environmental condition. The representativeness of the data will be assessed in three areas as follows:

1. the number of locations, matrices, and samples sufficient to accurately depict site conditions;

2. the sampling procedures must be designed so that individual samples accurately represent the condition of the matrix from which they were collected; and,
3. the appropriateness of the measurement method used to the type of sample obtained.

The monitoring approach and design for this project takes into consideration: the frequency of use by local bathers and tourists; number of users; public sanitation facilities; location of pollution sources (point and non-point); and, the minimal season variations regarding the environmental conditions that occur in Puerto Rico. Due to the relative small size of the beachfronts, one sampling point at each bathing zone fulfills the representativeness criteria.

## Completeness

Completeness is a measure of the amount of valid data obtained from any measurement system compared with the amount of data that was expected to be obtained under correct and normal conditions. Completeness refers to the percentage of samples, which are expected to be analyzed to achieve the completeness goal. For this project completeness must be 90%.

To determine the completeness of the project, the following formula will be used:

$$\%C = \frac{V}{T} \times 100$$

where V = no. of planned measurements judged valid;  
and, T = total no. of measurements.

## Comparability

Comparability is a measure of confidence with which one data set can be compared to another. In this project, it is ensured by using approved scientific methods and close quality control monitoring; adherence to preservation procedures and compliance with maximum holding times, and the use of consistent units for data reporting.

## Accuracy

Accuracy is the measure of confidence in a measurement. Quality control should be conducted on each new lot of Enterolert. To assure the quality of laboratory analysis for each lot of Enterolert, three sterile vessels filled with 100ml sterile water will be inoculated with *Enterococcus faecium* ATCC 35667 (fluorescence), *Serratia marcescens* (gram negative) ATCC 43862 (no fluorescence) and *Aerococcus viridans* (gram positive) ATCC 10400 (no fluorescence). These controls will be prepared freshly cultivating pure cultures received from manufacturers and preparing dilutions for the test. As another control one blank (sterile) water sample will be analyzed per route of samples received. Finally, at least one external reference sample must be run per year.

## Precision

Precision is the degree of agreement among repeated measurements. It provides information of uncertainty due to natural variation, sampling error, and analytical error. Precision will be performed via duplicate counts, which will be carried out once for every sampling route and will be documented on a notebook. Duplicate counts should be within 5% (same analyst) or 10% (multiple analysts). If these counts are off more than this amount, an investigation and appropriate corrective action should be under taken.

## Sensitivity

Sensitivity is the ability of the method or instrument to detect the target analytes at the level of interest. The detection limits and acceptance criteria in Table 3 (*Method and Instrument Detection Limit*) must be achieved by the WSD and PREQB ERLPR:

Table 3: Method and Instrument Detection Limit

Parameter	Detection Limit	Accuracy Goal	Precision Goal
Physical			
Temperature, °C	Measurement Limit 0-80 °C	± 0.3°C	± 0.3°C
pH	Measurement Limit 0-14	0.1 pH unit	0.1 pH unit
Microbiological			
<i>Enterococcus</i>	1 colony/100 mL	N/A	Duplicate counts ± 5% (same analyst) or 10% (multiple analysts)
N/A = Not Applicable			

## SAMPLING PROCEDURES

The primary purpose of this sampling program is to determine the quality of the water bodies, to protect the human health by achieving the primary contact recreation use. In accordance with this purpose, PREQB identified 36 beaches that will be sampled using a judgmental sampling design. PREQB pre-selected the number of sampling stations, which were documented using GPS coordinates as established in Appendix 3. Basically, to establish the number of sampling stations, the following criteria were considered: frequency of use by local bathers and tourists, number of users, public sanitation facilities, and location of pollution sources (point and non-point). On the water body, the samples will be collected against the current (whenever possible). The sampling technicians must collect the samples at knee high, as stated in SOP-100. Nevertheless, in those beaches of high-energy environments, samples will be collected at ankle depth because it is safer and easier for the sampler, as established in the National Beach Guidance and Required Performance Criteria for Grants, 2014 Ed. If this is the case, it has to be documented at the Field Data Record and Chain of Custody.

One grab sample will be obtained per sampling station by the WSD. During the sampling process, WSD personnel must follow all of the quality requirements established in Table 1 of this Plan and SOP-100 (See Appendix 4). Prior to collecting any sample, the sampling personnel at each station must fill the Field Inspection Form (See Figure 4 of Appendix 1), which documents general information at each station such as: date, time, station identification, weather conditions, and sources of contamination, among others. Then, personnel will proceed with sample collection. This involves the measurement of the field physical parameters first, followed by the collection of the sample fraction to be analyzed for the microbiological parameter by the PREQB ERLPR. All the field physical data at each monitoring station must be documented in the Field Data Record Form (See Figure 5 of Appendix 1). For each field route and sampling day, a replicate of the physical parameters measurements must be taken every 5 readings for quality control purposes. In the case that the route consists of less than 5 stations, the reading of a station must be taken as a replicate.

After taking the physical parameter measurements, surface water samples will be collected in a 500 mL PP bottle directly from the water body, at knee high; except in high energy environments where they will be collected at ankle depth. All the samples collected during the field activities will be documented in the Chain of Custody form (COC) and the sample container label in the collection order to

uniquely identify each sample (station number) and to keep track of the custody of the samples from the time of their collection to the time of their delivery to the laboratory. For the COC and samples label to be used for this purposes refer to Figures 6 and 7 of Appendix 1, respectively.

## ANALYTICAL METHOD

Samples will be analyzed using Defined Substrate Technology (Enterolert). When samples arrived at the PREQB ERLPR, the laboratory personnel must verify that the amount and type of samples documented in the COC correspond to the samples inside the sample coolers, and that the temperature inside the coolers is within the target temperature of 6 °C or less. He/she must also ensure that the samples are immediately refrigerate or kept at the target temperature of 6 °C or less. Samples must arrive at the laboratory within 6 hours of sample collection and must be analyzed within two hours of receiving the sample at the laboratory.

Samples must be analyzed following the procedure describe in the Defined Substrate Technology-Enterolert method and PREQB ERLPR SOP 021 (See Appendix 6). In addition, quality control checks must be carried out accordingly to analytical method and PREQB ERLPR SOP 021. The personnel performing the bacteriological analysis with Enterolert is an Environmental Microbiologist with more than 2 years of experience.

## SAMPLE HANDLING AND CUSTODY PROCEDURES

The COC is required to be completed during all the field sampling activities and serve as physical evidence of sample collection and custody. This document, which serves as a permanent record, must be completely filled with the following information: sampling site or location; sampler's name and signature; analytical test required; sample number, matrix and preservative; date and time of collection; type and amount of sample containers; amount and type of sample (i. e. grab sample); and signatures of the personnel responsible for the samples custody, at each sampling step.

To ensure that valid analysis results are obtained, proper handling of the samples must be followed after collection. Careful and complete documentation at all stages of sample collection process, including handling, storage, transportation, and

analysis, must be performed. Table 1 summarizes the Parameters and Analytical Methods for field measurements and sample collection at each project site.

Water samples will be collected by PREQB WSD personnel and remain under their custody, or other PREQB personnel, until their transfer to an authorized PREQB Environmental Laboratory personnel. The sample coolers must contain sufficient ice to cool and maintain all the samples within  $\leq 6$  °C, which is verified by the laboratory personnel at the laboratory by measuring the temperature of the Shipping Container Temperature Blank.

Sample delivery to the laboratory will be coordinated so that sample holding times are strictly fulfilled. The sample coolers will be delivered directly to the PREQB ERL, no later than six (6) hours after sample collection. Adequate shipping containers will be used to protect the sample, avoid breakage, and maintain proper temperatures during shipping. Containers will be leak-proof, to avoid cross-contamination and sample loss during transportation.

Before the samples are accepted, the laboratory personnel will confirm that each sample arrived in good condition. In addition, they will request a copy of the completed COC form and will verify it to ensure that the samples are managed and stored according to protocol.

Samples bottles will be labeled prior the time of the sample collection. All pertinent sample information will be recorded on the label including: sampling site, date, time, grab or composite, preservative, test requested, and sampler's name. Field forms are used to record the field data like field measurements that are taken by the sampling personnel during the collection activities. The calibrations are recorded in the calibration forms, as established in the respective SOPs (See Appendix 4).

All entries will be made in permanent and indelible ink. If errors are made when writing in any of the field forms, they will be crossed out with a single line, dated and signed with the initials of the person that making the correction.

## CALIBRATION PROCEDURES AND PREVENTIVE MAINTENANCE

Prior to start the survey, PREQB WSD and ERLPR personnel will carefully examine and test all sampling and analysis equipment required for the project. Any necessary

calibration, maintenance, repairs, or adjustments are made to ensure that all equipment are working properly.

## PREQB ERLPR Instruments

The instruments will be calibrated and preventive maintenance will be performed in accordance with PREQB ERLPR SOPs and the manufacturer's instructions, as summarized below.

Parameter	<i>Enterococcus</i>	<i>Enterococcus</i>	<i>Enterococcus</i>	<i>Enterococcus</i>	<i>Enterococcus</i>
Equipment	Quanti-tray sealer	UV Lamp	Incubator	Incubator thermometer	Autoclave
Maintenance activity	External cleaning.	Lamp replacement, when it damages.	External cleaning; weekly.	N/A	Annually
Testing activity	Dye test monthly	N/A	N/A	N/A	Check weekly the autoclave performance once starting operation.
Inspection activity	N/A	N/A	Temperature reading, two times a day.	N/A	Check the autoclave weekly.
Calibration activity	N/A	N/A	N/A	2 times a year; every 6 months	Check the timing device with a stop watch quarterly
Acceptance criteria	No color observed outside the wells after dye test performance.	N/A	41.0 ± 0.5 °C	The corrector factor of the incubator must be < 1°C; if greater replace the thermometer. The thermometers must be labeled with correction factors and that correction factors should be applied and record in the logbook.	N/A

Parameter	<i>Enterococcus</i>	<i>Enterococcus</i>	<i>Enterococcus</i>	<i>Enterococcus</i>	<i>Enterococcus</i>
Method	ERLPR	ERLPR	ERLPR	ERLPR	ERLPR
Reference or ERLPR SOP	SOP-023	SOP-023	SOP-022	SOP-022	SOP-022

## PREQB WSD Instruments

WSD will use HACH HQ40d or HQ30d pH meters to measure pH and temperature parameters. The instruments will be calibrated and preventive maintenance will be performed in accordance with PREQB WSD SOP for the Determination of the Potential of Hydrogen (pH) and Temperature, SOP-110 (See Appendix 4) and the manufacturer’s instructions, as summarized below.

Parameter	pH and Temperature
Equipment	Hach HQ30d or Hach HQ40d
Maintenance activity	As needed or required by the manufacturer.
Testing activity	Prior to conduct the calibration.
Inspection activity	Prior to conduct the calibration
Calibration activity	Calibration is checked before and after using the equipment
Frequency	Every day that the equipment is going to be used.
Acceptance criteria	±0.10
PREQB SOP	PREQB SOP 110

Calibration will be carried out using buffer solutions with calibration points of 4.00, 7.00, and 10.00. Calibration readings (including buffer used, lot number, expiration date, temperature and difference of instant reading) and calibration slope information must be recorded in the pH Meter Calibration Verification form included as part of SOP-110 in Appendix 4.

In case that the equipment fails to calibrate, two more calibrations will be performed to corroborate procedures and account for extraordinary situations. If after three consecutive calibrations the equipment fails to calibrate, it will be taken out for repairs. Under no circumstances an instrument that fails to calibrate will be used in the field. All equipment and instruments will be stored following the manufacturer recommendations.

It should be pointed out that, a final check of the calibration has to be carried out using one buffer (4.00, 7.00 or 10.00 calibration point) after completing the sampling route, to validate the performance of the instrument. This data must be documented in the pH Meter Calibration Verification form.

## INSPECTION AND ACCEPTANCE OF SUPPLIES AND CONSUMABLES

### PREQB ERLPR

The following list has PREQB ERLPR supplies that will use to carry out the Enterolert analysis.

1. Enterolert dry media snap packs
2. Quality control bacteria strains
3. 100 mL sterile bottles
4. 250 mL sterile bottles for quality control samples

When the supplies are received at PREQB ERLPR, technicians first inspect the box where they arrived, to guarantee that it is closed, sealed and labeled with the shipping information. Then, they inspect that all materials are in good conditions, not broken, sealed and with shrink-banded or tear-off labels. In addition, they verify that the materials have not exceeded their expiration date. All supplies that do not comply with acceptance criteria will not be used and will be discarded.

### PREQB WSD

The only supplies that the WSD uses are pH buffers and storage solutions. Once they are received at the WSD, technicians first inspect the box where they arrived, to guarantee that it is closed, sealed and labeled with the shipping information. Then, they inspect that all bottles are in good conditions, caps are not broken, and the pH buffers and storage solutions have not exceeded their expiration date. All bottles of solutions that do not comply with acceptance criteria will not be use and will be discarded.

# DOCUMENTATION, DATA REDUCTION AND REPORTING

## Documentation

Throughout the duration of the project, various forms will be filled out and maintained so that documentation will be available to assist in the evaluation of the information collected, assuring its reliability and accuracy. The project file will contain the field observations form and survey reports, as well as quality assurance forms that are required for documenting, including equipment performance and activities of the WQD and laboratory personnel. Field and analytical site related documents include the following:

- field forms
- chain of custody form
- sample analytical test results
- QA/QC data
- calibration forms
- sample management documentation
- reference information (if any)

## Data Reduction and Reporting

During each analytical procedure control samples are always analyzed. The report prepared by PREQB ERLPR analysts will contain the results of the samples and control samples. After the validation of the data, it will be entered into excel spreadsheets and Access database, as well as USEPA STORET database. The data will be available to USEPA Project Officer and USEPA QA Officer by e-mail, by mail or any other available alternative, accordingly to BMPNP Work Plan.

## Data Review, Verification, Validation, and Corrective Actions

All data generated at the field and laboratory level has to be reviewed and verified to assess its quality and determine if it is acceptable, precise, accurate, and scientifically valid to be useful for their intended purpose. This process is responsibility of field technicians, laboratory personnel, and QAO. The following table summarizes the process and personnel involve in it.

Table 4: Review, Verification and Validation Process

Review/Verification/Validation Input	Descriptions	Internal/External	Responsible for Verification/Validation
Chain of Custody	Form will be internally reviewed upon completion and verified against field logs. Provides sampling dates and time, identification of the field sampler, verification of sample ID, analytical methods requested, QC sample information, and all sample transference information.	Internal/External	WSD, PREQB ERLPR, PREQB QAO, PREQB
Field Forms	Field forms will be reviewed for accuracy and completeness and place in project file. The field forms detailing site activities and observations so that an accurate account of field procedures can be reconstructed. Some of these forms are Field Calibration Forms, Field Inspection Form and Field Data Record.	Internal	WSD, PREQB QAO, PREQB
Sampling Procedures	Evaluate whether sampling procedures were followed with respect to equipment and proper sampling support using audit and sampling reports.	Internal	QAO, PREQB
Laboratory Data	All laboratory data will be verified by the laboratory performing the analysis for completeness and technical accuracy. They must provide a data package <sup>1</sup> . Subsequently, the data will be validated for completeness, compliance, and usability in order to ensure that all analytical procedures were followed.	Internal External	QAO, PREQB ERLPR, PREQB
QAPP	All planning documents will be available to reviewers to allow reconciliation with planned activities and objectives.	Internal	QAO, PREQB
Audit Reports	Reports used to validate compliance of field sampling, handling and analysis activities with the QAPP.	Internal	QAO, PREQB
Data Narrative	Must be generated by PREQB ERLPR. The narrative specifies samples received, analytical methods employed, all deviations from methods and their impact.	Internal External	ERLPR, PREQB QAO, PREQB
Detection Limits or Quantitation Limits	Achieved as outlined in the QAPP.	Internal External	ERLPR, PREQB QAO, PREQB

Review/Verification/ Validation Input	Descriptions	Internal/ External	Responsible for Verification/Validation
Methods	Records support implementation of the SOP-sampling and analysis.	Internal	WSD, PREQB ERLPR, PREQB QAO, PREQB
<p>Note:</p> <p><sup>1</sup> This includes: Chain of Custody, Analytical Results (samples and controls), and Precision.</p>			

Review

Water sampling technicians has to review field data to ensure that: monitoring was performed in accordance with this QAPP and WSD SOPs; instruments calibration verification was performed in accordance with WSD SOPs; sample was properly preserved; field QC replicates were measured; and chain of custody was fill out properly. In addition, they must record any deviation from the QAPP and/or SOPs in the field documents.

On the other hand, PREQB ERLPR personnel must review chain of custody and laboratory data to ensure that: samples were stored properly; holding time limits were met; there is no errors regarding calculations, as well as with respect to data entry and transcription; laboratory QC samples were analyzed in accordance with this QAPP. They must record any deviation from the QAPP and/or SOPs.

Verification

WSD personnel must evaluate that data complies with SOPs specifications and that the reporting units are use appropriately. On the other hand, PREQB ERLPR must evaluate analytical data accordingly to the specifications of the method. This must include: checking dilution factors and appropriate reporting units, among others. Any deviation from specifications must be documented. It is presumed that the signature of supervisor(s)/Laboratory Manager in the analytical results report certify that the verification process of data was conducted and complies with method specification.

Quality Assessment and Verification

The QAO will review and assess the quality of the data to determine that it is acceptable, precise, accurate, scientifically valid to be useful for their intended purpose, and whether or not data met quality objectives established for the project

in this QAPP. The QAO will use the results of this review and verification process to identify data for which Quality Control requirements were not satisfied (See Table 5). He/she will report in written on the results of this process and certify that the data was generated in compliance with QA/QC requirements established in this document. In addition, the QAO will discuss how limitations on the data will be reported to the data users.

In addition, the QAO will evaluate the field data along with the analytical data using the procedures established in the SOP-132, *Field Sampling and Laboratory Data Validation* (See Appendix 5) and will generate a final report. The final report will identify raw data of questionable and unacceptable quality, as well as data that may have to be eliminated during validation. The documentation that will be reviewed are:

- Field
  - o Chain of Custody, calibration forms, Field Inspection Form, Field Data Record, audit reports (if any), and any other document generated from field sampling procedures
- Laboratory
  - o Analytical results and precision and accuracy data of the analysis. In addition, any narrative summarizing any discrepancy or deviation, including information on any corrective actions implemented, generated by the Laboratory.

Table 5: Quality Controls

DQI	Field QC Check	Frequency of Collection	Acceptance Criteria	Corrective Action(s)
Field: pH Measurement				
Precision	Replicate	For every field route and sampling day, a replicate will be taken for every 5 readings. In the case that the route consist of less than 5 stations, the reading of a station will be taken as a replicate.	Difference between two readings must be $\pm 0.10$ .	<ul style="list-style-type: none"> <li>&gt; Verbal briefing.</li> <li>&gt; Correct the practice immediately.</li> <li>&gt; Corrective Action Notice, if severe violations noted.</li> </ul>
Accuracy	Calibration Verification	Before taking the <i>in-situ</i> measurements	$\pm 0.10$ for every buffer used; Internal calibration slope: $-59 \text{ mV/pH}$ , 85-115% at 25°C	<ul style="list-style-type: none"> <li>&gt; Memorandum or Corrective Action Notice</li> </ul>
	Final Calibration verification	After finishing sampling activities	$\pm 0.10$	<ul style="list-style-type: none"> <li>&gt; Memorandum or Corrective Action Notice</li> </ul>
	Shipping Container Temperature Blank	One per cooler, per day of sampling.	Less or equal to 6°C	<ul style="list-style-type: none"> <li>&gt; Memorandum or Corrective Action Notice</li> </ul>
Representativeness	Data Quality Assessment and Verification	Every time the quality of the field data is assessed and verified.	Data collection adhered to field procedures	<ul style="list-style-type: none"> <li>&gt; Flag questionable data</li> <li>&gt; Memorandum or Corrective Action Notice</li> </ul>
Comparability	Data Quality Assessment and Verification	Every time the quality of the field data is assessed and verified.	Procedures at the field comply with requirements established in this QAPP (calibration, holding time, reporting units)	<ul style="list-style-type: none"> <li>&gt; Flag questionable data</li> <li>&gt; Memorandum or Corrective Action Notice</li> </ul>
Completeness	Data Quality Assessment and Verification	Depends on the number of <i>in-situ</i> measurements performed.	Greater or equal to 90%.	<ul style="list-style-type: none"> <li>&gt; Memorandum or Corrective Action Notice</li> </ul>

DQI	Field QC Check	Frequency of Collection	Acceptance Criteria	Corrective Action(s)
Sensitivity	Data Quality Assessment and Verification	Every time the quality of the field data is assessed and verified.	Dimensions being measured fit inside Table 3 ranges	> Memorandum or Corrective Action Notice
Laboratory: Defined Substrate Technology - Enterolert				
Precision	Duplicate counts	Once per sampling route	Duplicate counts $\pm$ 5% same analyst 10% multiple analysts	> Perform a third party count. > If third party exceeds acceptance criteria, identify and resolve the analytical problem before making further analyses.
Accuracy	Blank	One per route	No contamination present	> Identify and resolve the analytical problem before making analyses.
	Control Cultures (Positive and Negative)	For each lot, before using a culture medium of it	<i>Enterococcus faecium</i> : fluorescence; <i>Serratia marcescens</i> (gram -): no fluorescence; <i>Aerococcus viridans</i> (gram +): no fluorescence	> If controls indicate contamination, reject lot of the culture medium.
Representativeness	Data Quality Assessment and Verification	Every time the quality of the analytical data is assessed and verified.	Analysis adhered to method and SOPs procedures; data meet quality objectives control	> Flag questionable data; Memorandum or Corrective Action Notice

DQI	Field QC Check	Frequency of Collection	Acceptance Criteria	Corrective Action(s)
Comparability	Data Quality Assessment and Verification	Every time analytical data is revised, verified, and validated	Analytical procedures were followed as established in this QAPP, method reference and SOPs (calibration, holding time, reporting units)	> Flag questionable data; Memorandum or Corrective Action Notice
Completeness	Data Quality Assessment and Verification	Depends on the number of samples analyzed.	Greater or equal to 90%.	> Memorandum or Corrective Action Notice to PREQB-ELRPR regarding measures that should be taken to address problems.
Sensitivity	Data Quality Assessment and Verification	Every time the quality of the analytical data is assessed and verified.	Dimensions being measured fit inside Table 3 detection limit	> Memorandum or Corrective Action Notice

Anomalies on data will be reported as needed in order to implement a corrective action, if applicable. Corrective actions will be taken immediately after identifying the problem. The QAO will identify the problem and will report them to the WQA Manager, Sampling Division Chief, and/or Laboratory Manager. The manager will designate a person to correct the problem. The designated person shall develop an action plan, implement corrective measures, and document corrective processes and results until the problems are eliminated. It should be noted that, samples with exceedances in the holding time must be discarded (not analyzed) and a re-sampling must be carried out. Also, analysis out of the control limits should be evaluated by the QAO by the use of checklists and guidance's and based on it and their professional judgment a final determination will be made. In the worst case, data will be rejected and it should be necessary to carry out a re-sampling activity. The WQA Manager or Laboratory Manager should inform in writing, the reasons for exceedance of holding times. Table 6 includes the commonly kinds of assessments and the corrective action responses.

Table 6: Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response
Sample collection during field activities	<ul style="list-style-type: none"> <li>- Verbal briefing,</li> <li>- Corrective Action Notice, if severe violations noted</li> </ul>	<ul style="list-style-type: none"> <li>- Sampling personnel of the PREQB WSD</li> <li>- WSD Chief via WQA Manager</li> </ul>	<ul style="list-style-type: none"> <li>- Correct the practice immediately</li> <li>- Respond Corrective Action Notice</li> </ul>	PREQB QAO
Field Data review: chain of custody, Field forms	Memorandum or Corrective Action Notice	PREQB WSD via the WQA Manager	Memorandum or Response to the Corrective Action Notice	
Field Sampling Technical Audit	Field Audit Report	PREQB WSD Chief via the WQA Manager	Response of the Field Audit Report (if applicable)	
Laboratory Data: Data Narrative, Analytical methods, QA/QC data	<ul style="list-style-type: none"> <li>- Validation Report</li> <li>- Memorandum</li> <li>- Corrective Action Notice</li> </ul>	PREQB ERLPR Manager via WQA Manager	<ul style="list-style-type: none"> <li>- Memorandum</li> <li>- Corrective Action Response</li> <li>- Analytical Documentation</li> <li>- Certification</li> </ul>	
Proficiency Test Sample	<ul style="list-style-type: none"> <li>- Corrective Action Notice</li> </ul>	Microbiology Section Supervisor via PREQB ERLPR Manager	<ul style="list-style-type: none"> <li>- Corrective Action Response</li> <li>- Analytical Documentation</li> <li>- Certification</li> </ul>	PREQB ERLPR Manager

In order to guarantee a dynamic timeframe solving the problems and depending of the severity of the issue under consideration, different communication pathways should be implemented such as email communication, memorandums, or Corrective Actions Notices; and phone calls, among others. No matter the communication pathway used, evidence of it should be documented at the electronic or physical file.

## PERFORMANCE AND SYSTEMS AUDITS

The QAO shall conduct one technical performance audit of field and laboratory activities and any other audit that may be necessary during the project period. Findings will be reported to the manager of the area under audit, with a copy to the manager of the other areas that are part of the project. The manager is responsible for canalizing the report to the appropriate division for the corresponding action.

## QUALITY ASSURANCE REPORTS

The PREQB QAO is responsible of informing the WQA Manager on the QA status of the project and of any QA needs within the project. In addition, the QAO is also responsible for maintaining adequate communication with the PREQB Chief of the WSD and PREQB ERLPR Manager. In addition to these routine communication requirements, Table 7 includes the different QA Management Reports that are generated. It is important to emphasize that as required for Environmental Programs by the EPA Quality Manual for Environmental Programs (CIO 2105-P-01-0, formerly EPA Order 5360A1), a Quality Assurance Annual Report and Work Plan (QAARWP) will be prepared and submitted to the WQA and the USEPA QA Manager within 30 days from the completion of the fiscal year. This report will be prepared in accordance with requirements established in the Environmental Programs in the EPA Quality Manual for Environmental Programs for Agencies funded by the EPA, which require the following components:

- QA Annual Report:
  - o Quality Management Resources
  - o Trainings
  - o Management Accomplishments
  - o Management Assessments of the Approved Quality System
  
- Work Plan (for next fiscal year):
  - o Quality Management Resources
  - o Activities

In addition, this report contain, without being limited to, the following information:

- Status of the QA project plan;
- Significant QA problems, corrective actions undertaken, progress, needs, plans, and recommendations;
- Summary of QA/QC activities complete during the previous fiscal year:
  - o Results of the system audits, technical assessments and management assessment.
  - o Summary of Technical assistance given on systematic planning;

- o Summary of activities related to analytical data review, quality assessment and verification, including the evaluation of data accuracy, precision, completeness, representativeness, and comparability;
- o Summary of QA related training and activities;
- o Innovative Quality Management Practices developed and used in planning, implementing, or assessing the Quality System;
- Other information requested by management; and
- List and description of anticipated mayor QA/QC activities expected during the next year (e. g. QA/QC training; document review and revision; analytical data quality assessments; and management and technical assessments of participants of the Quality System).

Copies of this report will be provided to the PREQB WQA Manager, project officer, and the USEPA QA Manager.

Table 7: QA Management Reports

Type of Report	Frequency	Person(s) Responsible for Report Preparation	Report Recipient(s)
Field Change Request	As needed	- PREQB QAO - PREQB WSD	QAPP Recipients
QAPP reviews	As needed or as requested	- QA Officer - PREQB WSD	QAPP Recipients
Laboratory Results, Data Narrative and Quality Data	One/ after analysis of samples and laboratory checks	PREQB ERLPR Manager	PREQB QAO
Data Validation Report	One/ after the analytical and physical data is received from the laboratory and WSD.	PREQB QAO	- WQA Manager - PREQB Project Officer
Field Audit Report	One/ after field sampling audit activity is performed	PREQB QAO	PREQB WSD Chief via WQA Manager
Final Project Status QA Report	One/ in a year basis	PREQB QAO	- WQA Manager - PREQB Project Officer

## SPECIAL TRAINING NEEDS / CERTIFICATION

It will be required that the PREQB WSD personnel have updated the course of the 40 hours of Health and Safety, as well as the First Aid training. No specific training

is required for this project. The sampling team has been conducting water related research for over ten (10) years and all members are very experienced in this matter. The QAO performing the field technical audits must have the educational requirements and working experience established by the PREQB for their Job positions. It is essential that the QAO participated in classroom training courses, on-the-job basic trainings, and working knowledge in at least the following subjects: basic quality-related concepts and terms; quality systems; chemistry for environmental professionals; sampling designs to Support QAPP; chemical audits; environmental field sampling technics; contaminant free and certified sample containers; environmental media sampling; groundwater investigation and sampling procedures; sampling equipment selection; use, cleaning, and decontamination procedures; field sampling methods; water quality monitoring equipment; field monitoring equipment; and USEPA protocols for conducting field audits. Continuous education and training in the above subject, or any other area required to expand and improve the QA/QC and field sampling activities knowledge of the auditor, through internet-based training courses; in-house classroom and on-the-job training; and self-study is highly recommended. Finally, he/she must be familiar with the Agency QA regulations and current guidance documents.

For performing the review, quality assessment, and verification of analytical data, it is necessary that the QAO have a background in Science and strong knowledge on laboratory and analytical procedures. The person has to be capable of understand the analytical methods and interpret the analytical data. It is essential that the person be related with Quality concepts. Also, is better if the person has a previous experience working at an analytical laboratory.

Laboratory analysts are well trained and experienced in the laboratory's analytical methods. Analysts will be those who are dedicated exclusively to perform analyses on environmental water samples. Laboratory accreditation or certification is not required for participating in this study. However, a Proficiency Test (PT) by each analyst must be run once a year. If PT are not run, a Demonstration of Capability (DOC) should be done by each analyst. The results should be documented.

Nevertheless, before starting any task, all personnel of the laboratory and WSD participating in the study will be retrained on the procedures to be carried out, the equipment to be used, and the safety measures to be taken to avoid any accident. In addition, the QAPP will be discussed with the personnel that will participate in the project.

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# Appendix 1: Figures

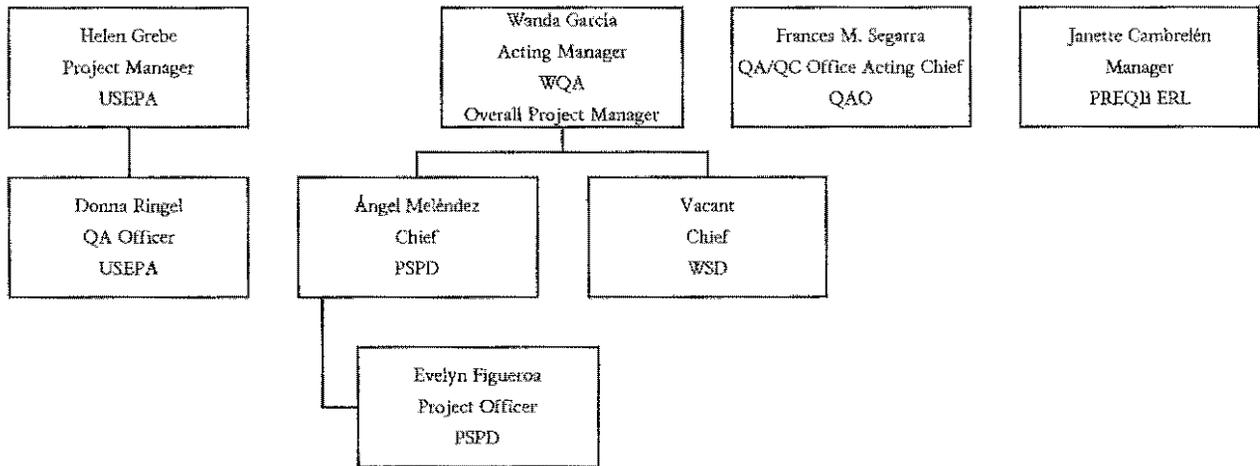
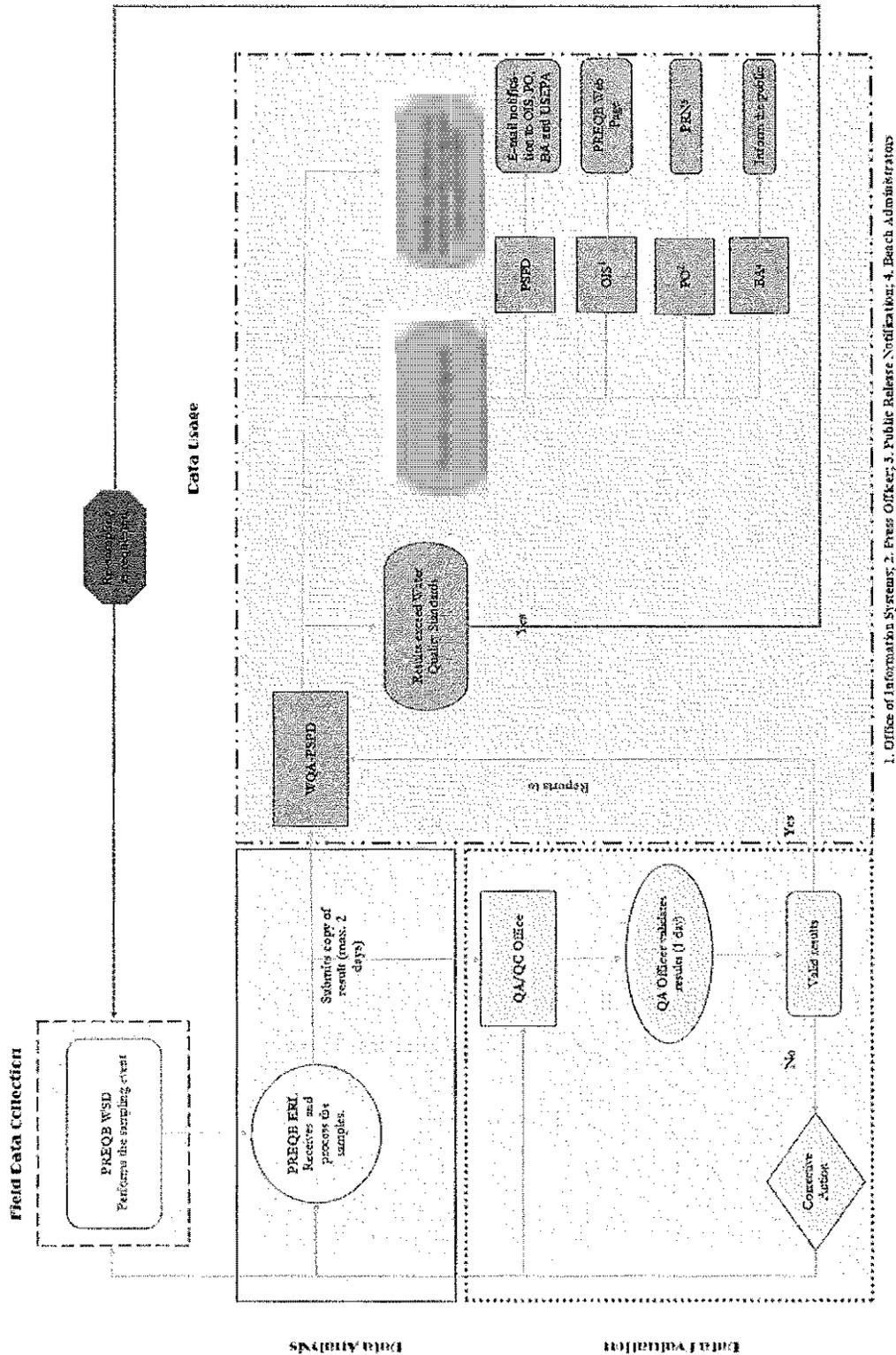
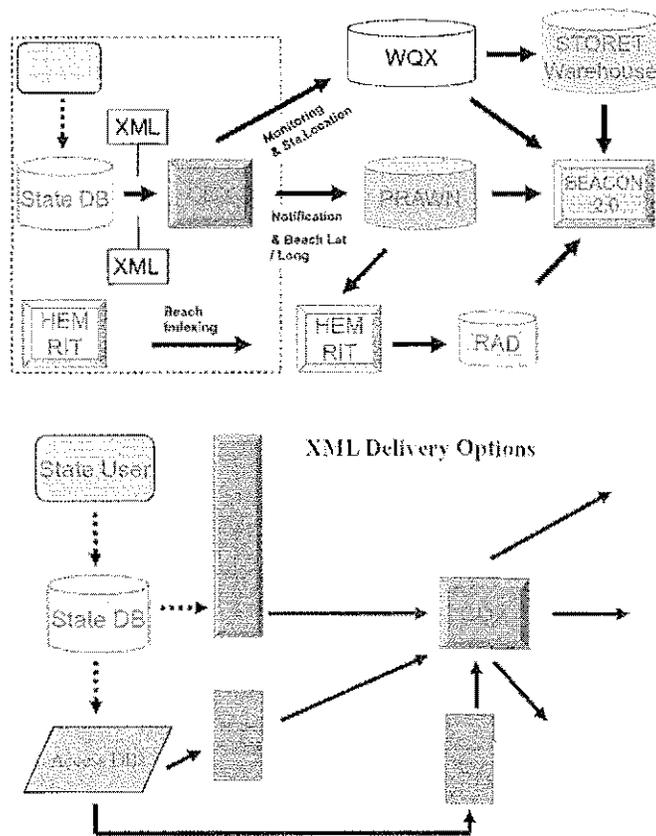


Figure 1: Organization Chart



1. Office of Information Systems; 2. Press Officer; 3. Public Release Notification; 4. Beach Administrators

Figure 2: Public Notification Procedure Flowchart



### Beach Data Flow Definitions

- BEACON 2.0 – Beach Advisories and Closings Online Notification system (modernized).
  - Internet application to display beach location, water quality and advisory data.
- CDX – Canal Data eXchange
- Node – computer that exchanges data between submitter and receiver; may validate against receiving schema and return error messages to submitter
- PRAWN – GST Program tracking, beach Advisory, Water quality standard, and Nutrient database.
  - Stores beach location and advisory data
- RAD – Beach Address Database.
  - Stores DW location data.
- STORET – OW STORAGE and REtrieval database
  - Stores OW water quality data and station location data
- HEM-RIT – PC based Beach Index Tool
  - Tool to add or edit geo-referenced location data
- WQX – Water Quality Exchange
  - Accepts and configures water quality data for transmission to STORET

Figure 3: Monitoring and Notification Process



JUNTA DE CALIDAD AMBIENTAL  
 ÁREA DE CALIDAD DE AGUA  
 DIVISIÓN DE MUESTREO DE AGUA

HOJA DE INSPECCIÓN DE CAMPO

INFORMACIÓN GENERAL										
PROYECTO - LOCALIZACIÓN:				FECHA:		HORA:                      am                      pm				
				TEMPERATURA DEL AGUA (°C):		POZO EN USO (N/A <input type="checkbox"/> ):				
				SI <input type="checkbox"/>		No <input type="checkbox"/>				
ID Y DESCRIPCIÓN DE LA ESTACIÓN :				TÉCNICOS:						
CONDICIONES DEL TIEMPO										
ANTES DEL MUESTREO		DURANTE EL MUESTREO		VIENTO (CALMADO <input type="checkbox"/> )			MAREAS (N/A <input type="checkbox"/> )			
Soleado	<input type="checkbox"/>	Soleado	<input type="checkbox"/>	Norte	<input type="checkbox"/>	Noreste	<input type="checkbox"/>	Alta	<input type="checkbox"/>	
Nublado	<input type="checkbox"/>	Nublado	<input type="checkbox"/>	Sur	<input type="checkbox"/>	Noroeste	<input type="checkbox"/>	Baja	<input type="checkbox"/>	
Lluvioso	<input type="checkbox"/>	Lluvioso	<input type="checkbox"/>	Este	<input type="checkbox"/>	Sureste	<input type="checkbox"/>	Alta / Baja	<input type="checkbox"/>	
Soleado/Nublado	<input type="checkbox"/>	Soleado/Nublado	<input type="checkbox"/>	Oeste	<input type="checkbox"/>	Suroeste	<input type="checkbox"/>	Baja / Alta	<input type="checkbox"/>	
ACTIVIDADES CERCANAS AL PUNTO DE MUESTREO										
RECREATIVAS (N/A <input type="checkbox"/> )		VEHICULOS ACUÁTICOS (N/A <input type="checkbox"/> )		OTRAS						
Área de bañistas	<input type="checkbox"/>	Anclados	<input type="checkbox"/>	Agrícolas	<input type="checkbox"/>	Construcción				<input type="checkbox"/>
Actividades con remo	<input type="checkbox"/>	En movimiento	<input type="checkbox"/>	Pecuarías	<input type="checkbox"/>	Mantenimiento				<input type="checkbox"/>
Pesca	<input type="checkbox"/>	Jet Sky	<input type="checkbox"/>	Comerciales	<input type="checkbox"/>	Vertedero				<input type="checkbox"/>
Recreación pasiva	<input type="checkbox"/>	Kayak	<input type="checkbox"/>	Industriales	<input type="checkbox"/>	Comunidades				<input type="checkbox"/>
Otro:	<input type="checkbox"/>	Otro:	<input type="checkbox"/>	Gravero	<input type="checkbox"/>	Otro:				<input type="checkbox"/>
CARACTERÍSTICAS CUERPO DE AGUA				POSIBLES FUENTES DE CONTAMINACIÓN						
Claro	<input type="checkbox"/>	Acetate/grasa	<input type="checkbox"/>	DESCARGAS (N/A <input type="checkbox"/> )		DESEMBOCADURA (N/A <input type="checkbox"/> )		SISTEMA PLUVIAL (N/A <input type="checkbox"/> )		
Turbio	<input type="checkbox"/>	Algas	<input type="checkbox"/>	Pozos sépticos		<input type="checkbox"/>	Abierta		<input type="checkbox"/>	
Sedimentado	<input type="checkbox"/>	Plantas	<input type="checkbox"/>	Residenciales		<input type="checkbox"/>	Obstruida		<input type="checkbox"/>	
Olores	<input type="checkbox"/>	Oleaje	<input type="checkbox"/>	Industriales		<input type="checkbox"/>	parcialmente		<input type="checkbox"/>	
Desp. sólidos:	<input type="checkbox"/>	Comerciales		<input type="checkbox"/>	totalmente		<input type="checkbox"/>	Obstruido		<input type="checkbox"/>
Animales:	<input type="checkbox"/>	Agrícolas		<input type="checkbox"/>	Sedimentada		<input type="checkbox"/>	parcialmente		<input type="checkbox"/>
Otro:	<input type="checkbox"/>	Otro:		<input type="checkbox"/>	Desperdicios sólidos		<input type="checkbox"/>	totalmente		<input type="checkbox"/>
COMENTARIOS:										

Edificio Agencia: Ambientales Cruz A. Matos  
 Ave. Ponce de León 1375, San Juan, PR 00926-2604  
 Apartado 11488, San Juan, PR 00910  
 Tel. 787-767-4181

Figure 4: Field Inspection Form





 <b>EOB</b> <small>Environmental Quality Board</small> Urb. San José Industrial Park Ave. Ponce de León 1375 San Juan, PR00926-2604		Environmental Quality Board Water Sampling Division	Sec. #
Sampling site: _____			
Date:	Time:	By:	
Grab	Comp.	Preservative	
Test Required:			

Figure 7: Bottle Identification Label

# Appendix 2: Public Release Notification Example



**PRESS RELEASE**

Notice of Noncompliance with Water Quality Parameter for # Beaches

The Water Quality Area of the Environmental Quality Board (EQB) reported that the following beaches exceeded bacteriological parameter in samples taken the \_\_\_\_ day of \_\_\_\_\_.

After being subjected to the appropriate test in Environmental Research Laboratory of the EQB the following beaches exceeded the parameter:

- -----
- -----
- -----
- -----

"Under the Federal Beaches Environmental Assessment and Coastal Health Act of 2000, the EQB has implemented the Beaches Monitoring Program and Public Notification (PMPNP), which aims to reduce the risk of developing diseases to bathers exposed to the bacteriological pathogens. Our main recommendation is that swimmers should avoid primary contact with these bodies of water, because the pathogenic organisms (enterococci) can cause diseases in the skin, eyes, nose, throat and gastrointestinal system ", said the president of the JCA, attorney Laura M. Vélez Vélez.

Other essential advice offered by the expert staff of the EQB is that after continuous rain events it is not recommended that you come in contact with water bodies until the end of twenty-four (24) hours period after these have been finalized. Moreover, citizens should avoid bathing in areas of the beaches that are near the mouths of rivers and streams.

"For more information please visit the website (<http://www.jca.gobierno.pr/>) where you can see the map with the location of the sampling stations and status of compliance with water quality standards," stated the president of the EQB.

Contact:  
Sara M. Justice Doll  
Manager of Communications and Education at the JCA  
787 552 8150

# Appendix 3: Beaches and Monitoring Stations Location

## Beaches and Monitoring Stations Location

Beach Name	Location	AU ID	Classification	Station ID and Location	Coordinates
<b>Route 1: Dorado - Loíza</b>					
Balneario Manuel "Nolo" Morales or Sardinera	Road PR-693 Int. PR-697, Costas de Oro, Dorado	PRNC08	SB	RW-18 At the front of the administration building.	18°28'28.90" 66°16'51.21"
Balneario Punta Salinas	Road PR-165, Levittown, Toa Baja	PRNC09	SB	RW-19 At the front of the administration building.	18°28'17.97" 66°11'09.58"
Balneario El Escambrón	Muñoz Rivera Ave., Stop 8, Puerta de Tierra, San Juan	PREC12	SB	RW-20A At the front of the showers and lifeguard stand.	18°28'02.05" 66°05'23.85"
Playa Sixto Escobar	Muñoz Rivera Ave., Stop 8, Puerta de Tierra, San Juan	PREC12	SB	RW-25A At the center of the bathers area.	18°28'00.23" 66°05'12.00"
Playita del Condado	Ashford Ave., west to El Condado Plaza Hotel, San Juan	PRC13	SB	RW-26 At the center of the bathers area.	18°27'40.07" 66°04'56.67"
Ocean Park	General Patton Street, San Juan	PREC13	SB	RW-27 At the center of the bathers area.	18°27'10.84" 66°02'55.97"
Playa El Alambique	Isla Verde Ave., José M. Tartak Street, Carolina	PREC14	SB	RW-28 At the center of the bathers area.	18°26'38.73" 66°01'19.74"
Balneario de Carolina	Road PR-187, Boca de Cangrejos, Carolina	PREC14	SB	RW-21C At the center of the bathers area.	18°26'45.56" 66°00'12.86"
Vacia Talega	Road PR-187, Loíza	PREC15	SB	RW-29 At the center of the bathers area.	18°26'52.29" 65°54'22.43"

Beach Name	Location	AU ID	Classification	Station ID and Location	Coordinates
<b>Route 2: Arroyo - Luquillo</b>					
Balneario Punta Guilarte	Road PR-3 Km 126, Arroyo	PRSC32	SB	RW-7 At the center of the bathers area	17°57'43.35" 66°02'24.00"
Balneario de Patillas	Road PR-3 Km 1.7, Los Bajos Ward, Patillas	PRSC32	SB	RW-6 At the center of the bathers area.	17°58'26.31" 65°59'20.33"
Playa Guayanés	El Ancla Beach Hotel, Yabucoa	PREC28C	SB	RW-30 At the center of the bathers area.	18°03'45.70" 65°49'09.10"
Balneario Punta Santiago	Road PR-3 Km 72.4, Humacao	PREC25	SB	RW-4 At the center of the bathers area.	18°09'30.29" 65°45'18.67"
Tropical Beach	Road PR-3 (confluence of Río Blanco and Río Santiago), Naguabo	PREC25	SB	RW-31 At the center of the bathers area.	18°11'12.94" 65°43'33.48"
Balneario Seven Seas	Road PR-195 Km 4.8 Las Croabas, Fajardo	PREC18	SB	RW-2 At the center of the bathers area	18°22'09.36" 65°38'09.86"
Playa Azul	Luquillo Beach Boulevard (east of town), Luquillo	PREC18	SB	RW-32 At the center of the bathers area	18°22'54.72" 65°43'06.45"
Balneario La Monserrate	Road PR-3, Luquillo	PREC17	SB	RW-1A In front of the administration building.	18°23'08.13" 65°43'46.1"
<b>Route 3: Lajas - Salinas</b>					
Playita Rosada	Camino de los Guayacanes, Lajas	PRSC41B2	SB	RW-33 In the pool.	17°58'18.18" 66°01'53.40"
Playa Santa	Road PR-325 Final, Providencia, Guánica	PRSC41B1	SB	RW-10 At the center of the bathers area, in front of AEELA building.	17°56'15.76" 66°57'18.71"

Beach Name	Location	AU ID	Classification	Station ID and Location		Coordinates	
Caña Gorda	Road PR-333 Km 2.6 Caña Gorda Ward, Guánica	PRSC40	SB	RW-9	At the center of the bathers area.	17°57'09.11"	66°53'04.42"
Playa del Hilton	Ponce Hilton Hotel, Ponce	PRSC36B	SB	RW-34	In front of the hotels bathers area.	17°58'9.42"	66°36'9.82"
Playa Jauca	Road PR-1 Parcelas Jauca 6 Street, Santa Isabel	PRSC34	SB	RW-35	At the center of the bathers area.	17°57'35.60"	66°22'13.50"
Balneario de Salinas	Road PR-1, Salinas	PRSC34	SB	RW-36	At the center of the bathers area.	17°58'39.32"	66°19'56.99"
<b>Route 4: Cabo Rojo</b>							
Playa el Combate	Road PR-3301 Final West Side, Cabo Rojo	PRSC43	SB	RW-12B	Near Los Salitrales	17°58'29.26"	67°12'46.46"
Playa Moja Casabe	Road PR-3301 Final, East Side, Cabo Rojo	PRWC43	SB	RW-14A	Alongside of where the office of the Department of Natural and Environmental Resources is located	17°58'57.49"	67°12'55.51"
Balneario de Boquerón	Road-101, Poblado Boquerón, Cabo Rojo	PRWC43	SB	RW-13	At the center of the bathers area.	18°01'09.99"	67°10'20.08"
Playa Buyé	Road PR-307 Km 3.8, Pederrales Ward, Cabo Rojo	PRWC44	SB	RW-8	At the center of the bathers area	18°02'55.94"	67°11'55.05"
Villa Lamela	Camino La Mela Final, Cabo Rojo	PRWC44	SB	RW-37	At the center of the bathers area.	18°03'52.32"	67°11'51.10"

Beach Name	Location	AU ID	Classification	Station ID and Location	Coordinates
<b>Route 5: Añasco - Aguadilla</b>					
Balneario de Añasco or Balneario Tres Hermanos	Road PR-115 Km 5, Hatillo Ward, Añasco	PRWC49	SB	RW-15 At the center of the bathers area.	18°17'16.79" 67°11'38.12"
Balneario de Rincón	Road PR-115 Int. Cabijas, Rincón	PRWC50	SB	RW-5 At the center of the bathers area.	18°20'27.33" 67°15'21.62"
Pico de Piedra	Road PR-115 Km 21, Aguada	PRWC51	SB	RW-22 At the center of the bathers area.	18°23'03.71" 67°12'46.76"
Balneario Crash Boat	Road PR-458 Final, Borinquen Ward, Aguadilla	PRWC52	SB	RW-16 At the center of the bathers area.	18°27'27.60" 67°09'49.60"
<b>Route: 6: Arecibo – Vega Alta</b>					
Muelle de Arecibo	Road PR-655, Arecibo	PRNC03	SB	RW-38 At the center of the bathers area.	18°28'45.33" 66°42'01.68"
Mar Chiquita	Road PR-648, Manatí	PRNC05	SB	RW-39 At the center of the bathers area.	18°28'22.50" 66°29'08.36"
Balneario de Puerto Nuevo	Road PR-692 Km 12, Vega Baja	PRNC06	SB	RW-23 At the center of the bathers area.	18°29'28.92" 66°23'56.56"
Balneario Cerro Gordo or Javier Calderón Nieves	Road PR-690, Cerro Gordo Ward, Vega Alta	PRNC07	SB	RW-17 At the center of the bathers area.	18°28'52.50" 66°20'26.36"
AU = Assessment Unit SB = Coastal and estuarine waters designated for primary and secondary contact recreation, and propagation and preservation of desirable species including threatened or endangered species, as defined in the PREQBWQSR. RW = Recreational Waters					

# Appendix 4: Water Sampling Division's SOPs

# Surface Water Sampling



COMMONWEALTH OF  
PUERTO RICO  
ENVIRONMENTAL QUALITY BOARD

TITLE: STANDARD OPERATING PROCEDURE (SOP): SURFACE WATER SAMPLING  
SOP NO.: 100  
REVISION NO. 1.0  
EFFECTIVE DATE: MARCH 27, 2015  
AREA/DIVISION: WATER QUALITY AREA/WATER SAMPLING DIVISION

PREPARED BY:

SIGNATURE: *Otto A. Osorio Nieves* DATE: 27 May 2015  
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POSITION: ENVIRONMENTAL SAMPLING OFFICER II, WATER SAMPLING DIVISION

SIGNATURE: *Stephenie M. Ayala Flores* DATE: 27 de mayo 2015  
NAME: STEPHENIE M. AYALA FLORES  
POSITION: ENVIRONMENTAL QUALITY SPECIALIST II, PLANS AND SPECIAL PROJECTS DIVISION

REVIEWED BY:

SIGNATURE: *Wanda E. García Hernández* DATE: March 27, 2015  
NAME: WANDA E. GARCÍA HERNÁNDEZ  
POSITION: ACTING MANAGER, WATER QUALITY AREA

SIGNATURE: *César O. Rodríguez Santos* DATE: March 27, 2015  
NAME: CÉSAR O. RODRÍGUEZ SANTOS  
POSITION: QUALITY ASSURANCE AND QUALITY CONTROL SPECIALIST II,  
QUALITY ASSURANCE AND QUALITY CONTROL OFFICE

APPROVED BY:

SIGNATURE: *Frances M. Segarra Román* DATE: March 27, 2015  
NAME: FRANCES M. SEGARRA ROMÁN  
POSITION: ACTING CHIEF, QUALITY ASSURANCE AND QUALITY CONTROL OFFICE



### Revision History

This table records the revision number, revision date, author and a brief summary of the changes that have been made to the SOP. The revision history log must be completed whenever an alteration to the document is made.

Revision History Log			
Revision No.	Date	Author(s)	Changes
1.0	March 2015	Otto A. Osorio Nieves / Stephenie M. Ayala Flores	<ul style="list-style-type: none"> <li>o Inclusion of the Revision History.</li> <li>o Item 2.1: SOP applicability.</li> <li>o Item 7.3: Name correction of SOP 131, Management and Maintenance of Coolers, Refrigerator and Ice Maker.</li> <li>o Item 12.1: Elimination of Tables 1, 2 and 3. Substitution of the tables with the following sentence Refer to the section of the QAPP with respect to the parameters, containers, and preservatives. In addition, any reference to the tables throughout the document was removed.</li> <li>o Item 14.2.6: Inclusion of the sentence "Nevertheless, in beaches of high-energy environments, collect the samples at ankle depth because it is safer".</li> <li>o Inclusion of item 15.10.11.19, referring to transference of samples to another authorized PREQB personnel.</li> <li>o Figures 1, 2, 3, 4, and 5 of Appendix 1</li> <li>o Insertion of the definition of various acronyms. Use of multiple acronyms throughout the document.</li> <li>o Modification of the SOP format as part of the Quality Assurance and Quality Control Office effort to standardize all PREQB SOP documents.</li> </ul>



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## 1. SOP TITLE

Standard Operating Procedure (SOP): Surface Water Sampling

## 2. APPLICABILITY

2.1. This SOP applies to the collection of aqueous samples from any surface, coastal or estuarine water source as defined by the Puerto Rico Water Quality Standards Regulation (PRWQSR).

## 3. PURPOSE

The duty of the Puerto Rico Environmental Quality Board (PREQB) - Water Sampling Division (WSD) is to collect water samples to be analyzed for different parameters in order to determine the quality of the water bodies; to assess any impact of point and nonpoint pollution sources in these water bodies, and to detect any violation to the PRWQSR.

3.1 This SOP is written primarily as specific instructions for the WSD technical personnel, to facilitate their investigations and the generation of the necessary environmental data, as well as the supporting documentation, in all field surface water sampling events.

3.2 Data collected will be included in the biannual report of the 305(b) project or any other required environmental report. In addition, this data will be uploaded into the Environmental Protection Agency (EPA) Storage Retrieval (STORET) Data Warehouse, and kept at the Plans and Special Projects Division (PSPD); so it can be available for the use of the general public and any state, local or federal government agency.



3.3 In addition, the data collected may be used as reference by laboratories, students, or any other person interested in the water quality of any surface water bodies.

#### 4. METHOD SUMMARY

4.1 Surface water sampling is an important tool to obtain data that will facilitate the scientific analysis of the water quality.

4.2 This document will emphasize on the techniques, procedures, and instrumentation that are used or required for surface water sampling.

4.3 Surface water sampling can be performed in natural or artificial water sources, which include: creeks, rivers, lakes, beaches, estuaries, reservoirs, streams or channels, springs, irrigation systems, and drainage systems.

#### 5. DEFINITIONS

5.1 Grab Sample: Is a simple sample collected at a point/place and specific time period, usually short time (seconds or minutes).

5.2 Composite Sample: Is a sample in which multiple grab sample fractions are collected from the same sampling location or station, and at different time intervals, but that are physically combined and mixed into a single sample. It is considered also a composite sample, the combination (into a single sample) of multiple grab samples collected in different places.

5.3 Field Replicate: Two or more readings taken or collected immediately one after another in the same place, to avoid variation in the results. This applies to the field readings performed with instrumentation *In situ*, which is necessary to establish data precision.



- 5.4 Field Duplicate or Triplicate: Independent samples that are collected as close as possible from the same point in space and time for a given parameter. These are samples that are collected from the same source, stored in separate containers and analyzed independently. The integrity of the sample handling is determined through these samples. Also, field duplicates are collected to check reproducibility of laboratory and field procedures.
- 5.5 Equipment/Rinse Blank: It is a sample collected to ensure that the process of decontamination of the equipment was performed adequately. The equipment blank is collected before using any non-dedicated, decontaminated, and reusable sampling equipment to collect the samples to be analyzed for the parameters established for a project, and by pouring analyte free water provided by the laboratory over the equipment and collecting this rinse water directly into the sample container. The container must be equal to the one required and used for the rest of the samples based upon the parameter for which the sample will be analyzed. Handling of this sample has to be performed in the same way as the samples to be collected. This sample has to be identified as Equipment Blank and submitted to the laboratory with the rest of the samples for analysis.
- 5.6 Trip Blank: This is a sample prepared by the laboratory with analyte-free medium. If the sample to be collected is saline, the trip blank should be of saline water. This sample must be transported from the laboratory to the sampling site and returned to the laboratory. This sample should be collected only when samples for the analysis of volatile organic compounds (VOCs) are collected. It is transported in all coolers along with the samples



to be collected for the analysis of VOCs, as it aims to document any changes in pressure, temperature, and contamination during the process of preservation and transportation of samples. Since it is the first sample to be carried on the cooler, it must have to have the sequence number previous to the one of the samples collected in the field. This sample cannot be opened. In addition, any reagent cannot be added to the sample, nor it can be transferred to another container. The sample has to be documented on the Chain of Custody, and it has to be transported and delivered to the laboratory for its analysis when delivering all samples.

- 5.7 Filter Blank: A filter is used from the same batch of filters used for Chlorophyll "A" parameter. The same amount of milliliters required for the parameter is filtered using analyte-free water, provided by the laboratory. Handling of this sample has to be performed in the same way as the samples for this parameter.
- 5.8 Field Blank: It is a sample used to evaluate the potential of an environmental contaminant not associated with the sampling. Also, this blank is collected to check for cross-contamination during sample collection, preservation, and shipment; as well as to check the sample containers and preservatives. The blank can be prepared in two ways, namely: 1) in the field, using water supplied by the laboratory, fill a container with the same volume and specifications of the parameter that will be analyzed; 2) the laboratory could give you the bottles ready to be used in the field. In both cases after reaching the study area, identify a place near the sampling point where you have visual contact with the container. The container is kept open for the duration of the sampling.



After the sampling process is finished, preserve the sample following the parameter specifications, close the bottle, and submitted it to the laboratory along with the samples collected.

- 5.9 Shipping Container Temperature Blank: This blank is not a sample. It is a bottle identified as “Temp”, which serves as a control for the laboratory that receives the samples collected to document the temperature at which the samples have been preserved and transported. The bottle is placed next to the samples collected in the field that require a preservation temperature equal or less than 6°C.
- 5.10 Global Positioning System (GPS): It is the instrument to be used for taking the coordinates of the place/point in which the samples are collected.
- 5.11 Quality Assurance Project Plan (QAPP) – Is the document that describes in detail the technical activities to be implemented to ensure that the results of the work carried out meet the established performance criteria.
- 5.12 Health and Safety Plan: The set of procedures and technical processes, as well as the persons responsible, of ensuring the compliance with the occupational health and safety levels required, minimizing the risks related to work.

## 6. HEALTH AND SAFETY CONSIDERATIONS

- 6.1 Always consider the weather conditions.
- 6.2 Handle carefully the glass bottles and the chemical reagents (acids and bases) used for sample preservation.
- 6.3 Always use latex gloves. The gloves not only protect the field technician, they also avoid samples cross contamination.



- 6.4 Do not use the ice cooler to store food or beverages, to avoid cross contamination of the samples and contamination of the food or beverage with any contaminant or chemical reagents (acids or bases) used for sample preservation, which may lead to injury.
- 6.5 Always wear a lifejacket when collecting samples from a boat.
- 6.6 A fire extinguisher must be carried in the boat at all times.

## 7. PRECAUTIONS

- 7.1 Comply with the requirements and specifications of the container according with the parameter (glass, plastic, amber) and ensure that all the containers are clean (Refer to the QAPP of the project).
- 7.2 Always decontaminate the equipment before and after using it. Refer to section 16.0 of this SOP.
- 7.3 Always keep clean the ice coolers used to transport the samples. Refer to SOP 131, Management and Maintenance of Coolers, Refrigerator, and Ice Maker.

## 8. SPECIAL CONSIDERATIONS

- 8.1 The laboratory could analyze more than one parameter per container.
- 8.2 Bring additional bottles per parameter, if any is broken or contaminated, or if you need to collect another sample for any suspicion of contamination.
- 8.3 Verify the proper operation of the field equipment before leaving the facility to collect the samples.
- 8.4 Remember to put the Shipping Container Temperature Blank in the ice cooler, before leaving the facility to collect the samples.



- 8.5 The technician shall use a pair of new gloves for every station and for every process of sample preservation.
- 8.6 When sampling rivers and creeks, collect the samples against the current, and start with the downstream sampling point or location first, and then move upstream until all the samples are collected from the water body.
- 8.7 Before using the equipment for sampling (e.g.: dipper, bucket, etc.), rinse it three times with water from the point where the samples are going to be collected.
- 8.8 The volume and type of bottle will be given according to the requirement in the methodology used by the laboratory.
- 8.9 Fill up to 90% of the volume of the bottles that will be preserved.
- 8.10 The containers of the samples for the analysis of VOCs and dissolved oxygen must be filled entirely; bubbles or a void space inside the sample container must be avoided.
- 8.11 The bottles used for oil and grease, bacteriological and metal parameters cannot be rinse, since the laboratory gives them a special wash.
- 8.12 When establishing the work schedule, the holding time of the samples, as specified at their respective method and QAPP, must be taken into account so it is not exceeded.
- 8.13 Bacteriological samples must be delivered to the laboratory within the statutory waiting period of six (6) hours.
- 8.14 When collecting samples using a boat, approach slowly to the station that is going to be sampled, to avoid turbulence.
- 8.15 The information collected and documented in the investigation and sample collection process must be consistent across all data sheets. Always check



the details, such as ID number, description of the stations, date, time, and labels.

- 8.16 The information included in the Chain of Custody must be the same as the specified on the labels and in all documents required for the specific investigation and sampling process.

## 9. INTERFERENCES

- 9.1 The sampling success is directly related to the care taken when collecting and handling the samples. This includes making sure that the required field sampling documentation (i. e. Chain of Custody Sheets, Field Inspection Form, etc.) is generated and completely filled with the required information.
- 9.2 Always use the clean bottles provided by the laboratory and make sure that they are the appropriate ones based upon the parameter for which the samples are required to be analyzed.
- 9.3 Seal with paraffin paper the samples that have been preserved with acids or bases.
- 9.4 Samples taken for organic parameters, such as VOCs, SVOCs, pesticides and total hydrocarbons (TPH DRO and GRO), should not be sealed with wax paper, as the plastic interferes with the laboratory results. The caps of the bottles used for these compounds must be of Teflon.
- 9.5 Do not use any seal for the bacteriological parameters samples. The bottle used is sterile and has a special lock that prevents liquid from entering or leaving it.



- 9.6 To reduce cross contamination, avoid as much as possible, the handling and transfer of samples to other containers. However, this must be avoided. If an incident occurs that may increase the sample potential of cross contamination, it must be documented in the Chain of Custody and any other required document.
- 9.7 Never touch the inside of the lid and the bottle used to collect the sample.
- 9.8 To avoid cross contamination, the sampling technician must ensure that the disposable latex gloves are replaced by a new pair after the collection of samples in one station and before the collection of samples in the next station.
- 9.9 Do not use dippers with metal components to collect samples which will be analyzed for metals.
- 9.10 When taking samples for the analysis of organic components, do not use dippers with plastic components.
- 9.11 If the investigation includes collecting sediment samples, the surface water samples must be collected before the sediment samples of any specific sampling point. Also, the samples should be collected starting with the downstream sampling point or location first, and then moving in an upstream direction until all the samples are collected.
- 9.12 Once the samples are delivered to the laboratory, discard the ice used for their preservation. Do not reuse it.

## 10. STAFF TRAINING

- 10.1 The technical staff should be trained in the use and handling of chemical reagents, as well as the preservation of the samples with them.



- 10.2 The technical staff must possess a license for use, handling and transportation of explosives, issued by the Police Department of Puerto Rico, which authorizes the use, handling and transportation of chemical reagents that are used to preserve the samples.
- 10.3 The technicians must be trained on the filtration process regarding chlorophyll "A".
- 10.4 The technicians should be trained on the calibration, the use and management of the field instruments for the readings of pH, dissolved oxygen, salinity, conductivity, temperature, turbidity, and transparency, as well as GPS.
- 10.5 It is required that the technical employees have updated the course of the 40 hours HAZWOPER, as well as the First Aid training.
- 10.6 For the use of boats, the technician must have a navigation license, which is issued by the Department of Natural and Environmental Resources.

## 11. EQUIPMENT AND MATERIALS

- 11.1 The following list includes the equipment and materials needed to carry out the surface water sampling:
  - Bottles (the necessary according to the parameters specify by the Method for which samples are going to be taken and additional containers if a bottle is broken or contaminated)
  - Ice cooler and Shipping Container Temperature Blank
  - Distilled or deionized water, sufficient to decontaminate the sampling equipment



- pH indicator or Litmus paper
- GPS
- Watch
- Funnel
- Paraffin paper
- Absorbent paper
- Disposable pipettes
- Sampling equipment (Kemmerer, bucket with rope, and dipper, among others)
- Instruments to measure the field physical parameters
- Waterproof marker
- Chemical reagents to preserve the samples
- Disposable latex gloves
- Safety spectacles
- First Aid kit
- Safety boots
- Rubber or Wellington boots
- Spill control equipment for acids and bases
- Camera
- Navigation equipment (Refer to SOP 130)
- Data compilation sheets
- Plastic bags to dispose of the solid waste generated during sampling



## 12. PARAMETERS, CONTAINERS, AND PRESERVATIVES

- 12.1 Refer to the section of the specific QAPP and /or Method with respect to the parameters, containers, and preservatives.

## 13. TYPES OF SAMPLES

- 13.1 There are two basic forms for the collection of samples: simple or grab and composite.
- 13.2 A grab sample is a single sample collected at a specific point and period of time interval. Therefore, it represents a “snapshot” in time and space of the site.
  - 13.2.1 Discrete grab samples are taken at a selected location, depth, and time.
  - 13.2.2 Depth-integrated grab samples are collected over a predetermined part or the entire depth of a water column, at a selected location and time in a given body of water.
  - 13.2.3 When a source is known to vary with time, grab samples collected at suitable intervals and analyzed separately can document the extent, frequency, and duration of these variations. When the source composition varies in space (i.e., from location to location) rather than time, collect samples from appropriate locations that will meet the objectives of the study (for example, upstream and downstream from a point source, etc.).
- 13.3 A composite sample is obtained from the physical combination of multiple grab samples collected at the same place and at different periods of time.



Also, a composite sample is obtained from the combination (in a single sample) of multiple grab samples collected at different places.

13.3.1 Composite samples can be obtained by combining portions of multiple grab samples or by using specially designed automatic sampling devices.

13.3.2 Sequential (time) composite samples are collected by using continuous, constant sample pumping or by mixing equal water volumes collected at regular time intervals.

13.3.3 Flow-proportional composites are collected by continuous pumping at a rate proportional to the flow, by mixing equal volumes of water collected at time intervals that are inversely proportional to the volume of flow, or by mixing volumes of water proportional to the flow collected during or at regular time intervals.

13.3.4 Use composite samples only to determine parameters that can be identified and that remain unchanged during the collection, preservation and storage of samples.

## 14. PROCEDURE FOR SURFACE WATER SAMPLING COLLECTION

### 14.1 Before Collecting the Sample

#### 14.1.1 Locate the station

14.1.1.1 The location of the sampling station can be preselected in the QAPP. Be aware about the purpose of the project and the sampling.

14.1.1.1.1 Identify and observe the characteristics of the point where the samples will be collected.



Record the information in the Field Inspection Form (See Figure 2 of Appendix 1).

14.1.1.2 Nevertheless, if an emergency occurs, you have to select the point where the samples will be collected.

14.1.1.2.1 Select a convenient, accessible, practical, and safe place for both the technicians and the equipment.

14.1.1.2.2 Consider the weather conditions. Record the observations in the Field Inspection Form and any other documents that apply.

14.1.1.2.3 Identify and observe the characteristics of the point where the samples will be collected. Set the sampling point where the stream is as homogeneous as possible. Collect the sample in the point that is more representative for the study.

14.1.1.2.4 Sometimes, convenience or accessibility becomes secondary to the representativeness of the sample; therefore, take all safety measures.

14.1.1.2.5 Identify the factors that could affect the outcome of the samples, such as: tributaries near the point where you will collect the sample, authorized and unauthorized



discharges, industries and mouths of water bodies, among others.

14.1.1.2.6 Describe the point at which the samples will be collected. Establish reference points, including diagrams and maps to locate it.

14.1.1.2.7 Take coordinates with a GPS (Refer to SOP 120). Record the coordinates in the sheet identified as *Notas de Campo – GPS* (See Figure 1 of Appendix 1).

14.1.1.2.8 Document your observations in the Field Inspection Form and Chain of Custody (See Figures 2 and 3 of Appendix 1 of this SOP).

14.1.2 Determine the type of sample to be collected (grab, composite or both). This information is included in the specific QAPP and Method of the project. Similarly, select the sampling method to be employed (direct method [grab sample] or Kemmerer, among others).

14.1.3 If you collect a composite sample:

14.1.3.1 Select a wide-mouth container with lid (alpha bottle), with sufficient capacity to collect the volume that will be generated from the collection of grab samples.

14.1.3.2 The container must be clean and must be rinsed with water from the point where the grab samples will be collected.

14.1.3.3 Set the number of grab samples to be collected and the time interval between them.



14.1.3.4 If a composite sample is collected in a column, collect the samples from top to bottom in the column of water.

14.1.3.5 As you collect the grab samples, pour each one in the selected container to combine them (mix). Once completed the collection of grab samples, cover the receptor container and shake it to homogenize the sample.

14.1.3.6 Pour the sample, using a funnel, in the container labeled and preserve according to the specifications for the parameter.

#### 14.2 Surface Water Sampling in Rivers, Creeks, Channels, and Beaches

14.2.1 Previously, label the sample bottles as described in section 15 of this SOP. In addition, complete the following information of the Chain of Custody, as described in section 15.10 of this SOP: Survey, Sampling Site, Technician (s) name and Initials, Sampling Information (ID, Description, QC, Seq. #, Type, Date), Volume and Quantity, Container, Matrix, and Analysis Requested.

14.2.2 If the station location is unknown, locate the sampling point using a GPS and record it in *Notas de Campo* sheet. If the station is part of a water monitoring network, collect the sample and measurement in the point established in the QAPP for the project.

14.2.3 Proceed to collect the samples.

14.2.4 To collect the samples you may use different instruments or equipment designed for this purpose. The choice of the equipment depends on the characteristics that exist in the sampling point when carrying out the investigation.



- 14.2.5 Determine the equipment needed to collect the samples, such as: Kemmerer or dipper, bucket (stainless steel or plastic), bottles of different sizes and specifications, rope, etc.
- 14.2.6 This sampling equipment can be used in docks, bridges, piers or when you want to establish a uniform distance from the shore to the sampling point in a river, channel, beach or discharge.
- 14.2.7 Whenever you use a dipper, a bucket or other non-disposable sampling equipment, these must be decontaminated before and after each station. Collect an Equipment Blank before collecting samples and in between one station and another, to ensure that the equipment is clean and does not affect the result of the analysis of the samples.
- 14.2.8 Wear gloves and boots. The sampling technician must ensure that the disposable latex gloves are replaced by a new pair after the collection of samples in one station and before the collection of samples in the next station.
- 14.2.9 Record the sampling hour in all field documents (Chain of Custody, Field Inspection Form, etc.).
- 14.2.10 If your investigation includes more than one sampling point in the same segment of a creek, channel or river, start to collect the samples downstream of the water body and continue moving in an upstream direction until all the samples are collected.
- 14.2.11 Enter the water body in front of the wave or in front of the current, as applicable.



14.2.12 In bathing zones (beaches), samples must be collected at knee depth. Nevertheless, in beaches of high-energy environments, collect the samples at ankle depth because it is safer. Document this event in the Chain of Custody.

14.2.13 Proceed to take the data of the field physical parameters required for the investigation (pH, dissolved oxygen, etc.). Refer to the respective SOPs for each parameter. Write down the data in the Field Data Record (See Figure 4 of Appendix 1).

14.2.14 If sampling equipment (bucket, dipper) is needed to collect the samples, before using the equipment, rinse it three times with the water from the point where samples will be collected. Discard the water far from the point where the sample is going to be collected.

14.2.15 Allow the suspended material to precipitate.

14.2.16 Enter the container in the water body at a depth of six (6) inches below the surface, uncover the container and let it fill. Proceed to collect the samples in the following order:

14.2.16.1 Volatile Organic Compounds

14.2.16.2 Semi-Volatile Organic Compounds

14.2.16.3 Pesticides

14.2.16.4 Oil and Grease

14.2.16.5 Metals

14.2.16.6 Mercury

14.2.16.7 Phenols and Cyanides

14.2.16.8 Sulfates and Chlorides

14.2.16.9 Nitrates, Ammonia, TKN



#### 14.2.16.10 Bacteriological Samples

14.2.17 Preserve the samples according to the QAPP and/or Method of the specific project.

14.2.18 Once the bottles are filled, organize and handle them following the procedures and specifications for each parameter established in the QAPP of the project.

14.2.19 Check that the amount of sample containers correspond to the amount documented in the Chain of Custody.

14.2.20 Finish filling the field documentation and the Chain of Custody following the procedure set out in item 15.10 of this SOP.

14.2.21 Transport and deliver the samples to the laboratory that will analyze them.

### 14.3 Surface Water Sampling in Lakes, Reservoirs, Pond, and Sea

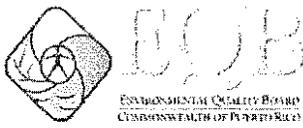
14.3.1 Follow the procedure described in items 14.2.1 through 14.2.5 of this SOP.

14.3.2 Whenever you use a dipper, a bucket or other non-disposable sampling equipment or instrument, these must be decontaminated before and after each station. Collect an Equipment Blank before collecting samples and in between one station and another, to ensure that the equipment is clean and does not affect the result of the analysis of the samples.

14.3.3 Use a boat to get to the sampling point. Always follow the laws and rules of navigation and safety.



- 14.3.4 Slowly move closer to the station, to avoid turbulence. Anchor the boat in the place where you are going to collect the samples and turn off the engine.
- 14.3.5 Wear gloves and proceed to collect the data from field observations. Record the data on the Field Inspection Form and Chain of Custody.
- 14.3.6 Proceed to take the data for the field physical parameters required for the investigation. Refer to the respective SOPs for each parameter. Write down the data in the Field Data Record (See Figure 4 of Appendix 1).
- 14.3.7 Collect samples against the current, so that the boat is not a contaminating factor of the sample.
- 14.3.8 If you need to discard or rinse any container, do it on the side of the boat opposite to the site where the sample is being taken.
- 14.3.9 For reservoirs/lakes, when the station has 10 or more feet deep, take the field physical parameter data in the water column (surface, middle and bottom). Begin collecting the samples in the water column from top to bottom.
- 14.3.10 To take samples, immerse the bottles previously labeled six (6) inches below the surface, following the order established in section 14.2.16 of this SOP.
- 14.3.11 Complete the procedure described in items 14.2.17 to 14.2.21.
- 14.4 Surface Water Sampling at Specific Depths Using a Kemmerer Sampler
- 14.4.1 Set the total depth of the water column of the sampling point. Refer to SOP 118, Standard Operating Procedure for the Determination of Depth in Surface Water.



- 14.4.2 Collect the samples in the water column from top to bottom to avoid disturbing the sediment, which could affect the sample.
- 14.4.3 Leave a distance of 2 to 3 feet from the bottom to collect the sample from the depth of the water column.
- 14.4.4 Separate the rubber seals from both ends of the tube.
- 14.4.5 Rinse the instrument (Kemmerer Sampler) three (3) times with water from the surface.
- 14.4.6 Place the cables of each rubber or polyurethane seal on the pins of the upper part in the handle of the instrument.
- 14.4.7 Close the air and water valves before submerging the instrument.
- 14.4.8 Lower the sampling device to the determined depth. Be guided by the calibrated line of the instrument.
- 14.4.9 Send the weighted messenger down the guide line, closing the sampling device and collecting the water.
- 14.4.10 Raise the Kemmerer and open the valve (yellow screw) to release the pressure and turn out the black valve.
- 14.4.11 Discard (on the side of the boat opposite to the side from where the sample was collected) the first 10-20 mL of water through the drain valve to flush out any potential contamination in the valve.
- 14.4.12 Separate in a calibrated beaker 500 mL of the water collected. Proceed to take the reading of the physical field parameters required for the investigation (pH, dissolved oxygen, etc.). Refer to the specific SOP of each parameter. Write down the information in the Field Data Record form.



14.4.13 Repeat the procedure described in items 14.4.7 to 14.4.12 as many times as necessary to complete the sample collection.

14.4.14 Collect the samples following the order established in item 14.2.16.

14.4.15 Fill the bottles with the water collected in the previously labeled containers.

14.4.16 Complete the procedure described in items 14.2.17 to 14.2.21.

## 15. LABELING AND DOCUMENTATION OF SAMPLE

15.1 Documentation during sampling is extremely important. For this reason, the person that records the information in the corresponding documents must be very careful when making his annotations, and has to maintain the continuity of the information collected.

15.2 Always be sure to verify that the information documented is consistent in all the parts.

15.3 Completely fill the documents using an indelible and waterproof ink, preferably blue.

15.4 Indicate the sections that do not apply by writing N/A in the available space.

15.5 If an error is made during an entry, cross it out, made the corrections (if applicable), and write your initials. Do not use corrective ink or other method of correction.

15.6 To identify the samples, use the official labels from the Water Sampling Division (See Figure 5 of Appendix 1).

15.7 Fill the label in its entirety.

15.8 Use a waterproof marker to prevent the information from being deleted.



- 15.9 Document all samples in the Chain of Custody.
- 15.10 Fill the Chain of Custody in its entirety. Indicate the sections that do not apply by writing N/A in the available space. This document makes you responsible and custodian of all the samples until they are delivered to the laboratory that will perform the required analysis.

15.10.1 The data that should be included in the Chain of Custody consist of:

15.10.1.1 Name of the survey

15.10.1.2 Sampling Site

15.10.1.2.1 May be identified by: the route established in the project, municipality in which the project is located, name of the facilities where the study will take place or the physical address of the site.

15.10.1.3 Sampling technician (s) name and initials who participated in the sampling.

15.10.1.4 ID number of the station.

15.10.1.5 Description of the point where the sample was collected.

15.10.1.5.1 If the point is part of a Project, use the description provided in the QAPP. The description of the sampling stations written in the Chain of Custody and all documents required for the investigation and sampling process must be the same that is detailed in the QAPP of the project.



15.10.1.6 Write down the Quality Control information required:

S = Simple, D = Duplicate, and T = Triplicate.

15.10.1.7 The sequence number corresponding to the order in which samples are collected.

15.10.1.7.1 Samples of quality control corresponding to

blanks must occupy the first numbers of the sequence in the chain of custody. Refer to section 5.0 of this SOP. Identify these samples in the ID column of the Chain of Custody as "LAB", and in the box corresponding to the type description of the station write the type of blank being collected (trip blank, equipment blank or field blank).

15.10.1.8 Type of sample collected, (g) if it is grab, or (c) if it is composite (See section 13.0 of this SOP). Once identified the type of sample, continue filling the information in the line corresponding to the selection.

15.10.1.9 Include the date; writing first the day, then the first three letters of the month and the year.

15.10.1.10 Specify the time at which the sample was collected, indicate if it is am or pm.

15.10.1.11 Write down the temperature that the field parameter's meter read when collecting the sample.



15.10.1.12 Specify the number and volume of the sample containers or bottles used to collect the sample; for example:  
1/100mL = one bottle of 100 milliliter.

15.10.1.13 Identify with a letter (p) if the bottle used is of plastic or (g) if it is of glass.

15.10.1.14 Select, as follows, the number that corresponds to the matrix being sampled during the investigation :

Matrix	Identification Number
Aqueous	1
Sediment	2
Sludge	3
Soil	4

15.10.1.15 Select the preservative required for the sample as follows:

Letter	Preservative
A	Sulfuric Acid
B	Nitric Acid
C	Other

15.10.1.16 The next boxes correspond to the parameters for which samples will be collected. Mark with an (X) the parameter or parameters for which samples are being collected. If a parameter is not in the list, add it in the empty boxes available.

15.10.1.17 In the available space for comments, document relevant information regarding the sample or situations that may have adverse effect on the analysis of the sample. Also,



unforeseen situations that occur when delivering the samples to the laboratory can be documented.

15.10.1.18 Carefully check that everything is in order and without errors before delivering the samples, in order to facilitate the delivery of the samples to the laboratory and avoid situations that may represent a conflict that affects the validity and usability of the samples and their analysis.

15.10.1.19 If samples are handed out to another authorized PREQB employee that will carry them to the laboratory, sign the Chain of Custody in the *Relinquished by* space. The other authorized PREQB employee must sign the Chain of Custody in the *Received by* space and write down the date and time.

15.10.1.20 Deliver the samples to a laboratory employee and verify that all the information regarding the samples being delivered are consistent with the information documented. Clarify at that point any questions regarding the samples. If necessary, document in the comments space any changes or decisions made regarding the samples.

15.10.1.21 The laboratory employee receiving the samples will measure the temperature inside the cooler using the Shipping Container Temperature Blank, and document the measured or recorded temperature in the Chain of Custody.



15.10.1.22 Once everything is verified: sign the Chain of Custody in the *relinquished by* space, write down the date and time of submitting the samples.

15.10.1.23 Upon receiving the samples, the laboratory employee will assign the control number to the Chain of Custody, in which he/she will sign it and will write down the date and time of delivery at the space identified as *Received at the laboratory by*.

15.10.1.24 The transfer of custody between the person that delivers the samples and the one who receive them must maintain continuity. This is confirmed by the signing of the documents and the annotation of the date and time.

15.10.1.25 Hand a copy of the Chain of Custody to the laboratory employee. The original Chain of Custody will be kept by the sampling technician delivering the samples to the laboratory at the Water Sampling Division.

15.10.1.26 In case of delivering samples to a private laboratory, follow the protocols established by the institution that offers the services. Nevertheless, the private laboratory staff must sign the Chain of Custody completed by the PREQB personnel, as evidence that the transfer of the custody of the samples was carried out, and there was continuity in the process.



15.10.2 Completely fill the field inspection and the instruments calibration documents.

15.10.2.1 Indicate the sections that do not apply by writing N/A in the available space.

15.10.3 If additional information is required, refer to the SOPs for each equipment and parameters.

## 16. EQUIPMENT CLEANING PROCEDURE

16.1 All reusable, non-disposable, and non-dedicated equipment/instruments entering in contact with a sample or sampling station, should be thoroughly cleaned before, during and after use. An Equipment Blank must be collected before taking samples for the established parameters. Clean the equipment before collecting the samples using analyte-free water provided by the laboratory. Pour the water used in the rinse into a container equal to that required for the parameter of the sample. Handle this sample in the same way that you manage the rest of the samples of the sampling event. Identify the sample as Equipment Blank and deliver it to the laboratory for its analysis.

16.2 Set an area for storage of the equipment. Care should be taken to avoid placing clean sampling equipment on the ground or other contaminated surfaces.

16.3 Rinse the instrument using potable water.

16.4 Use non-phosphate soap and with the help of a brush, remove the particles.

16.5 Rinse thoroughly with at least five volumes of potable water.



- 16.6 Rinse with at least five volumes of distilled water and let the equipment air dry.
- 16.7 Place the equipment clean and dry in aluminum paper, sterile packaging or clean wrapping. Care should be taken to avoid wrapping the equipment with aluminum foil before it is completely dry.

#### 17. MANAGEMENT OF SOLID WASTE GENERATED IN THE FIELD

- 17.1 All of the liquid and solid waste generated during the sampling should be disposed of adequately and based upon their characteristics and their degree of danger.
- 17.2 Dispose of solid waste that has contact with samples in a labeled container or plastic bag and deliver it to the laboratory to be discarded appropriately.
- 17.3 Once it is determine that the waste generated is non-hazardous, this solid waste will be disposed of in a plastic garbage bag. Discard it in a solid waste container after making sure that the bags are securely fastened.

#### 18. QUALITY CONTROL AND ASSURANCE

- 18.1 The procedures described in the SOP are subject to the auditing of the Quality Assurance and Quality Control Officer.
- 18.2 Except for bacteriological parameters, for every field route, 10% of the samples will be taken as duplicate or triplicate, as applicable. In the case that the route consists of less than 10 stations, a duplicate or triplicate sample will be taken in one station, as applicable.



- 18.3 For the physical parameters, for each field route and sampling day, take a replicate for every 5 readings. In the case that the route consists of less than 5 stations, take the reading of a station as a replicate.
- 18.4 To establish a quality control and verify the quality of the processes of decontamination, sample collection and handling, and equipment handling, it is require that for each field route and sampling day the following additional samples be collected: field blanks, filter blanks, equipment blanks, and trip blanks (QA/QC samples),.
- 18.5 Notice that the above blank samples do not have to be collected in every sampling event, and that the frequency in which these samples will be collected will depend upon the parameters to be analyzed per project, study, or emergency. Follow the specifications in the QAPP of the work.

## 19. REFERENCES

- 19.1 Standard Methods for the Examination of Water and Wastewater. (1998). 20th Edition.
- 19.2 U.S. Environmental Protection Agency, Region 9. Field Sampling Guidance Document #1225.
- 19.3 U.S. Environmental Protection Agency. (9/99). Surface Water Sampling.
- 19.4 U.S. Environmental Protection Agency. (March 2001). Requirements for Quality Assurance Project Plans, QA/R-5.
- 19.5 U.S. Environmental Protection Agency. (2007). Water Quality Assurance, Standard Operating Procedure.



COMMONWEALTH OF PUERTO RICO  
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ENVIRONMENTAL QUALITY BOARD  
WATER QUALITY AREA

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- 19.6 U.S. Geological Survey. (1997). Quality-Control Design for Surface-Water Sampling in the National Water-Quality Assessment Program, Open-File, Report 97-223.
- 19.7 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Laboratory Manual, USEPA, Third Edition. Chapter One.  
<http://www.epa.gov/osw/hazard/testmethods/sw846/online/index.htm>.



## Appendix 1: Figures











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 Urb. San José Industrial Park Ave. Ponce de León 1375 San Juan, PR 00926-2604		Environmental Quality Board Water Sampling Division	Sec. #
Sampling site: _____			
Date:	Time:	By:	
Grab	Comp.	Preservative	
Test Required:			

Figure 5: Label

# pH and Temperature Determination



COMMONWEALTH OF  
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ENVIRONMENTAL QUALITY BOARD

TITLE: STANDARD OPERATING PROCEDURE (SOP) FOR THE DETERMINATION OF THE POTENTIAL OF HYDROGEN (PH) AND TEMPERATURE IN WATER BODIES

SOP NO.: 110

REVISION NO. 1.0

EFFECTIVE DATE: MARCH 27, 2015

AREA/DIVISION: WATER QUALITY AREA/WATER SAMPLING DIVISION

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### Revision History

This table records the revision number, revision date, author and a brief summary of the changes that have been made to the SOP. The revision history log must be completed whenever an alteration to the document is made.

Revision History Log			
Revision No.	Date	Author(s)	Changes
1.0	March 2015	Otto A. Osorio Nieves / Stephenie M. Ayala Flores	<ul style="list-style-type: none"> <li>o Revision History</li> <li>o Item 3.1.2: Inclusion of STORET definition, as well as substitution of the Evaluation Strategic Planning Area for Water Quality Area.</li> <li>o Figures 1, 2, and 3</li> <li>o Modification of the SOP format as part of the Quality Assurance and Quality Control Office effort to standardize all PREQB SOP documents.</li> </ul>



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## 1. SOP TITLE

Standard Operating Procedure (SOP) for the Determination of the Potential of Hydrogen (pH) and Temperature

## 2. APPLICABILITY

2.1. This SOP applies to the determination of the potential of hydrogen (pH) and temperature in surface, estuarine, and coastal waters, as well as groundwater, as defined in the Puerto Rico Water Quality Standards Regulation (PRWQSR).

## 3. PURPOSE

3.1. The duty of the Puerto Rico Environmental Quality Board (PREQB) - Water Sampling Division (WSD) is to collect water samples to be analyzed for different parameters in order to determine the quality of the water bodies; to assess any impact of point and nonpoint pollution sources in these water bodies, and to detect any violation to the PRWQSR.

3.1.1 This SOP is written primarily as specific instructions for the WSD technical personnel, to facilitate their investigations and the generation of the necessary environmental data, as well as the supporting documentation for the determination of pH and temperature.



3.1.2 Data collected will be included in the biannual report of the 305(b) project or any other required environmental report. In addition, this data will be uploaded into the Environmental Protection Agency (EPA) Storage Retrieval (STORET) Data Warehouse, and kept at the Plans and Special Projects Division (PSPD); so it can be available for the use of the general public and any state, local or federal government agency.

3.1.3 In addition, the data collected may be used as reference by laboratories, students, or any other person interested in the water quality of any surface water bodies.

#### 4. METHOD SUMMARY

- 4.1 The potential of hydrogen in a water sample indicates its tendency to accept or give hydrogen ions in a scale from 0 (very acid) to 14 (very basic).
- 4.2 Temperature affects many aspects of an ecosystem, so it is a vital component for the survival of organisms such as: insects, fish, and bacteria. In addition, it affects the saturation level of oxygen in water.
- 4.3 To obtain the data, a meter with a probe (glass electrode) is used.
- 4.4 The equipment is calibrated.
- 4.5 The probe is introduced into the water body or into an analysis container, and then, the pH and temperature values obtained are read. The reading unit for temperature is degree Celsius (°C).



## 5. DEFINITIONS

- 5.1 Surface Water – Any natural or artificial water source including all streams, lakes, reservoirs, inland watercourse and waterways, irrigation systems, drainage systems, intermittent streams and other inland water bodies or accumulated waters.
- 5.2 Ground Waters – Sub-surface waters present at or beneath the water table, including waters in caves and caverns when the presence of water results from the manifestation of the characteristics of the saturated zone beneath the water table.
- 5.3 Estuarine Waters – That portion of the mouth or lower course of a river, stream, canal or lagoon, in which the fresh water meets the sea water and is subject to the ebb and flow of the tides.
- 5.4 Coastal Waters – Marine waters within the jurisdiction of the United States of America and the Commonwealth of Puerto Rico, as established by Article 8 of the *Puerto Rico Federal Relations Act of 1917*, as amended, and shore waters which are subject to the ebb and flow of the tides.
- 5.5 pH (hydrogen potential) - Is a measure of the acidity and alkalinity of a solution. The pH indicates the concentration of the hydronium ions ( $H_3O^+$ ) present in a substance. The acronym stands for “potential of hydrogen”; from the Latin terms: *pondus hydrogenii* or *potentia hydrogenii*. This term was introduced by the Danish chemist Sørensen, who defined it as the minus decimal logarithm of the hydrogen ion activity in a solution, namely:

$$pH = -\log_{10} [a_{H_3O^+}]$$



5.6 Quality Assurance Project Plan (QAPP) – Document that describes in detail the technical activities to be implemented to assure that the results satisfy the established execution criteria.

## 6. HEALTH AND SAFETY CONSIDERATIONS

6.1 Whenever performing the calibration, calibration verification, or reading process, use disposable latex gloves and safety spectacles.

6.2 Always follow the universal safety measures.

## 7. CAUTION MEASURES

7.1 Be careful when introducing the probe into the sample. In some models, the probe is not sealed and water could enter the system, damaging it. Follow the manufacturer's recommendations.

7.2 Always protect the equipment from bumps and falls. Use the protective case to transport the equipment.

7.3 Always rinse carefully the probe, and dry it before and after using it.

7.4 Avoid exposing the meter and the buffers to high temperatures.

7.5 Verify the conditions of the batteries before using the equipment.

7.6 If it applies, make sure that the electrode probe is filled with gelatin, without bubbles.



## 8. INTERFERENCES

- 8.1 Before sampling, rinse three times the container that is going to be used with the water of the point to be sampled. Then, fill the container with the necessary volume of liquid to take the pH and temperature readings.
- 8.2 Verify the expiration date of the buffers.
- 8.3 If a container is used to collect the sample, the readings should be taken within the first 15 minutes from the time the container was filled with the sample.

## 9. STAFF TRAINING

- 9.1 It is required that the technical staff has the HAZWOPER course updated.
- 9.2 The technical staff must have knowledge on the equipment operation manual, its management and specifications.
- 9.3 All technical staff must have a hands-on training using the equipment, before using it for the first time.

## 10. MATERIALS AND EQUIPMENT

- 10.1 The following list includes the necessary equipment and materials for taking the pH and temperature readings:
  - 10.1.1 Deionized or distilled water
  - 10.1.2 Potable water
  - 10.1.3 Absorbent paper (*Kimwipes*)



- 10.1.4 Buffers, namely pH 4.00, pH 7.00, and pH 10.00
- 10.1.5 Disposable latex gloves
- 10.1.6 Extra batteries
- 10.1.7 pH meter
- 10.1.8 Container (plastic or glass) of 250 or more milliliters (mL)
- 10.1.9 Chain of Custody
- 10.1.10 Field pH Meter Calibration Verification Form
- 10.1.11 Field Data Record Form

## 11. PROCEDURE

### 11.1. Before Taking the Readings

#### 11.1.1 Standard calibration for the Hach HQ30d and HQ40d meters

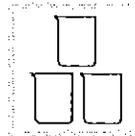
- 11.1.1.1 Power on the equipment
- 11.1.1.2 Ensure that the temperature measuring unit is Celsius (°C).
- 11.1.1.3 Push the  key and select the Celsius option using the UP and Down keys.
- 11.1.1.4 Press Exit to return to measurement mode.
- 11.1.1.5 Connect the probe to the meter. Make sure the lock nut of the cable is plugged firmly into the meter. Turn on the meter.



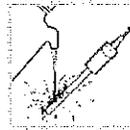
- 11.1.1.6 When two probes are connected (HQ40d model), push the UP or DOWN keys to change to the single display mode in order to show the Calibrate option.
- 11.1.1.7 Press Calibrate. The display shows the buffers that are required for calibration.



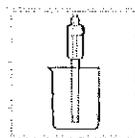
- 11.1.1.8 Prepare the fresh buffers in separate beakers or other suitable containers.



- 11.1.1.9 Rinse the probe with deionized water. Blot dry with a lint-free cloth.



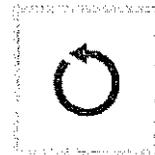
- 11.1.1.10 Put the probe in the pH buffer solution and stir gently. Make sure the reference junctions are completely submerged. Shake the probe from side to side in the standard solution for refreshing the reference junction.



11.1.1.11 Push Read. The display will show “Stabilizing” and a progress bar as the probe is stabilized in the standard. The display shows the buffer that has just been read and shows the temperature corrected pH value when the reading is stable.



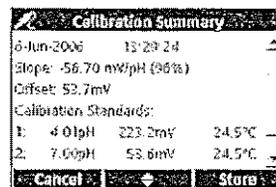
11.1.1.12 Repeat steps 11.1.1.8 through 11.1.1.10 until the minimum number of calibration points specified in the current method have been collected.



11.1.1.13 Push Done to view the calibrating summary. The display will not show Done, until the minimum number of calibration points have been collected.



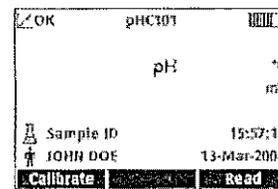
11.1.1.14 The Calibration Summary will appear.



11.1.1.15 Press Store to accept the calibration and go back to measurement mode.



11.1.1.16 When the calibration is successful, the display will show OK in the upper left corner.



11.1.2 Reviewing Hach HQ30d and HQ40d meters calibration

11.1.2.1 Make sure that the probe is connected.

11.1.2.2 To see current calibration, press the Data Log key.

11.1.2.3 Use the UP and DOWN keys to highlight View Probe Data.

11.1.2.4 Press the GREEN/RIGHT key under Select.

11.1.2.5 If two probes are connected (HQ40d only), the connected probes will be displayed. Use the UP and DOWN keys to highlight the probe. Press the GREEN/RIGHT key under Select.

11.1.2.6 Using the keys, highlight Current Calibration.

11.1.2.7 Press the GREEN/RIGHT key under Select.

11.1.2.8 The meter display shows the calibration details for the reading. Record the calibration readings, temperature, difference, and calibration slope information in the pH Meter Calibration Verification Form (See Figure 1 of Appendix 1), as well as the date, time and other information required.

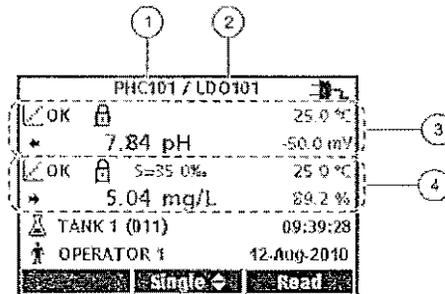
11.1.2.9 Press Exit until reaching the main menu.

## 11.2. pH Reading Using Hach HQ30d and HQ40d

11.2.1. Hach HQ30d and HQ40d meters are designed to work in the field, therefore, it can be used to take readings directly from the water body. Since the probe of these equipment is sealed this will not cause damage to it.

11.2.2. When two probes are connected to the HQ40d meter, the display can either show the reading of just one probe or the one from both of them simultaneously.

11.2.3. To change the screen mode to single or dual screen, use the UP and DOWN keys. In dual screen mode, the UP key will select the probe on the left and the DOWN key will select the probe on the right.



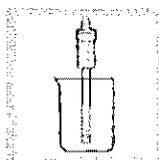
1. Probe that is connected to port on left
2. Probe that is connected to port on right
3. Measurement information for probe on the left
4. Measurement information for probe on the right

11.2.4. If necessary, fill a container of no less than 250 mL (plastic or glass) with water of the water body to be sampled.

11.2.5. Clean the probe with distilled water. Use absorbent paper (*Kimwipes*) to gently blot the probe.

11.2.6. Insert the probe into the sample or directly in the water body to be sampled and stir gently. Make sure that the reference junctions are

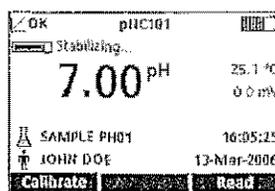
completely submerged. Do not put the probe on the bottom or sides of the container. Shake the probe from side to side in the sample to refresh the reference junction.



11.2.7. Push Read. The display will show "Stabilizing" and a progress bar as the probe stabilizes in the sample. The display will show the lock icon when the reading(s) stabilizes. If another measurement is needed, repeat steps 11.2.6 through 11.2.7.



11.2.8. Once stabilized the given value, the display will show detailed information regarding the reading.



11.2.9. Record the pH and temperature readings obtained, as well as date, time and other information required, in the Chain of Custody and Field Data Record Form (See Figures 2 and 3 of Appendix 1).

## 12. SAMPLE HANDLING

12.1 If you are taking the readings directly from the water body, do it right away.



12.2 If a sample is taken in a container, the readings should be taken within a period no longer than 15 minutes.

### 13. DATA AND DOCUMENTATION

13.1 Documentation during sampling is extremely important. For this reason, the person that records the information must be very careful and has to establish continuity of the information collected.

13.2 Always be sure to verify that the information is consistent in all parts.

13.3 Complete the documents in their entirety and in the sections that do not apply write N/A in the available space.

13.4 If a mistake is made during the entry of the information into the documents, cross out it with a single line, make the correction (if necessary), and write your initials on it. Do not use corrective ink or other method of correction.

13.5 Document all readings in the field documents.

### 14. EQUIPMENT CLEANING PROCEDURE AND STORAGE

#### 14.1 Regular use

14.1.1 All the equipment that are in contact with a sample or a sampling station must be thoroughly cleaned before and after use.

14.1.2 Rinse thoroughly with potable water and finish with distilled water and let the equipment air dry.

14.1.3 Place the equipment clean and dry in its protective case.



## 14.2 Special circumstances

### 14.2.1 Clean the probe when:

14.2.1.1 Drifting/inaccurate readings occur as a result of contamination on the glass sensor the probe being left dry for extended periods of time.

14.2.1.2 Slow stabilization time occurs as a result of contamination on the glass sensor.

14.2.1.3 A calibration error occurs as a result of contamination on the glass sensor.

14.2.1.4 The type of contamination will determine the cleaning solution needed

#### 14.2.1.4.1 General contaminants

14.2.1.4.1.1 Rinse the probe with deionized water and blot dry with a lint-free cloth.

14.2.1.4.1.2 Soak the glass bulb for 12 to 16 hours in Hach Electrode Cleaning Solution.

14.2.1.4.1.3 Rinse or soak the probe for 1 minute in deionized water.

14.2.1.4.1.4 Soak the probe in pH 4 buffer for up to 20 minutes, then rinse with deionized water.

14.2.1.4.1.5 Blot dry with a lint-free cloth.



14.2.1.4.2 For mineral deposits

14.2.1.4.2.1 Rinse the probe with deionized water and blot dry with a lint-free cloth.

14.2.1.4.2.2 Soak the glass bulb for 10 to 15 minutes in 0.1 M HCl.

14.2.1.4.2.3 Rinse or soak the probe for one minute in deionized water.

14.2.1.4.2.4 Soak the probe in pH 4 buffer for up to 20 minutes. Rinse with deionized water.

14.2.1.4.2.5 Blot dry with a lint-free cloth.

14.2.1.4.3 For fats, grease and oils

14.2.1.4.3.1 Soak the glass bulb in a warm detergent solution for up to 2 hours.

14.2.1.4.3.2 Rinse or soak the probe for 1 minute in deionized water.

14.2.1.4.3.3 Soak the probe in pH 4 buffer for up to 20 minutes, then rinse with deionized water.

14.2.1.4.3.4 Blot dry with a lint-free cloth.

14.3 Short-term and long-term storage



- 14.3.1 For the best probe performance, do not let the reference junction become dry.
  - 14.3.2 Rinse the probe with deionized water. Dry the probe with a lint-free cloth.
  - 14.3.3 Fill the probe storage cap or soaker bottle half full with Hach Electrode Storage Solution or 3 M potassium chloride (KCl) solution.
  - 14.3.4 Put the soaker bottle on the probe and tighten the soaker bottle cap.
  - 14.3.5 Make sure that the solution in the storage cap or soaker bottle completely covers the glass bulb and reference junction.
- 14.4 If the glass bulb becomes dry, rehydrate it.
- 14.4.1 Soak the probe tip in the 4.01, 7.00 and 10.01 buffers each for 5 minutes.
  - 14.4.2 Rinse the probe with deionized water. Blot dry with a lint-free cloth.
  - 14.4.3 Calibrate the probe.

#### 14 QUALITY ASSURANCE AND QUALITY CONTROL

- 15.1 The procedures described in the SOP are subject to be audited by the Quality Assurance and Quality Control Office.
- 15.2 For the pH and temperature parameters, for every 5 readings, take a replicate. In the case that the route consists of less than 5 stations, take the reading of a station as a replicate.
- 15.3 To validate the performance of the equipment before commencing the sampling route, perform a calibration check following the steps set forth in



item 11.1. Include the information in the Field pH Meter Calibration form (See Figure 1 of Appendix 1).

15.4 To validate the performance of the equipment after completing the sampling route, carry out a final calibration check using a buffer and following the steps set forth in item 11.2. Include the information in the Field pH Meter Calibration form (See Figure 1 of Appendix 1).

## 16 REFERENCES

- 16.1 Hach. (2010). Basic User Manual (HQ40d, HQ30d, HQ14d, HQ11d). Edition 2. DOC022.97.80017.
- 16.2 Hach. (2011). User Manual, General Use pH Probe: Models PH20101, PH20103. Edition 1. DOC022.52.80197.
- 16.3 U.S. Environmental Protection Agency, Region 9. Field Sampling Guidance Document #1225.
- 16.4 U.S. Environmental Protection Agency. (March 2001). Requirements for Quality Assurance Project Plans, QA/R-5.
- 16.5 U.S. Environmental Protection Agency. (2007). Water Quality Assurance, Standard Operating Procedure.



# Appendix 1: Figures



COMMONWEALTH OF PUERTO RICO  
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ENVIRONMENTAL QUALITY BOARD  
WATER QUALITY AREA

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COMMONWEALTH OF PUERTO RICO  
OFFICE OF THE GOVERNOR  
ENVIRONMENTAL QUALITY BOARD

Water Sampling Division

**pH METER CALIBRATION VERIFICATION**

PROJECT:	DATE:	TIME:
	pH METER MODEL:	CAL. EXP. DATE:
ROUTE:	ID NUMBER:	S/N:

**FIELD CALIBRATION VERIFICATION**

BUFFER	LOT NUMBER	EXPIRATION DATE	INST. READING	TEMP (°C)	DIFFERENCE ±0.10

DIFFERENCE = (INST. READING - BUFFER)

**INTERNAL CALIBRATION SLOPE**

HQ30d: \_\_\_\_\_ (-59 mV/pH, 85-115% at 25°C)

HQ40d: \_\_\_\_\_ (-59 mV/pH, 85-115% at 25°C)

**FINAL VERIFICATION**

TIME: \_\_\_\_\_

BUFFER	INST. READING	TEMP (°C)	DIFFERENCE ±0.10

DIFFERENCE = (INST. READING - BUFFER)

FIELD TECHNICIAN: \_\_\_\_\_ DATE (MM-DD-YY): \_\_\_\_\_

QA/QC APPROVAL: \_\_\_\_\_ DATE (MM-DD-YY): \_\_\_\_\_

COMMENTS: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

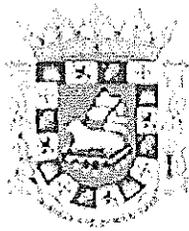
Environmental Agencies Building Cruz A. Matos  
1375 Ponce de León Avenue, San Juan, PR 00926-2604  
P.O. Box 11488, Santurce, PR 00910  
Tel. 787-767-8181 Fax 787-767-2592

Figure 1: pH Meter Calibration Verification Form





# Appendix 5: Field Sampling and Laboratory Data Validation SOP



ESTADO LIBRE ASOCIADO DE  
P U E R T O R I C O  
JUNTA DE CALIDAD AMBIENTAL

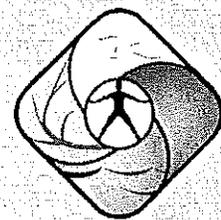
SOP#: 132

Rev.#: 0

Date: August 2013

## Standard Operating Procedure (SOP)

ESTADO LIBRE ASOCIADO DE PUERTO RICO  
JUNTA DE CALIDAD AMBIENTAL



Prepared by:

Water Quality Area

### Approval Page

Standard Operating Procedure (SOP)  
Field Sampling and Laboratory Data Validation  
SOP Title

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Prepared by:

*Marisol Marrero*  
Marisol Marrero

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Quality Control and Assurance Specialist, Water Quality Area

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Date

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Date

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Position

*August 16, 2013*

Date



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## Standard Operating Procedure (SOP)

### 1. SOP TITLE

Standard Operating Procedure for the Field Sampling and Laboratory Data Validation

### 2. APPLICABILITY

2.1 This Standard Operating Procedure (SOP) applies to the field data generated by the Environmental Quality Board (EQB) Sampling Division and the organic and inorganic data generated by the EQB Environmental Laboratory.

### 3. PURPOSE

3.1 The Water Quality Area (WQA) has to ensure that all the environmental decisions are based on data of known quality. The purpose of this SOP is to establish the tools, which can be employed to assess the quality of reported field and laboratory data. It provides a technical judgment on the validity of the laboratory results as a first step in determining their overall usability and legal defensibility.

### 4. METHOD SUMMARY

4.1 This SOP establishes the requirements for the validation of the generated data. This validation involves the data from the field and from the laboratory. Basically, the validation is performed by the use of checklists. These checklists are completed for the field phase and for the laboratory data generated in the different projects of the WQA Sampling Division. If exist specific information not covered by the checklists, the reviewer makes their evaluation based on the analytical method, SOP, or any applicable guidance. Before data validation, the reviewer needs to acquire project knowledge, verify the sampling procedures, determine the purpose of the validation, and the data available for the data validation. During the technical evaluation, the reviewer uses a checklist to apply evaluation criteria's established by this SOP. Finally, the reviewer prepares a report with the comments, corrective actions or recommendations of the evaluated data.



## 5. DEFINITIONS

- 5.1 Data Validation – the first step in assessing data quality. It is a standardized review process for judging the analytical quality and usefulness of a discrete set of chemical data. Thus, data validation identifies the "analytical error" associated with a data set. Data validation can also identify some (e.g., incorrect preservation techniques), but not all of the "sampling error" associated with a data set. Data validation can be viewed as a decision making process during which established quality control criteria are applied to the data.
- 5.2 Quality Assurance Project Plan (QAPP) – The document that describes in detail the technical activities to be implemented to ensure that the results satisfy the established performance criteria.
- 5.3 Limit of Detection (LOD)- The lower concentration that can be determined to be statistically different from a blank. Measure the ability of the test procedure to generate a positive response and have nothing to do with the accuracy of that response.
- 5.4 Matrix Spike (MS)- A measured amount of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to assess the bias of a method in a given sample matrix.
- 5.5 Matrix Spike Duplicate (MSD)- Intra-laboratory (within the same laboratory) split samples spiked with identical concentrations of target analyte(s). MSD are used to assess the precision and bias of a method in a given sample matrix.
- 5.6 Relative Percent Difference (RPD)- Is a measure of precision. It is defined as the difference between the two measurements divided by the average of the two measurements.
- 5.7 Recovery Percent (%R)- Is the quantitative measure of accuracy. It is calculated as the difference between the spiked sample amount and the unspiked sample result divided by the spike amount.

## 6. HEALTH AND SAFETY CONSIDERATIONS

- 6.1 Not applicable.

## 7. PRECAUTION MEASURES

- 7.1 Not applicable.



## 8. SPECIAL CONSIDERATIONS

8.1 The Sampling Division of the EQB has as one of their functions to work with different projects like: beaches, lakes, bathing zones, coasts and groundwater, among others. Each one of these projects establishes specific field and analytical parameters, some of these parameters are repeated across projects and some are specific for a particular project. This SOP is not intended to be used for a particular project, or for a particular parameter, it was designed to cover the general requirements. The evaluator that performs the validation has to be related with the analytical method or the method SOP to determine the applicability to their project and evaluate it in accordance with that.

## 9. STAFF TRAINING

9.1 For performing data validation, it is necessary a background in science and strong knowledge on laboratory and analytical procedures. The person has to be capable of understanding the analytical methods and interpret the analytical data. It is essential that the person be related with quality concepts. Also, is better if the person has a previous experience working at an analytical laboratory.

## 10. EQUIPMENT AND MATERIALS

10.1 Not applicable.

## 11. PROCEDURE

### 11.1 Pre-Evaluation

#### 11.1.1 Project Knowledge

11.1.1.1 It is necessary to comprehend the background of the project before any evaluation. As mentioned before, the Sampling Division of the WQA works with different projects (beaches, lakes, bathing zones, coasts and groundwater, etc.) and each project have an approved QAPP. It is necessary that the reviewer be familiar with the specific requirements of the QAPP under evaluation in order to verify that they comply with: field and analytical parameters, sampling frequency, sampling stations and quality control samples, among others.



## 11.1.2 Data Availability

11.1.2.1 The reviewer has to determine if the data provided from the field sampling phase and the analytical phase is adequate to perform the data validation. If the information is not available, the reviewer has to analyze how essential is the missing information and select one of the following alternatives:

11.1.2.1.1 Begin the evaluation and require the missing information.

11.1.2.1.2 Does not begin the evaluation until the information is provided.

11.1.2.1.3 Does not require the information due that the reviewer understands that is not essential for the evaluation.

11.1.2.1.4 Begin the evaluation and not require the information due that the data has other deficiencies.

11.1.2.1.5 Perform the evaluation with the available information and specify the deficiencies in the report in order to initiate corrective action process.

11.1.2.1.5.1 With the purpose of complete an evaluation, the missing information can be required by the reviewer to the involved parties. The missing information can be obtained by electronic mail, telephone, fax, internal memo, through a letter or a meeting depending on the reviewer criterion.

## 11.2 Technical Evaluation

### 11.2.1 General Field Sampling Validation

11.2.1.1 To perform the validation of this phase it is necessary that the reviewer has available all the field documentation generated during the sampling activities. After that, the reviewer uses the checklist, named *General Sampling Validation Checklist* (Appendix 1) to make the validation. The field documentation that must be included to perform the validation of this phase are :

11.2.1.1.1 Chain of Custody (COC)

11.2.1.1.2 Field Data Record

11.2.1.1.3 Field Inspection Form

11.2.1.1.4 Calibration Forms



- 11.2.1.1.5 Additional Field Documentation- considering that the Sampling Division works with different projects, if one of these projects generates additional information not mentioned at 11.2.1.1.1 to 11.2.1.1.4, it will be evaluated under this part. The checklist provides for the identification of the additional documentation and their respective evaluation.
- 11.2.1.2 Once the reviewer finishes with the validation of the field sampling data and completes the checklist included at the Appendix 1, the reviewer using their professional judgment will determine one of the following actions:
  - 11.2.1.2.1 Accept the field sampling phase due that no deficiencies were found.
  - 11.2.1.2.2 Accept the field sampling phase, since the deficiencies found does not compromise the quality of the data.
  - 11.2.1.2.3 Require a corrective action, and does not approve the field data until the deficiencies are corrected.
  - 11.2.1.2.4 Does not accept the sampling phase due to gross errors that compromise the quality of the data.
  - 11.2.1.2.5 Does not accept the sampling phase and require a re-sample.
- 11.2.1.3 After the validation of the field sampling data, the reviewer will perform the validation of the analytical data from the documentation provided by the laboratory. It is recommended that validates first the field data and later the laboratory data, the results of both validations are included in a report. The reviewer can generate a report of each validation phase if it is necessary or required for any particularity of the project.
  - 11.2.1.3.1 If the field sampling data is not accepted due to deficiencies that compromise the quality of the data, the reviewer has to communicate with the General Project Manager for determine, according with the case, if proceed with the analytical data validation, if re-sampling is necessary or determine any other action.



## 11.2.2 Analytical Data Validation

The reviewer uses the checklist named *General Analytical Data Validation Checklist* (Appendix 2) for the validation of the documentation provided by the laboratory.

### 11.2.2.1 General Requirements<sup>1</sup>

11.2.2.1.1 Case narrative

11.2.2.1.2 Chain of Custody (COC)

11.2.2.1.3 Approved QAPP

11.2.2.1.4 Sampling results table

11.2.2.1.5 Laboratory Data Report

11.2.2.1.5.1 Recovery Percent (%R) and Relative Percent Difference (RPD).

11.2.2.1.5.1.1 Field blanks- field duplicate, field blank, trip blank and equipment blank.

11.2.2.1.5.1.2 Laboratory blanks- method blank, matrix spike, Laboratory Control Sample (LCS), surrogates (only for organics), Interference check sample (if apply- inorganics), serial dilutions (if apply- inorganics).

11.2.2.1.5.2 Holding time

11.2.2.1.5.3 Calibration

11.2.2.1.5.3.1 Calibration Tuning and Calibration Verification.

11.2.2.1.5.4 Raw data

11.2.2.1.5.4.1 Chromatograms and quantitation reports, Absorbance (if apply - inorganics), sample preparation, etc.

### 11.2.2.2 Technical Evaluation

11.2.2.2.1 Case narrative

<sup>1</sup> Depending on the parameters and methods under consideration, some of these requirements may vary.



11.2.2.2.1.1 It is a source of information. Problems with matrixes, insufficient sample volume, samples out of control, broken containers and unusual events must be documented in the narrative. If not included does not invalidate the data, but difficult the evaluation since the reviewer will not have the input of the laboratory. The reviewer has to perform the data validation and make the observation at the final report.

11.2.2.2.2 Chain of Custody (COC)

11.2.2.2.2.1 The COC must be submitted with the analytical results, if not submitted it must be required. At this point, the COC may not be a concern, since it was evaluated during the field sampling validation phase. However, if exist any problem that could not be resolved previously, the reviewer has to use their professional judgment to determine the utility of the data. It will be make on a case to case basis and depending of the complexity of each situation.

11.2.2.2.3 Accordance with the approved QAPP

11.2.2.2.3.1 If the data does not follow the procedures in accordance with the QAPP or approved SOP, it is necessary that the reviewer analyze the changes to oversee if accomplish with the project objectives and can be used. However, the reviewer has to make the deviation comments from the approved Plan. If the changes do not meet the objectives of the project, the data has to be rejected (unusable).

11.2.2.2.4 Recovery Percent (%R)- matrix spike (MS )

11.2.2.2.4.1 The matrix spike provides information of the matrix effects in the sample



preparation procedures and the method measurement. If the recovery of the matrix spike is within the limits specified in the method, then the method is judged to be applicable to that sample matrix. If not, poor recoveries could indicate that either the analytical method does not perform well on that particular sample matrix (due to interfering substances present in the sample), or that the laboratory is performing the method poorly (analytical process out of control). The reviewer has to follow any of the following options: not to take any action based on MS/MSD alone, verify if the laboratory made any corrective action, use their professional judgment along with other QA/QC criteria's or verify the different EPA guidance's and determine how the data can be qualified and use the data in accordance with that qualification.

#### 11.2.2.2.5 Relative Percent Difference (RPD)

11.2.2.2.5.1 If the RPD is not within control limits, it could indicate a non-homogeneous matrix, poor laboratory technique, or that the method does not perform well on the matrix. In any case, poor precision casts doubts on all of the analysis because you cannot be certain that the data are reproducible. In these cases, the reviewer has to verify if the laboratory made any corrective action or verify the different EPA guidance's and determine how the data can be qualified and use the data in accordance with that qualification.

#### 11.2.2.2.6 Surrogates



11.2.2.2.6.1 Surrogate recovery is a quality control measure limited to use in organic analysis. Surrogates are compounds added to every sample at the beginning of the sample preparation on an individual sample basis. The surrogate compounds should be infrequently found as background in the samples and are selected based on their ability to mimic the behavior of specific target analytes held to be particularly sensitive to the sample preparation manipulations. Surrogate recoveries are often expressed as percent recovery (%R), although some methods have established surrogate recovery acceptance criteria that are part of the method, for most part acceptable surrogate recoveries need to be determined by the laboratory. If surrogate recoveries are below the acceptance limits, the efficiency of the extraction and analysis may be subject to question. However, recoveries above the acceptance limits may indicate spiking errors and calculation errors. When this situation occurs, the reviewer has to verify the different EPA guidance's and determine how the data can be qualified and use the data in accordance with that qualification.

#### 11.2.2.2.7 Field blanks

11.2.2.2.7.1 If the blanks are not performed as part of the sampling activity, the data has to be considered as *an approximate concentration of the analyte* and the reviewer must use professional judgment to determine the reliability of the data.



#### 11.2.2.2.8 Method blank

11.2.2.2.8.1 Method blanks are also referred to as preparation blanks and batch blanks. They consist of a portion of analyte-free water or solid of the same size as that use for the routine sample preparation. This blank is taken through the entire sample processing procedure just as if it were a regular sample and serve as a monitoring control for a variety of possible sources of contamination in the laboratory. There is little need for concern, if a contaminant is present in a blank, but not an analyte of concern for the project or if a contaminant is present in a blank, but not present in a sample. If the acceptance criteria are not attained, the reviewer can inquire to the laboratory regarding the action (if any) that they performed to correct the problem. If does not exist evidence of reanalysis or corrective action performed by the laboratory, the reviewer can verify the different EPA guidance's and determine how the data can be qualified and use the data in accordance with that qualification. It is important to notice that sample results are never corrected for contamination found in any blank.

#### 11.2.2.2.9 Laboratoy Control Sample (LCS)

11.2.2.2.9.1 Laboratory control samples (LCS) are also known as quality control samples. LCS consists of a portion of analyte free water or solid phase sample that is spiked with target analytes at a known concentration. The LCS is processed through the entire method procedure and the results examined for target analyte recovery. The cases in which



the LCS and LCSD fail to generate acceptable results, is cause of concern regarding the validity of the results for all samples in the batch. Inability to obtain acceptable results for the perfect sample is directly related to an inability to generate acceptable results for any sample. The LCS and LCSD results are frequently used as a fall-back position by the laboratory in cases where MS/MDS have failed to achieve acceptable recovery or precision. The interpretation is that the sample gave poor results due to matrix interference; however, the LCS gave good results, which indicates that the analytical system in the laboratory was in good operational condition.

#### 11.2.2.2.10 Holding time

11.2.2.2.10.1 It can be notice the existence of many holding times, preservation and container tables which are not uniform. For this reason, the sample holding times are reviewed relative to the specifications of the approved QAPP. The reviewer must use professional judgment to determine the reliability of the data and the effects of additional storage. If technical holding times are exceeded, the data associated has to be considered as an approximate concentration of the analyte. In these cases, the reviewer has to make the observation at the report. The data will be rejected if the holding time are grossly exceeded (greater than two times the required time for the compounds) or in accordance with the reviewer determination.



#### 11.2.2.2.11 Calibration

11.2.2.2.11.1 In the calibration, different factors have to be considered depending of the method and if the analysis is from organic or inorganic compounds. In general, the method calibration has to accomplish with the required check compounds, blanks, %RSD (based on response factors) or correlation coefficient (based on square regression), calibration standards, etc. It is necessary to accomplish with all the method requirements for calibration, if not, most of the methods require a re-calibration. If a re-calibration is not presented and some calibration requirements are not attained, the data is rejected.

#### 11.2.2.2.12 Calibration tuning

11.2.2.2.12.1 Some of the methods have tuning requirements like tune criteria's and a timeframe; these requirements must be accomplished before of the calibration. If some of these method requirements are not accomplished, additional information must be required and the data can be rejected.

#### 11.2.2.2.13 Calibration Verification

11.2.2.2.13.1 The calibration verification involves the Initial Calibration Verification (ICV) and the Continuing Calibration Verification (CCV). The ICV, check the accuracy of the calibration curve immediately after it is prepared and the CCV, periodically check the calibration curve to determine if it is still reliable. Specifications for calibration verification are generally given as a percentage difference (deviation) from the test



### 13. QUALITY CONTROL AND ACCURACY

- 13.1 It is necessary to have an approved QAPP in order to perform a data validation. However, if an approved plan does not exist and an evaluation is required, the reviewer will evaluate the data (if possible) and at the report it will be included as a finding. With the purpose of performing a complete evaluation, the reviewer can request any information that considers necessary.

### 14. REFERENCES

- 14.1 Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality Related Documents. EPA QA/G-6; November 1995.
- 14.2 Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality Related Documents. EPA QA/G-6; April 2007
- 14.3 Berger W., McCarty H., Smith R. Environmental Laboratory Data Evaluation. Apichemical Consultants, Douglasville, GA; 1996.
- 14.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846). Laboratory Manual, USEPA, Third Edition; December 1996.
- 14.5 USEPA Contract Laboratory Program, National Functional Guidance for Inorganic Data Review. Office of Emergency and Remedial Response, EPA 540/R-94/013; February 1994.
- 14.6 USEPA Contract Laboratory Program, National Functional Guidance for Organic Data Review. Office of Emergency and Remedial Response, EPA 540/R-94/012; February 1994.
- 14.7 Laboratory Documentation, Requirements for Data Validation (Draft). Quality Assurance Program USEPA Region 9, San Francisco, California; 9QA-07-97; July 1997.



# Appendix 1: General Sampling Validation Checklist





## Water Quality Area

### General Sampling Validation Checklist

Project Title: \_\_\_\_\_

Route: \_\_\_\_\_

Sampling Date: \_\_\_\_\_

QAPP Title: \_\_\_\_\_ Approval date: \_\_\_\_\_

Date: Initial Eval. \_\_\_\_\_ Final Eval. \_\_\_\_\_ Reviewer: \_\_\_\_\_

I. General Information

1. The validation report applies to a total of \_\_\_\_\_ samples.
2. Sample Information:

KIND OF SAMPLES (Mark with an "x")			
Sample Id.	Sample (station)	QA/QC sample	Comments





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KIND OF SAMPLES (Mark with an "x")			
Sample Id.	Sample (station)	QA/QC sample	Comments

3. Field Parameters under Evaluation (Mark with an "x"):

Parameter	Under Evaluation (X)
Temperature	
Depth	
Transparency	
Salinity	
Conductivity	
pH	
Dissolved Oxygen	
Turbidity	
Settable Solids	





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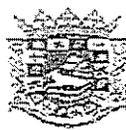
Parameter	Under Evaluation (X)
Residual Chlorine	
Other (Specify: _____)	

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

4. Analytical Parameters (Mark with an "x"):

Parameter	Under Evaluation (X)
Alkalinity	
BOD	
Boron	
Chloride	
Chlorophyll "a"	
COD	
Cyanide	
Dissolved Oxygen	
<i>Enterococcus</i>	
Fecal Coliforms	





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Parameter	Under Evaluation (X)
Fluoride	
Hardness	
Mercury	
Metals	
NH <sub>3</sub> -N	
NO <sub>2</sub> -N	
NO <sub>3</sub> -N	
(NO <sub>3</sub> + NO <sub>2</sub> )-N	
Oil and Grease	
Orthophosphate	
Pesticides	
Fecal <i>Streptococcus</i>	
Sulfate	
SVOCs	
TDS	
TKN	
Total Coliforms	
Total Organic Carbon (TOC)	
Total Phosphorous	
TSS	





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Parameter	Under Evaluation (X)
Turbidity	
VOCs	
Others (Specify: _____)	

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

### II. Chain of Custody (COC)

Yes \_\_\_\_\_ NO \_\_\_\_\_ NA \_\_\_\_\_

Requirements	Y/N/NA	Comments
1. The COC was identified properly (survey, sampling sites).		
2. For each sample the following information is provided:		
a. sample id.		
b. date collected		
c. time of sample collection		
d. sample temperature		
e. sample volume and quantity		
f. number/type of containers collected		





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Requirements	Y/N/NA	Comments
g. sample matrix		
h. sample preservation		
i. analytical parameters		
3. The parameters collected for laboratory analysis are in accordance with the established at the QAPP.		
4. This project required the collection of QA/QC samples. If YES:		
a. Field duplicates /triplicates		
Were identified properly		
Collected at the proper frequency		
b. Trip blanks		
Were identified properly		
Collected at the proper frequency		
c. Field blanks		
Were identified properly		
Collected at the proper frequency		
d. Equipment blank		
Were identified properly		
Collected at the proper frequency		
5. Sampler's signature		





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Requirements	Y/N/NA	Comments
6. The COC is signed and dated for each transfer of samples.		
7. The temperature at which the samples were delivered to the laboratory is in accordance with the QAPP (4°C+/-2).		
8. The COC is identified with a control number assigned by the laboratory.		

### III. Field Data Record (FDR)

Yes \_\_\_\_\_ NO \_\_\_\_\_ NA \_\_\_\_\_

Requirements	Y/N/NA	Comments
1. The FDR is identified properly.		
2. Provide the sampler's names		
3. The id. of the station (sample) is in accordance with the COC.		
4. The date and time are in accordance with the COC.		
5. The field parameters obtained are in accordance with the QAPP approved.		
6. The replicate samples were obtained at the proper frequency.		





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### IV. Field Inspection Form (FIF)

Yes \_\_\_\_\_ NO \_\_\_\_\_ NA \_\_\_\_\_

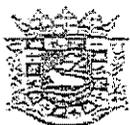
Requirements	Y/N/NA	Comments
1. The project name, date and time are identified properly.		
2. The station id. corresponds to the station description and Municipality.		
3. Technician's names were included.		
4. Weather conditions filled properly.		
5. Contamination sources filled properly.		

### V. Calibrations Forms (CFs)

Yes \_\_\_\_\_ NO \_\_\_\_\_ NA \_\_\_\_\_

Requirements	Y/N/NA	Comments
1. All the CFs corresponds to the measures obtained in the field.		
2. The equipment was calibrated at proper frequency.		
3. The Project name and date were filled properly in accordance with the COC, FDR, and FIF.		





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Requirements	Y/N/NA	Comments
4. The calibration time is previous to the sample collection.		
5. The model of the equipment, the id. number (property number) and the serial number (S/N) corresponds.		
6. The calibration was performed in accordance with the SOP's and QAPP.		
7. If the equipment requires a final verification, the time of the verification is after the collection of the last sample (verify with the COC).		
8. Signature of the field technician and date were provided.		

### VI. Additional Field Documentation

Yes \_\_\_\_\_ NO \_\_\_\_\_ NA \_\_\_\_\_

- List any other documentation form received as part of the field activities:

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Requirements	Y/N/NA	Comments
1. The documents are properly identified with the project name, date, and time (if applicable) in accordance with other forms.		
2. The documentation was filled properly in accordance with other forms and the QAPP.		

VII. General Comments

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

VIII. Corrective Actions

Corrective action required? Yes \_\_\_\_\_ NO \_\_\_\_\_

- To present documentation
- Re-sample
- Other (Specify: \_\_\_\_\_)

Prepared by: \_\_\_\_\_

Date: \_\_\_\_\_

QAPP = Quality Assurance Project Plan; Y/N/NA = Yes/No/Not applicable; COC = Chain of Custody



# Appendix 2: General Analytical Data Validation Checklist





General Analytical Data Validation Checklist

Project Title: \_\_\_\_\_

Route: \_\_\_\_\_

Sampling Date: \_\_\_\_\_

QAPP Title: \_\_\_\_\_ Approval date: \_\_\_\_\_

Date: Initial Eval. \_\_\_\_\_ Final Eval. \_\_\_\_\_ Reviewer: \_\_\_\_\_

REQUIREMENTS	Y/N/NA	COMMENTS
1. The laboratory included all the documentation (data package) for the data validation.		
2. The report included a case narrative with the results in which the laboratory establishes any deviation or an explanation of the data results (i.e. blanks).		
3. The samples were analyzed for all the sampling parameters in accordance with the QAPP of the project.		





## Water Quality Area

REQUIREMENTS	Y/N/NA	COMMENTS
4. The analytical samples were evaluated by the analytical methods established in the approved QAPP. If the answer is NO: a. The alternative method is applicable to the project.		
5. The detection and quantitation limits used apply to the project.		
6. The results were presented in the appropriate analytical units.		
7. The analytical results included the date in which the analyses were performed.		
8. The samples accomplished the holding time for each parameter. If the answer is NO, specify which exceeded: Sample                      Parameter _____                      _____ _____                      _____ _____                      _____		
9. The data report included the Chain of Custody (COC) If the answer is YES: a. The COC was filled properly and signed. b. The samples included in the data report agree with the samples included in the COC.		





## Water Quality Area

REQUIREMENTS				Y/N/NA	COMMENTS
10. The data report included the results of the following quality controls and their contamination (if any):					
FIELD BLANKS					
a. Field blank	parameter	sample	[ ]n		
	_____	_____	_____		
	_____	_____	_____		
	_____	_____	_____		
b. Trip blank	parameter	sample	[ ]n		
	_____	_____	_____		
	_____	_____	_____		
	_____	_____	_____		
c. Equipment blank	parameter	% R	limits		
	_____	_____	_____		
	_____	_____	_____		
	_____	_____	_____		
d. Field duplicate	parameter	% R	limits		
	_____	_____	_____		
	_____	_____	_____		
	_____	_____	_____		





## Water Quality Area

REQUIREMENTS				Y/N/NA	COMMENTS
<b>LABORATORY BLANKS</b>					
a. Method blank	parameter	% R	limits		
	_____	_____	_____		
	_____	_____	_____		
b. MS/MSD	parameter	% R	limits		
	_____	_____	_____		
	_____	_____	_____		
c. Laboratory Control Sample (LCS)	parameter	% R	limits		
	_____	_____	_____		
	_____	_____	_____		
d. Sample replicate	parameter	% R	limits		
	_____	_____	_____		
	_____	_____	_____		





## Water Quality Area

REQUIREMENTS				Y/N/NA	COMMENTS
e. Surrogates (organic)	parameter	% R	limits		
	_____	_____	_____		
	_____	_____	_____		
f. "Performance evaluation sample"	parameter	% R	limits		
	_____	_____	_____		
	_____	_____	_____		
g. Others:	parameter	% R	limits		
	_____	_____	_____		
	_____	_____	_____		
<p>The mentioned quality controls are not necessary during all the analysis or sampling activities. It will depend of the activity design, the analytical method and the QAPP approved.</p>					
<p>11. The data report included the recovery percent (%R) calculation for some or all the blanks mentioned at # 10.</p> <p>If the answer is YES:</p> <p>a. The data report included the %R limits</p> <p>b. The %R's are within the acceptability range</p>					

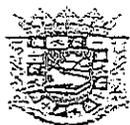




## Water Quality Area

REQUIREMENTS	Y/N/NA	COMMENTS															
If the answer is NO, specify: <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="width: 30%;">Parameter</th> <th style="width: 15%;">%R</th> <th style="width: 15%;">Limits %R</th> </tr> </thead> <tbody> <tr><td>_____</td><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td><td>_____</td></tr> </tbody> </table>	Parameter	%R	Limits %R	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____		
Parameter	%R	Limits %R															
_____	_____	_____															
_____	_____	_____															
_____	_____	_____															
_____	_____	_____															
12. The data report included the Relative Percent Difference (RPD) calculation for some of the blanks included in #10. If the answer is YES: a. The data report included the RPD limits b. The RPD's are within the acceptability range If the answer is NO, specify: <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="width: 30%;">Parameter</th> <th style="width: 15%;">RPD</th> <th style="width: 15%;">Limits RPD</th> </tr> </thead> <tbody> <tr><td>_____</td><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td><td>_____</td></tr> <tr><td>_____</td><td>_____</td><td>_____</td></tr> </tbody> </table>	Parameter	RPD	Limits RPD	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____		
Parameter	RPD	Limits RPD															
_____	_____	_____															
_____	_____	_____															
_____	_____	_____															
_____	_____	_____															
13. The field blanks included at number #10 were analyzed at the frequency required by the approved QAPP: a. Field blank  b. Trip blank																	





ESTADO LIBRE ASOCIADO DE  
**PUERTO RICO**  
 JUNTA DE CALIDAD AMBIENTAL

## Water Quality Area

REQUIREMENTS	Y/N/NA	COMMENTS
c. Equipment blank		
d. Field duplicate		
14. The calibration curve was made in accordance with the analytical method or SOP. If the answer is YES: a. An initial calibration was made using different standards at the required concentrations. b. The calibration verification was made at the required frequency. c. The calibration criteria's were met d. When apply, the RSD limits and the correlation coefficient (0.995) were met.		

QAPP = Quality Assurance Project Plan; Y/N/NA = Yes/No/Not applicable; COC = Chain of Custody; [ ]<sup>n</sup> = Concentration; % R = Recovery percent; RPD = Relative Percent Difference; RSD = Relative Standard Deviation

General Comments:

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JUNTA DE CALIDAD AMBIENTAL

## Water Quality Area

### Corrective Actions:

Corrective action required?      Yes \_\_\_\_\_      NO \_\_\_\_\_

- To present documentation
- Re-sample
- Other (Specify: \_\_\_\_\_)

Prepared by: \_\_\_\_\_

Date: \_\_\_\_\_



# Appendix 6: PREQB ERLPR SOP for Enterolert



Standard Operating Procedure SOP-021:  
Defined Substrate Technology (Enterolert).

PREPARED BY:

SIGNATURE:

DATE:

March 30, 2015

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EILEEN C. VILLAFANE DEYACK, REM PhD

POSITION:

MICROBIOLOGY SECTION SUPERVISOR

REVISED BY:

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DATE:

March 30, 2015

NAME:

JANETTE CAMBRELÉN, MEP

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APPROVED BY:

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DATE:

March 30, 2015

NAME:

FRANCES M. SEGARRA ROMÁN

POSITION:

PREQB QUALITY ASSURANCE AND QUALITY CONTROL OFFICE CHIEF



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## 1.0 Scope and Applicability:

1.1 This Standard Operating Procedure is applicable to the Analytic Method of Defined Substrate Technology (Enterolert). This method of analysis is used as an indicator of bacteriological quality of water. The test detects enterococcus bacteria such as *E. faecium* and *E. faecalis* in samples of freshwater, potable water, marine water and/or residual water.

## 2.0 Summary of Method

2.1 Enterolert Defined Substrate Technology is a method that utilizes 4-methyl-umbelliferyl- $\beta$ -D-glucoside nutrient indicator that fluoresces when metabolized by enterococci. When enterococci utilize their 3-glucosidase enzyme to metabolize Enterolert's nutrient indicator the sample fluoresces. After 24 hours of incubation, 3-glucosidase enzyme hydrolyzes the substrate and provokes a change in color (blue fluorescence).

2.2 The enzyme, 3-glucosidase fluoresces under ultra-violet light (365nm).

## 3.0 Definitions

3.1 In the context of this method the following definitions apply:

3.1.1 Enterococcus bacteria- The enterococcus group is a subgroup of fecal streptococci that includes *S. faecalis*, *S. faecium*, *S. gallinarum* and *S. avium*. Enterococci are a valuable indicator for determining the extent of fecal contamination in recreational surface waters.

## 4.0 Health and Safety Warnings

4.1 Wear rubber gloves when performing the sampling and analysis procedures.

4.2 Wash hands when sampling and analyzing potentially contaminated waters and/or before leaving the facilities of the Environmental Research Laboratory.

4.3 Always work under aseptic conditions.

4.4 Make sure that the UV light source is 6 watt, long wavelength (365nm).

4.5 In case of using a more powerful UV light source (approximately 15watt), use protective goggles or a face shield.



- 4.6 The access panel of the Quanti-Tray Sealer should only be opened by personnel qualified to clean the inside of it. Do not open the access panel or tilt this sealer if sample is dripping from the unit.

## 5.0 Interferences

- 5.1 The manufacturer of Enterolert recommends diluting marine water samples at least 1:10 with sterile fresh water in order to reduce the possibility of interference by marine bacilli.
- 5.2 If chlorinated water is to be analyzed, sterile ample bottles must contain sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) to neutralize any residual.
- 5.3 If non-chlorinated water is to be analyzed, thiosulfate should not be used because it can serve as an additional food source for the bacteria.
- 5.4 Inappropriate sterilization processes can contaminate the equipment and/or materials.
- 5.4.1 The rubber insert of the Quanti-Tray Sealer may be autoclaved, or it may be cleaned with isopropyl alcohol or household bleach, taking the usual precautions when handling such liquids.
- 5.4.2 Clean the outside of the sealer with a soft, dry cloth. A soft cloth moistened with water, household bleach, or isopropyl alcohol may also be used, taking the usual precautions when handling such liquids.
- 5.4.3 To clean and disinfect the interior of the Quanti-Tray sealer you should practice the following steps:
- 5.4.3.1 Ensure power supply is off, sealer is unplugged and unit has completely cooled down for 90 minutes.
- 5.4.3.2 Remove input tray sheif. Loosen four quarter-turn fasteners and remove the access panel.
- 5.4.3.3 Loosen hold-down screws, which secure the lower roller assembly to the bottom plate of this sealer.
- 5.4.3.4 Remove lower roller by lifting straight up and then out.
- 5.4.3.5 Use mild detergent, diluted bleach or isopropyl alcohol to clean all accessible surfaces inside this sealer and the lower roller assembly.

5.4.3.6 Dry interior and roller assembly with paper towels or soft cloth.

5.4.3.7 Reinstall bottom roller assembly on locating pins and tighten the hold-down screws. Fasten the access panel and reattach the tray shelf.

5.4 Do not prepare culture media with incorrect quantities of reagents.

5.5 Materials should be preserved in designated places meeting manufacturer's requirements and excessive handling of sterilized materials should be avoided.

5.6 Reagents should be stored out of direct sunlight.

## 6.0 Personnel Qualifications

6.1 The personnel performing the bacteriological analysis with Enterolert should be an Environmental Microbiologist with 2 or more years of experience in Environmental Microbiology area. The analyst must demonstrate capability on PT or blind QC sample analysis.

## 7.0 Equipment and Supplies

The necessary equipment and supplies for the water sample analysis include:

### 7.1 Equipment:

7.1.1 Quanti-Tray Sealer

7.1.2 A 6watt, 365nm, ultra-violet lamp (Long-wave UV lam: 365-366nm)

7.1.3 Environmental incubator

7.1.4 Incubator (set at 41°C)

### 7.2 Supplies:

7.2.1 Quanti-Trays

7.2.2 Enterolert dry media in snap packs, stored in the dark at 4-30°C



7.2.3 Quality Control Cultures:

Strain	ATCC Number
<i>Enterococcus faecium</i>	35667
<i>Serratia marcescens</i> (gram-)	43862
<i>Aerococcus viridians</i> (gram+)	10400

7.2.4 Sterile 100 ml bottles

7.2.5 Sterile disposable pipette

7.2.6 Antibacterial solution

7.2.7 Safety equipment

7.2.8 Quanti-Tray MPN Table

7.2.9 Disposable gloves

7.3 Quanti-Tray Sealer is operated as follows:

7.3.1 Turn power switch on. The amber power light should illuminate.

7.3.2 Allow the sealer to warm up and the green ready light to come on, indicating that the unit has reached operating temperature. Place an empty Quanti-Tray or Quanti-Tray/2000 rubber insert on the input shelf with the large cut out facing away from the sealer.

7.3.3 Place a Quanti-Tray or Quanti-Tray/2000 filled with sample and reagent on to the rubber insert, making sure that the tray is properly seated in the rubber insert, and with each well of the tray in its corresponding rubber insert hole.

7.3.4 Slide the rubber insert with tray into the sealer until the motor grabs the rubber insert and begins to draw it into the sealer.

7.3.5 In approximately 15 seconds, the tray will be sealed and partially ejected from the rear of the sealer.

7.3.6 If at any time you wish to reverse the motor drawing the rubber insert into the sealer, press and hold their verse button. However, do not reverse the motor once the rubber insert has been drawn fully into the inputs lot.

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7.3.7 Multiple rubber inserts can be run consecutively without pausing (seals up to 4 trays per minute).

7.3.8 Turn off sealer when not in use.

## 8.0 Preservation of Sample

8.1 Compare sampling data with received chain of custody data.

8.2 All samples should have maintained a target temperature of 6°C or below when arriving to the Environmental Research Laboratory.

8.3 Refrigerate or keep samples at a target temperature of 4°C or below. Laboratory analysis should begin within 6 hours of sample collection and within two hours of receiving the sample at the lab.

## 9.0 Procedure

9.1 Select the work place and after sterilizing continue with the processing of samples. Shake sample bottles vigorously 25 times before removing aliquots for preparing dilutions.

9.2 Prepare a dilution 1:10 with distilled sterile water (10 mL water sample and 90 mL sterile water) in a container labeled with the sample number.

9.3 Open the pack by snapping back the top of the score line. Do not touch the opening of the pack.

9.4 Tap the snap pack to ensure that all of the powder is at the bottom of the mixing bottle (Appendix 1). Add to the sample and shake until dissolved.

9.5 Once the media is mixed with the sample aliquot and distilled water in the 100 mL bottle, it is then poured into the Quanti-Tray.

9.6 Prepare a blank run with each batch of sample with 100mL of distilled water.

9.7 Use one hand to hold a Quanti-Tray upright with the well side facing the palm.

9.8 Squeeze the upper part of the Quanti-Tray so that the Quanti-Tray bends toward the palm.

9.9 Gently pull foil tab to separate the foil from the tray. Avoid touching the inside of the foil or tray.



- 9.10 Pour there agent/sample mixture directly into the Quanty-Tray, avoiding contact with the foil tab (Appendix 1).
  - 9.11 Tap the small wells 2-3 time store lease any air bubbles. Allow foam to settle.
  - 9.12 Place the sample-filled Quanti-Tray onto the Quanti-Tray/2000 rubber insert of the Quanti-Tray Sealer with the well side (plastic) of the Quanty-Tray facing down.
  - 9.13 Seal in the Quanty-Tray Sealer. Verify that all the wells were filled with the sample.
  - 9.14 Incubate for a minimum of 24 hours at  $41^{\circ}\text{C}\pm 0.5$  and to a maximum of 28 hours. Record the incubation time (Appendix 1).
  - 9.15 Following the incubation period, observe and count the number of positive (fluorescent) wells in a dark room. For enterococci look for blue fluorescence using the 6 watt, 365nm, UV light within 5 inches (13centimeters) of the sample. The number of wells that fluoresce are to be recorded.
  - 9.16 Face light away from your eyes and towards the sample. The fluorescence intensity of positive wells may vary.
  - 9.17 Refer to the Most Probable Number (MPN) Table specific to the type of Quanty-Tray used (51 well or 97 well type of Quanty-Tray) to obtain a MPN for enterococci of sample. Alter obtaining the initial MPN result from the table; multiply that result by the dilution level to obtain the final result. Results are to be reported as CFU/100mL.
- 10.0 Data and Record Management
- 10.1 If the sample is inadvertently incubated over 28 hours without observation, the following guidelines apply:
    - 10.11 Lack of fluorescence after 28 hours is a valid negative test.
    - 10.12 Fluorescence after 28 hours is an invalid result.
  - 10.2 Data validation and reporting should be done according to Standard Operating Procedures of the Water Quality Area of the Environmental Quality Board.
  - 10.3 Data will be stored in an active file for at least five (5) years. After this period, the records will be kept in a non-active file for 5 more years before destruction. Data entry will be done using the STORET program. Also, a Standard Bacteriological Count Bench Sheet will be used to summarize the data (Appendix 2).



## 11.0 Troubleshooting

- 11.1 Fuses are located in the fuse holder just above the power switch.
- 11.2 If it is necessary to change a fuse, turn off this sealer and unplug the power cord from this sealer before opening fuse holder.
- 11.3 Use 6 Amp Buss MDL-6 fuses or equivalent in the 115 watt unit and 4 Amp Buss GDC-4 fuses or equivalent in the 230 watt unit.

## 12.0 Quality Control and Quality Assurance

- 12.1 The following quality control procedure is required for each lot of Enterolert:
  - 12.1.1 Inoculate three (3) sterile vessels filled with 100 ml sterile water with the following:

Strain	ATCC Number	Expected Result
<i>Enterococcus faecium</i>	35667	Fluorescence
<i>Serratia marcescens</i> (gram-)	43862	No fluorescence
<i>Aerococcus viridians</i> (gram+)	10400	No fluorescence

- 12.1.1 Results of the positive and negative controls must be recorded
- 12.2 Duplicate Counts: A duplicate count should be performed per route of samples received. Duplicate counts should be within 5% (same analyst) or 10% (multiple analysts).
- 12.3 Negative Blanks: One blank (sterile) water sample will be analyzed per batch of samples processed.
- 12.4 External reference samples: One external reference sample must be run per year.
- 12.5 New lots of sample containers: will be checked for sterility using positive and negative controls.
- 12.6 All Quanti-Trays that have any positive wells must be sterilized in a 30 minute autoclave cycle and disposed of in an appropriate manner.



### 13.0 References

- Comisión Federal para la Protección contra Riesgos Sanitarios. (2004). *Lineamientos para determinar la calidad de agua de mar para uso recreativo con contacto primario*. Retrieved on June 18, 2007 from: [http://www.sld.cuigsealerias/pdf/sitios/rehabilitacion-ballineamientos\\_para\\_determinar\\_la\\_calidad\\_del\\_agua\\_de\\_mar\\_para\\_uso\\_recreativo.pdf](http://www.sld.cuigsealerias/pdf/sitios/rehabilitacion-ballineamientos_para_determinar_la_calidad_del_agua_de_mar_para_uso_recreativo.pdf)
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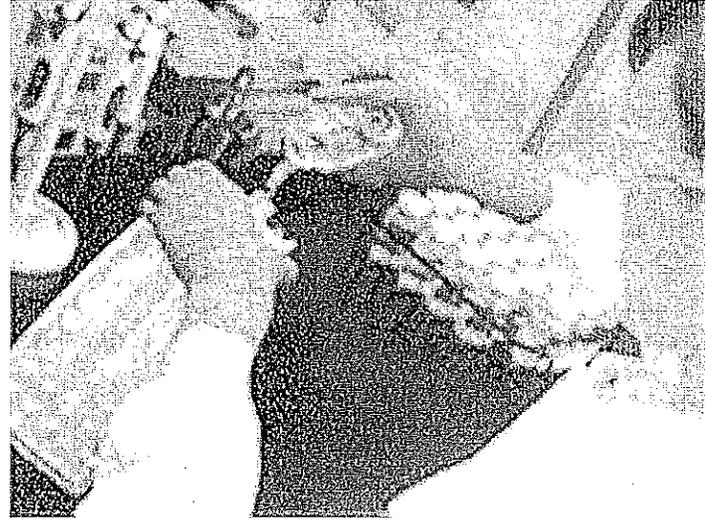
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Appendix 1: Methodology Illustrations:

Addition of the reagent to sample



Addition of the sample reagent to tray



Tray Sealing and Incubation

