Document Number: WEAC-AB-TM.005	Revision # : 04 Revised: 06 Jan 2022
Title: Determination of γ-ray Emitting Radionuclides in Food Matrices Using Cerium Bromide γ-ray Spectrometry	
	WEAC-AB-TM.005

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1. Purpose

This procedure describes the sample counting and processing related steps for the determination of gamma-ray emitting radionuclides in food samples using Cerium Bromide spectrometers. Details of how to accomplish efficiency calibrations, instrument set up, facility configuration, etc. can be found in the Genie-2000 Software user's manual.

2. Scope

This document details the methods for preparing food samples for gamma counting, for using the high-purity germanium spectrometers, for using the associated spectrometers and spectroscopy software. The procedure is suitable for sample collections that provide a minimum of 500mL of the edible

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portion of the sample. The method is designed to measure 137Cs, 134Cs, and 131I with a bias of <38.74% and a 1 σ precision of <16% at the corresponding derived intervention levels (DILs) for each radionuclide. Instructions are provided for the server-side users only. The spectral analysis parameters are set so that each of the radionuclides contained in the spectral library may be identified in a spectrum for quantification. The procedure is reliable and reproducible over the range of typical food densities (approximately 0.4 g/mL to 1.5 g/mL). Samples with very low density (<0.5 g/mL) or those counted in a high background area may need longer count times to meet the desired I-131 detection limit of <1/3 of the DIL. Any additional SOP's related to use in the field are maintained in the field location.

3. Responsibility

- A. Supervisors
 - 1. Ensure this procedure is properly implemented.
 - 2. Ensure that the appropriate personnel are trained to perform the analysis using this SOP.
 - 3. Ensure that the analysts are capable of providing acceptable analytical results through proficiency evaluation.
- B. Method Monitors
 - 1. Maintain and review the method QA documentation related to the method.
 - 2. Ensure that method QC trending is available as needed.
 - 3. Assist in investigations related to the method.
- C. Analysts
 - 1. Adhere to this SOP.
 - 2. Perform and document required function verification and preventive maintenance on the spectrometer used for the analysis.
 - 3. Ensure all analytical results are fully supported by acceptable quality control data.
 - 4. Inform their supervisor when problems arise that could negatively impact timely sample analysis or the quality of sample results.
 - 5. Document sample analyses on appropriate worksheet.

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4. Background

A 16oz (~475mL) propylene deli container is filled with the sample and used for analysis by counting on a Cerium Bromide (CeBr) scintillation detector. The detector consists of a 2 x 2 "CeBr crystal in an aluminum housing including a photomultiplier tube, magnetic/light shield, and a high-voltage power supply. The detector is housed in a modular and mobile steel shield. The detector is stable across a wide temperature range due to its automatic detector gain adjustment that occurs during detector quality control check count. A system background is established using a 16 oz deli container filled with lab grade water and placed in the center of the detector platform. The system background check is used to confirm no radioactive contamination of the detector system. A system background count can be used when longer sample count times are employed and is subtracted from the sample results.

The detector/shield system is portable and easily disassembled and reassembled. When the location of the system is changed, quality control checks must be performed prior to sample analysis even in cases where they have already been performed that day.

5. References

- A. <u>Compliance Policy Guide: Sec. 555.880 Guidance Levels for</u> <u>Radionuclides in Domestic and Imported Foods (CPG 7119.14).</u> <u>November 19, 2020.</u>
- B. Food and Drug Administration (FDA) FoodScreen-Plus; Canberra Instrument Validation Document.
- C. Accidental Radioactive Contamination of Human Food and Animal Feeds: Recommendations to State and Local Agencies. August 1998.
- D. The Genie-2000 Operations Manual; 9233652E v3.0*.
- E. Basic User Reference The APEX Lab Productivity Suite User's Manual; 923527C v1.3
- F. The Genie-2000 Customization Tools Manual; 9233653E v3.0*

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6. Procedure

6.1. Instructions for Sample Processing and Field Spectrometer

6.1.1. Instrumentation, Equipment, and Supplies

- A. <u>Gamma-Ray spectrometer</u>: A Cerium Bromide spectrometer. The field deployable spectrometers are operated with GENIE- Field Pro Software.
- B. <u>Data Server Computer:</u> Canberra Apex Client/Server with Auto-Import add-on feature installed on the data server computer. Data analysis is performed on the server computer using APEX/GENIE-2000 Canberra Spectroscopy Software. Nuclide identification is dependent on energy calibration, spectral analysis parameters, and the library used.
- C. <u>Balance</u>: A calibrated balance capable of weighing samples up to 1 kg with a readability of one tenth of one gram.
- D. <u>Sample counting container</u>: A commercially available 16oz (~475mL) deli container with measurements of approximately: Bottom Diameter-8.5 cm, Top Diameter-10.6cm, and Height-7.8cm.
- E. <u>Utensils:</u> Mortar and pestle, food processors, and common food utensils such as spoons, cutting knives, spatulas, etc. for homogenizing food samples.

6.1.2. Reagents and Standards

- A. <u>Laboratory Grade Water</u> (from Milli-Q system or equivalent water purification system)
- B. <u>Quality Control Check Sample (QCC)</u> A NIST-traceable gamma reference standard that contains radionuclides with photons that represent the low, middle, and high energy range. An NRC license exempt button source, such as Eu-152, may be used for this purpose.
- C. <u>Laboratory Control Sample (LCS)</u> A NIST-traceable mixed- gamma reference standard prepared in the same geometry as that used for the samples. The standard must contain radionuclides with photons that represent the low, middle, and high energy range. An LCS is counted once per calendar week (i.e. a batch starts on Sunday and ends after Saturday) for each detector used to count samples. It is recommended to count the LCS more frequently and preferably the day of sample analysis.

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6.1.3. Analytical Procedure

- A. Sample Preparation
 - 1. Sample Preservation

Samples are typically thawed and maintained at refrigeration temperatures until composited.

- a. Samples at WEAC need to be obtained and secured according to WEAC-QMS.5.8, WEAC Sample Handling Procedures. Samples obtained at remote locations should have chain of custody documented appropriately.
- b. Maintain sample in accordance with its labeled instructions for preservation and storage. Ensure that all refrigerators and freezers used meet the QC requirements. WEAC-AB.3.0, Monitoring of Freezers, Incubators, Ovens, Waterbaths, and Refrigerators.
- c. When no labeling or storage instruction is indicated, take appropriate measures (usually refrigerating or freezing) to maintain the sample's quality until it's composited.
- d. To minimize uncertainties due to composite layer separation and settling, ensure counting proceeds as soon after compositing as possible. When preservation and storage procedures are atypical (e.g., a sample is refrozen or preserved with formaldehyde), record these specifics.
- 2. Sample Compositing
 - a. Remove the inedible portion of the sample from all portions that will be used for analysis. Ensure utensils used for sample preparation are clean. To prevent cross-contamination, do not reuse utensils until they've been cleaned. WEAC-LAB.23.0, Laboratory Glassware Washing and General Maintenance.
 - b. Combine the edible portions of sample subs (usually using a food processor or blender) to create a homogenous composite.
 WEAC-AB.8.0, Laboratory Sub Sampling.
- 3. Sample Weighing, Counting Preparation
 - a. Ensure that the balance meets QC requirements. WEAC-LAB.6.0, Operation and Maintenance of Laboratory Balances.

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- b. Obtain a counting container. Mark the container with a unique identification number or information and record it on the worksheet.
- c. Tare container with lid to zero, then transfer the sample to the counting container filling to just below the top of the container. Allow enough room for the lid to be attached without displacing any sample in the container.
- d. Record the mass of the analytical portion to the nearest 0.1g. Determine the density of the sample using 450 mL as divisor.
- B. Sample and Laboratory Control Sample Analysis
 - 1. Routine Operation
 - a. Verify that the daily spectrometer QC calibration check was performed, the daily background/method blank was counted, and that the detector meets QC specifications. Indicated by the "BACKGROUND CHECK" and "CALIBRATION CHECK" buttons turning from red to green.

🤺 Genie-FoodPro FOOD_STA	and the second	
CONTAINER 16 oz Deli •	COUNT SAMPLE	BACKGROUND CHECK
CONTENTS		Last Background:4/27/2016 9:18:34 AM Next Background:5/27/2016 9:18:34 AM
LCS ·		CALIBRATION CHECK
		Last Calibration:4/27/2016 8:38:31 AM Next Calibration:4/28/2016 8:38:31 AM
Maintenance - Stopped: 1200.0 / 300 live (0.21% dead)		
T		
		GPS Location Take Photo
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- b. If the daily calibration check has not been performed, place the QCC source on the detector and press the "CALIBRATION CHECK" button.
 - i. A progress bar and remaining count time will appear.
 - ii. Upon completion of a passing count, the "CALIBRATION CHECK" button will be green.

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- c. If the daily background check has not been performed, place the background check container on the detector and press the "BACKGROUND CHECK" button.
 - i. A progress bar and remaining count time will appear.
 - ii. Upon completion of a passing count, the "BACKGROUND CHECK" button will be green.
- 2. To count a Sample or LCS using FoodPro software:
 - a. On the main screen select the applicable description of sample from the contents drop down menu. Options include: Low Density (0.4-0.7 g/mL), Medium Density (0.7-1.1 g/mL), High Density (1.1-1.5 g/mL), or LCS.

Genie-FoodPro FOOD_STA	And in the second s	- 0 X
CONTAINER 16 oz Deli •	COUNT SAMPLE	BACKGROUND CHECK
CONTENTS		Last Background:4/27/2016 9:18:34 AM Next Background:5/27/2016 9:18:34 AM
LCS .		CALIBRATION CHECK
Mentenance - Stopped: 1200.0 / 300 live (0.21% dead)		Lest Calibration:4/27/2016.8:38:31 AM Next Calibration:4/28/2016.8:38:31 AM
		GF1 Locari
📀 C 📋 🔼 📼 🔍	THE REAL	▲ 🌯 🔯 🕅 🌒 957 AM

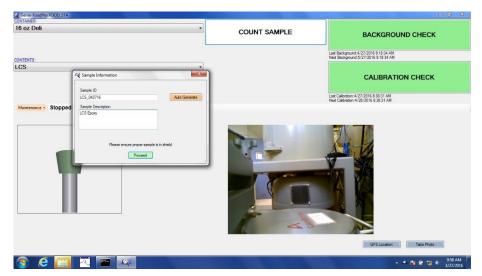
 Enter the Sample ID and Sample Description in the "Sample Information" pop up window. Alternately, select "Auto Generate" to propagate a unique Sample ID. Select "Proceed" button to start the count.

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c. Upon count start, a progress bar and count time remaining field will appear. The "COUNT SAMPLE" button will now say "COUNTING...(STOP). This button can be used to stop the current count.

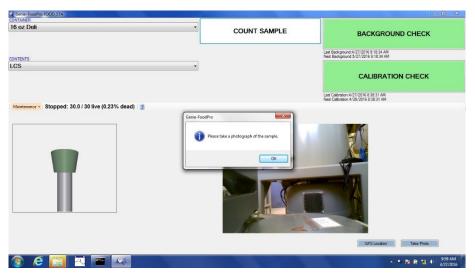
Genie-FoodPro FOOD_STA		- • • •ו
CONTAINER 16 oz Deli -	COUNTING (STOP)	BACKGROUND CHECK
CONTENTS		Last Background:4/27/2016 9:18:34 AM Next Background:5/27/2016 9:18:34 AM
LCS -		CALIBRATION CHECK
Maintenance - Counting: 00:18:57		Last Calibration:4/27/2016 8:38:31 AM Next Calibration:4/28/2016 8:38:31 AM
T		OFS Location Trive Prote
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d. Upon completion of the count, a pop-up window saying "Please take a photo of the sample" will appear. Remove the sample from the shield and place it, with unique identification visible. Press "OK". On bottom right of main screen press "Take Photo".

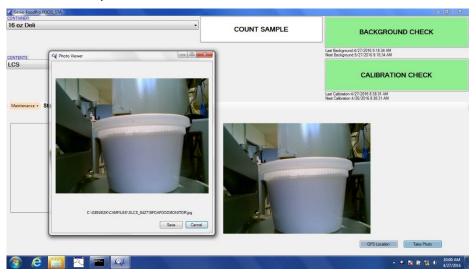
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e. A photo viewer window will pop up with a picture of the sample. If the photo is acceptable, press "Save". The photo will be saved in the same folder as the spectral file with a matching ID. If the photo is not acceptable, press "Cancel", adjust the sample, and take another photo.



- C. Data Transfer
 - 1. If the field instrument computer and data server computer are on the same network and file transfer parameters are set:
 - a. Select the FoodPro icon on the bottom right hand of the screen.
 - b. Select File Transfer Manager to open a window containing a list of sample files ready for transfer.

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- c. Press "Send File".
- d. A pop-up window will confirm successful transfer.
- 2. If the field instrument computer and data server computer are NOT on the same network:
 - a. Use an approved secure method to transfer data files from one system to another. Some examples include via drop box, email (to: ORAWEACPortableG@fda.hhs.gov), or ironkey.

6.2. Procedure - Instructions for Server-Side

6.2.1. Data Processing Spectral Files and Preparing Worksheet Attachments

- A. If the field computer has successfully sent files using the File transfer Manager, proceed to 6.2.1.C. If transferring files via secure USB drive, proceed to 6.2.1.B. If accepting files via email or drop box:
 - 1. Watch the ORAWEACPortableG@fda.hhs.gov email user for new messages. When new messages are received, copy all the attachments to a USB drive that meets the FDA's IT security requirement.
 - Create a data folder for the data (e.g., for Alaska samples, H:\Radiochemistry\Analytical Branch\Rad-Chem\Gamma\AKData\yyyymmdd) and move the data files and a copy of any associated files or messages to the corresponding folder. Note: file drop boxes may have a time limit associated with data retrieval).
 - 3. Use the snipping tool to create an image of the files related to the sample as received (save the file as a .jpg) on your FDA issued computer. This includes QC, Background, Sample File, Sample Pictures, etc. it is not necessary to include a picture of files for long background counts with the sample. For example, see below.

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Name ^	Date modified	Туре	Size
B1048280416FDAFOODMONITOR.cnf	4/28/2016 11:08 AM	CNF File	183 KB
C1042280416FDAFOODMONITOR.cnf	4/28/2016 10:47 AM	CNF File	183 KB
C1042280416FDAFOODMONITOR.txt	4/28/2016 10:47 AM	Text Document	1 KB
SMD04281602FDAFOODMONITOR.cnf	4/28/2016 11:52 AM	CNF File	242 KB
SMD04281602FDAFOODMONITOR.jpg	4/28/2016 11:50 AM	JPEG image	65 KB
SMD04281602FDAFOODMONITOR.pdf	4/28/2016 11:51 AM	Adobe Acrobat Doc	7 KB
SMD04281602FDAFOODMONITOR.txt	4/28/2016 11:31 AM	Text Document	1 KB

B. Bring the USB drive to the data server computer and copy all the files to the "AutoImport" folder (C:\Canberra\Apex\Root\Default\Sample Import\Auto Import)(see shortcut icon below).



- C. If a system background file is associated (for longer count times), import the background file through the Sample tab and the Import screen. Select the detector and retain imported calibrations, browse to select the system background file that you want to import. (Note: ensure that the system background geometry matches the sample for which it will be applied. If multiple sample geometries are being processed, this will require the background be imported for each).
- D. Once the background is imported, go to the Quality Assurance tab and the Background screen to approve the background for use.
- E. Open APEX to the Main tab. Double click on the "AutoImport" Program icon to automatically import sample spectral files into APEX (see icon below).



F. During the import function, sample files will appear at the bottom of the sample queue in APEX.

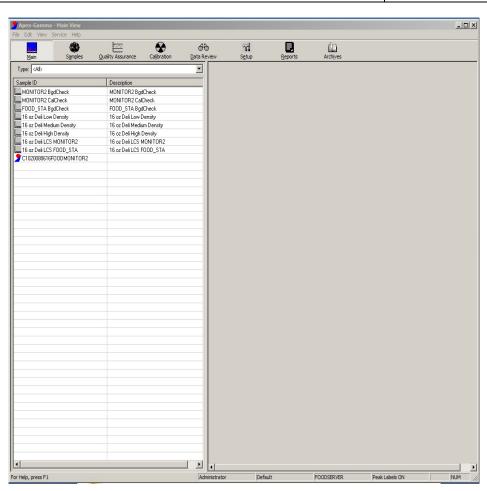
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G. Once they are all imported, go to the "Data Review" page. Perform a search using the appropriate detector parameter(s). Find the appropriate file(s) highlight and click next.

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	Apex-Gamma - Data Review View				-D×
	File Edit View Service Help	Calibration	Setup Reports	<u>Archives</u>	
	Normal Composite Select a Sample Status: <alb< td=""> Sample Type: <alb< td=""> Sample ID: <alb< td=""> Batch ID: Batch Samples Imported Samples Date/Time Filter Class Sample Date: Class Samples Batch Samples Batch Samples Batch Samples Batch Samples Batch Samples Batch Samples 219.24 PM Before: 7/13/2016</alb<></alb<></alb<>		Sample ID A., Assay Da	None Available	eg: Records >>
				Nex	
	For Help, press E1	Administr	ator Default	FOODSERVER	k Labels ON

H. The sample spectrum and report will now be available for review.

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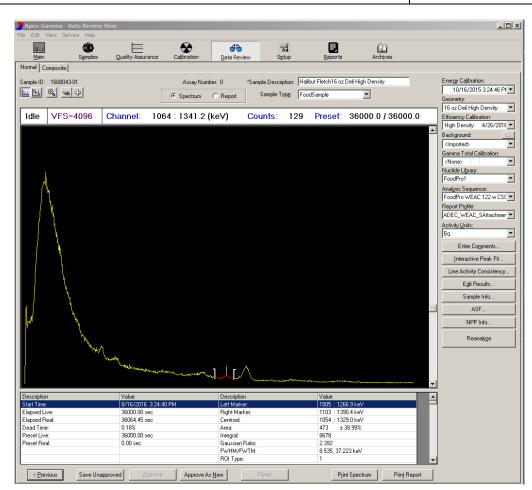
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 Ensure that the sample information and processing parameters are appropriate for the standard used. Enter a sample description if there isn't one (e.g. QCC, Eu-152, etc). Then click reanalysis. Once the reanalysis is complete, click the "Save Unapproved" button, answer yes – you do want to transfer to the QA file and OK.

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- J. Open the calibration check (usually DETECTORNAME CALCHECK) and background check (usually DETECTORNAME BCKCHK) files using the APEX editor to verify that the detector passes QC requirements.
- K. Find the LCS file that was analyzed on the same start date as the sample (or closest previous date), highlight, and click next. Review the report (including verification of appropriate peak integration, standard reference date and standard identification). Save the report for inclusion in the sample worksheet as an attachment.

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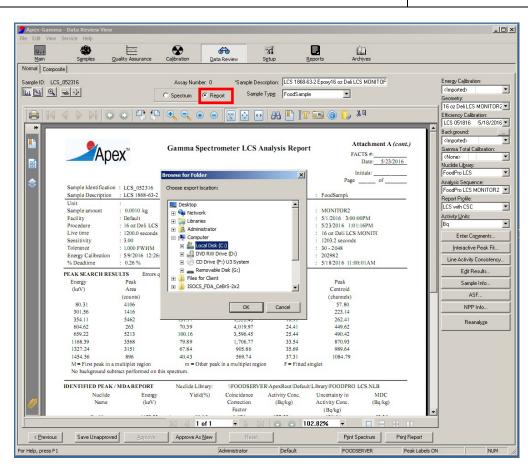
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- L. Find the sample file, highlight, and click next. Review the report (including verification of appropriate peak integration, reference date and sample geometry). If changes are made (for example to the sample geometry after review), click reanalysis and save upon completion both in APEX. Save the report for inclusion in the sample worksheet as an attachment.
- M. Transfer all the report files to your FDA issued computer. Open the sample worksheet form, click tools > content > edit object (right click) > place image (select filename .jpg saved in Section 6.2.1.A.3) and position and resize the image in the file transferred section at the top of the page. Complete the worksheet and append the sample picture, sample processing sheet, collection report, etc. as attachments.
- N. Digitally sign worksheet and place the .pdf files into the appropriate H: drive folder. For Alaska ADEC samples: H:/Analytical Branch/Radiochemistry/Rad-Chem/ADECSampleWS\ADEC Samples

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folder. Name the sample folder with the FDA FACTS number, an underscore and the ADEC state id (e.g. 123456_stateid).

6.3. Quality Assurance

QC Results and control charts are maintained on the server-side computer. Supporting data for QC (for example, QC criteria) is maintained in the LIMS online equipment folder.

A. Quality Control Check (QCC)

A QCC count is performed by counting an Eu-152 source. The QCC is performed on each day of use. Data is collected for FWHM at 122keV, 344keV and 779keV and for activity at 123keV and 779keV. The specification for FWHM is established based on historical data and the activity specification is set at $\pm 10\%$ (1-sigma) of the certified value. Additional data may be collected for troubleshooting (e.g. centroid, or cps of peaks). Values for FWHM and activity are acceptable if they are within the warning limits (-2 to 2 sigma) or between the warning and control limits (-3 to -2 or 2 to 3 sigma) not more than three consecutive times.

B. Quality Control Background (QCB)

A short quality background check is performed to verify the absence of contamination. The QCB is also performed on each day of analysis. Data is collected in the low and high energy regions for background count rate. The specifications are established based on historical data. The count rate values are acceptable if the FWHM is within the warning limits (-2 to 2 sigma) or between the warning and control limits (-3 to -2 or 2 to 3 sigma) not more than three consecutive times.

C. Long System Background Count

If a long sample count is used, it may be paired with a system background count of an equal count length, especially where K-40 will be quantified. The count is performed within 3 days of the sample count. This spectrum is reviewed and approved by the analyst who ensures that all peaks are due to naturally occurring background and are properly integrated.

D. Laboratory Control Sample (LCS)

The LCS must be analyzed on each day of use. The LCS results for activity of Ba-133, Cs-137 and Co-60 are collected. The specification is set at $\pm 10\%$ (1-sigma) of the certified value. Values are acceptable if they are within the warning limits (-2 to 2 sigma) or between the warning

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and control limits (-3 to -2 or 2 to 3 sigma) not more than three consecutive times.

E. Proficiency Test Sample (PT)

Follow the requirements of WEAC-QMS.5.9 WEAC Proficiency Testing for annual proficiency study participation.

7. Glossary/Definitions

MCA: Multichannel Analyzer

8. Records

- A. Portable Worksheet
- B. Files (.cnf, .jpg and .txt) for analysis
- C. Spectrometer reports Sample and LCS
- D. Quality Control Charts (found on the spectrometer)

9. Supporting Documents

None

10. Document History

Revision #	Status* (D, I, R)	Date	Author Name and Title	Approving Official Name and Title
1.0	I	9/26/2016	Kelly Garnick, Chemist Thomas Scott, Chemist	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
02	R	2/24/2021	Kelly Garnick, Chemist Thomas Scott, Chemist	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
03	R	07/15/2021	Kelly Garnick, Chemist Thomas Scott, Chemist	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR
04	R	See InfoCard	THOMAS SCOTT, CHEMIST KELLY GARNICK, CHEMIST	PATRICK REGAN, ANALYTICAL BRANCH DIRECTOR

* - D: Draft, I: Initial, R: Revision

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11. Change History

Revision #	Change
04	Reworded Purpose, Scope, Responsibilities and Background sections; added D. and E. to References, revised section 6 Procedure to include: sections for client-side operations, and revise sections for server-side operation to expand applicability of sample data processing to different client-side units.

12. Attachments

None