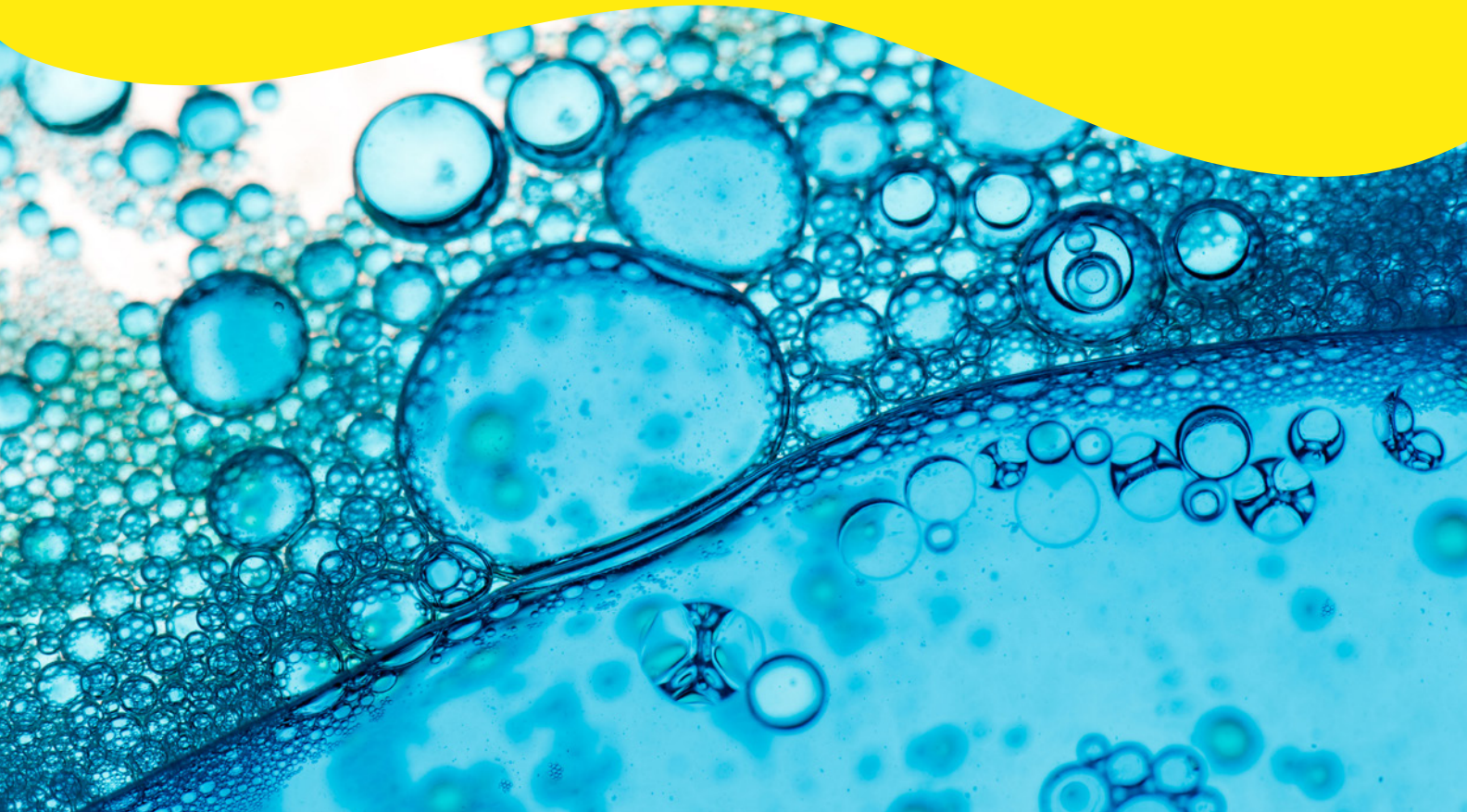
Guide to the analysis of radioactivity in drinking water



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Introduction

Since the publication of the European Council Directive 2013/51/Euratom in October 2013,[1] the analysis of drinking water has been the focus of many laboratories throughout Europe. Within this regulation are changes to requirements such as sampling rate, detection limits, and more which have forced many labs to upgrade instrumentation and adjust their previous methods.

With this new directive, gross α/β-measurement has once again become a measurement of interest. In many cases, the need for additional investigation can be avoided if the activity of the sample measures below 0.1 Bq/L for α-nuclides and below 1.0 Bq/L for β-nuclides. When the measured gross α/β-activity is below these thresholds, it can be assumed that based on the average annual consumption of water, the annual delivered dose will be less than 0.1 mSv/a and therefore the drinking water does not pose a risk to health in terms of ingested radionuclides. Starting with gross α/β-measurements can potentially save laboratories time and hassle by avoiding other measurement if the measured activity is low enough.

Council Directive 2013/51/Euratom does not give detailed information about the required instrumentation and methods - it only stipulates that the method has to reach the defined detection limits. Other regulations, such as ISO 11704[2,3] or ISO 11929[4] referred to in the council directive must be used to ensure compliance with the council directive.

Revvity has the expertise to guide you to ensure your compliance with Council Directive 2013/51/Euratom and other regulations. This guide has been developed to do just that and provides a fast, gross α/β-screening method as a reference for those looking to establish a new method.

Should you find yourself needing additional support, Revvity can also provide on-line or on-site trainings to help you set up new methods on Revvity liquid scintillation counters. These training can include, but are not limited to, the theory of liquid scintillation counting, sample preparation, determination of optimum windows and pulse shape analysis (PSA) settings, performing the measurements of standards and unknown samples, and preparing the necessary documentation according to ISO 11929 which is required by Council Directive 2013/51/Euratom. The methods described in this guide are primarily based on the existing regulations around drinking water.[1-8] It is worth noting that there is a plethora of publications available related to gross α/β-counting, some of which are referenced throughout this guide.

A liquid scintillation counter is used to perform gross α/β-measurements. In such an instrument, the energy of the radioactive nuclide is converted into light using a scintillation cocktail. The counter uses photo sensitive photo multiplier tubes (PMTs) to detect the light and convert the light into electrical pulses which are saved in a Multi-Channel Analyzer (MCA) for further analysis. Within the MCA, the detected pulses are sorted by energy, typically measured in keV, which results in a spectral distribution. Although the energy of α-nuclides is much higher (typically 2-8 MeV) than the energy of β-nuclides (typically 0-2 MeV), the resulting spectra can overlap. This overlap is due to poor energy conversion of α-nuclides (where only ~10% of the energy of α-nuclides is converted to light) combined with energy loss due to collisions of the large α-particles with other elements. This results in a detection of most α-nuclides in the energy range of 100-800 keV (depending on quench) which can overlap with many β-nuclides.

Due to the spectral overlap of α- and β-nuclides, the ability to discriminate between the two is required for accurate gross α/β-measurements. This discrimination is based on the pulse length of the nuclides. On older models of Tri-Carb® liquid scintillation counters, this discrimination was

accomplished using Pulse Decay Analysis (PDA), while more modern systems now use Pulse Shape Analysis (PSA). Typically, the PDA value in older Tri-Carb systems ranged between 100-150, which is compared to the PSA value in the Quantulus™ GCT liquid scintillation counters and the newer Tri-Carb models (models 4910, 5110 and later). Discriminator values mainly depend on the quench of the sample and vial type used. Both in the cases of PDA and PSA, discriminators are based on the pulse length, but the algorithms used to obtain the final value are different.

Due to the detection limits enforced by the European Council Directive, we strongly recommend using very sensitive LSCs to reach the detection limits for the gross α-measurement within an acceptable time frame. Typically, a few hours of counting time are required for gross α-measurements but only minutes for ^{3}H and ^{222}Rn measurements are needed to reach the required detection limits. The system we recommend is the Quantulus GCT liquid scintillation counter which is equipped with most sensitive detection possibilities and α/β-discrimination. The Quantulus GCT reaches unsurpassed sensitivity using TR-LSC, BGO guard detectors, and the patented GCT method. Alternatively, Tri-Carb liquid scintillation counters equipped with ULLCM might be acceptable, but counting times for some nuclides can be significantly longer.

In addition to hardware sensitivity, a good scintillation cocktail is required to obtain accurate measurement of low activity levels. An appropriate scintillation cocktail should have low amounts of natural radioactivity, high sample uptake capacity, stability against mineral acids, good α/β-discrimination properties, high quantum yield for the conversion of the decay energy into light, and good quench stability. The scintillation cocktail we recommend for ^{3}H and gross α/β-measurement is Ultima Gold LLT. For the extraction of ^{222}Rn, Ultima Gold F is the preferred cocktail.

An overview of gross α/β-, ^{222}Rn, and ^{3}H measurements

100-150 mL Water

1. Filter if necessary
2. Acidify with HNO_3 to pH 1.7

Extract ^{222}Rn with Ultima Gold F

Direct measurement

Counting α-activity after 3 h in glass vial/foil or Teflon coated vials (^{222}Rn, ^{218}Po, ^{214}Po) with α/β-discrimination on TriCarb, typically without discrimination on Quantulus GCT for lower background and higher efficiency using GCT. In cases where high Uranium or ^{226}Ra activities are expected, the organic phase will be separated in a separation funnel. Triplicates are recommended since loss of ^{222}Rn is difficult to avoid.

Distill almost to dryness, avoid formation of solid salts

Use distillate

8 mL of distillate plus 12 mL Ultima Gold LLT for determination of ^{3}H.

In cases where iodine is expected, $Na_2S_2O_3$ is to be added before distillation.

Dissolve residue with 8 mL HNO_3 in water of pH 1.7 and add 12 mL Ultima Gold LLT

The total indicative dose should be below 0.1 mSv/a. If the gross α-activity and gross β-activity are less than 0.1 Bq/L and 1.0 Bq/L respectively, the Member State may assume that the indicative dose (ID) is less than the parametric value of 0.1 mSv and radiological investigation is not needed.

α/β-measurements with optimized PSA settings.

If the total indicative dose determined with LSC is above 0.1 mSv/a, the measurement of individual nuclides might be necessary to determine the origin of the elevated dose.

Sampling collection and preparation

All water samples should always be analyzed by laboratories following ISO/IEC 17025[5] which details organizational and management requirements, handling of complaints and nonconforming testing, corrective actions, preventive actions, documentation, as well as technical requirements such as instrumentation, methods, and qualified personnel.

It is important to note that the simple act of collecting a water sample can trigger errors in results, therefore, special care should be taken during the collection process especially for the gross α/β-measurement and the determination of ^{222}Rn in water.

The water collected should be a representative sample taken from either a water reservoir, tap water, or other source such as bottled water. Multiple samples taken from the same location, date, and time should give reproducible results. Careful documentation of important parameters such as location, date, time, weather conditions, and other important parameters that could have an influence on the produced data is required. The appropriate sampling frequency is also something that must be considered. In some cases, the activity concentration of radionuclides in water might be stable, allowing the analysis of only a few water samples per month or even per year. However, in other cases, activity concentrations might be subject to continuous variations over time and/or space and in such cases, assessment of the activity concentrations should also be continuous and special sampling programs[6] should be in place to consider possible variations. This could be done through programs that estimate statistical sampling errors and with sampling procedures which eliminate or minimize changes in the activity concentrations of nuclides by the sampling process itself or during the time between sampling and analysis in the liquid scintillation counter.

When the analysis will also include the determination of ^{222}Rn, the highest risk to the sample integrity is loss of radon gas. The solubility of radon in water is very limited and heating, turbulences in the water either during sampling or in the final container, especially plastic container, can result in degassing of the water sample and the loss of radon. For this reason, water samples should always completely fill sample container leaving no space for gaseous radon. In addition, it is important that the process of sampling does not create any turbulence. To accomplish this, the water should slowly flow into the container and if tap water is being sampled, the flow of water should be slow to avoid any turbulence. When the collected sample is from tap water, the water should be run for some time before being collected to remove the old water from the pipes and tubing. A good indication that the water was run for long enough is the temperature of the water or the electrical conductivity, which should give constant values before starting the sampling procedure.

In most cases, obtaining the gross α/β-measurements will require the transport of the samples back to a laboratory that houses the instrumentation required for analysis. Therefore, it is important that the integrity of the collected samples remains constant from moment of collection through preparation for analysis. Because loss of radon is difficult to avoid, it is recommended that duplicate or triplicate samples are taken, and measurements are obtained, to ensure stable and reproducible results. The collection container should not be made from polyethylene given that some cocktails, and even radon itself, can diffuse through polyethylene. In cases where radon measurement will be included the analysis, it is recommended that a glass collection container with a tight glass stopper be used for sample collection.

Details of the sampling process, integrity of samples, sample preservation, sample storage and storage time, reagent and materials, storage container, transport and sampling report are discussed in detail in ISO 5667-3.[7] Possible errors during the sampling process and, quality control mechanisms to avoid these errors

(such as technical and staff requirements including training of sampling staff, standard operating procedures, strategy and organization of the sampling process) are discussed in detail in FprEN ISO 5667-14:2016.[8]

According to ISO 5667-3,[7] filtration of a water sample, especially for ground water, river water or sea water samples, should be done on-site with a 0.4-0.45 µm filter. If for some reason the filtration of such sample is not done on-site, a reason as to why should be mentioned in the sampling report. For gross α/β- or ^{3}H-measurements on-site filtration is doable and well-advised. For ^{222}Rn measurement, a filtration step should not be done before extraction of radon into an organic cocktail, due to the likelihood of loss of radon during a filtration step.

The delay between sample collection and sample analysis in the liquid scintillation counter needs to be consistent and carefully documented, if half-life correction is necessary which is often the case for ^{222}Rn analysis. For ^{222}Rn analysis, there should be less than a day between sample collection and analysis. For all analysis, the sample should be cooled and acidified to pH < 2 with nitric acid, in accordance with ISO 5667-3,[7] and stored in the dark. Acidifying with nitric acid keeps radionuclides in solution and prevents adsorption on surfaces. For this reason the filtration should only be done after adjusting the sample pH < 2.

Why is it necessary to acidify with nitric acid?
For the gross α/β-measurements, it is important to note that some nuclides tend to adsorb on surfaces such as glass surfaces and plastic surfaces. Therefore, in most cases it is necessary to acidify the sample directly after the sampling process to keep all nuclides in solution. This is especially true for the measurements of actinides. An investigation of the adsorption of ^{241}Am by Lovett et al.[9] in normal and acidified sea water samples demonstrated a loss of up to 50% of activity of ^{241}Am due to adsorption on the surface of the container in normal water. Acidifying water samples can also help to dissolve nuclides which are already adsorbed to the surface of the container material. Bernabee et al.[10] and Milkey[11] demonstrated that the removal of metal cations is mainly due to intermolecular forces, so-called van der Waals interaction. Acidifying with nitric acid until the pH value is 1.7 can avoid adsorption of nuclides on the container surface.

The use of the liquid scintillation counter for the determination of activity concentrations of gross α/β-nuclides, ^{3}H and ^{222}Rn also involves working with standards. Standards typically contain much higher activities than the activities present in water samples intended for human consumption. In many cases, the activity of the standards will be several orders of magnitude higher than in environmental samples. This introduces the possible risk of cross contamination. To avoid such contaminations, handling of standards should be done in areas that are not used for unknown samples. Pipettes and other laboratory equipment should not be used for both the unknown water samples and for standards without careful cleaning between the individual steps. If possible, disposable tips with filters should be used. Working with tritiated water should be done under a fumehood to avoid any contamination of the sample by tritiated water via air transportation.

Calibration and activity determination of the gross α/β-measurement[2]

Please note, this application requires a liquid scintillation counter with α/β-discrimination. While this feature is standard on the Quantulus GCT, it is an optional add-on on all other Revvity instruments (with the exception of the Tri-Carb 2810 and 4810TR, and some older instruments of the Tri-Carb series). Before you start, it is important to make sure this option is installed on your instrument if you are using a Tri-Carb model.

A pure α- and β-standard is required for the determination of the efficiency of α- and β-nuclides and for the determination of the best discriminator

setting to distinguish α- and β-nuclides. The standards should contain a count rate between 50,000 and 80,000 CPM but not less than 30,000 CPM. Very often ^{90}Sr and ^{241}Am are used. The standards must be calibrated and certified standards (for example NIST traceable standards or comparable sources) with known activity, precision, and standard errors. In cases where ^{90}Sr is used as β-standard, the activity on the certificate is typically based on ^{90}Sr, which means the standard sample has twice the activity mentioned on the document due to equilibrium with ^{90}Y. Both standards must have same pH-and tSIE (Transformed Spectral Index of the External Standard) values, which will be measured for the unknown samples. Therefore typically nitric acid has to be added to commercially available standards to set the pH value to 1.7. Also, to compensate for the quenched background, a background sample must be prepared from dead water with the same tSIE as the unknown sample. All samples should be adjusted to a pH of 1.7 using nitric acid. A pH meter controlling the pH value is sufficient in most cases to make sure all samples have same tSIE, unless other strong quenchers are present in the sample. For further accuracy, a simple CPM measurement with activated tSIE can be done to determine the tSIE of each sample. For maximum sensitivity, plastic vials or Teflon coated plastic vials with Ultima Gold LLT should be used. In most cases 8-9 mL of water plus 12 mL of Ultima Gold LLT can be used.

Before the measurement of unknown samples, it is necessary to determine the PSA level for the α/β-discrimination in an α/β-standards assay. This assay type is available in all Quantulus GCT and Tri-Carb models using the QuantaSmart software with the α/β-option installed. Running the standards assay will determine the optimum discrimination level to allow simultaneous detection of α– and β-emitters. The count rate of the α– and β–standard should at least give between 50,000-80,000 CPM to ensure good counting statistics within the short counting time of a few minutes maximum.

To run the α/β-standards assay

1. Define an α/β-standards set in the α/β-standards library.
2. Determine the weight of an empty scintillation vial in an analytical balance and pipette the required amount of β-standard into a scintillation vial.

Note: It is important to ensure that both the scale and the pipette used are both precise and calibrated.

3. Weigh again to exactly determine the amount of added β-standard.
4. Add Ultima Gold LLT until the vial is full.
5. Repeat the procedure for the β-standard

Note: Should recently calibrated ^{90}Sr/^{241}Am standards be used, containing 4 kBq/g of activity, ~0.15 g ^{90}Sr should be added into the vial for the β-standard and 0.3 g of ^{241}Am standard into the vial for the α-standard.

6. Position the pure β-standard in position 1 and the pure α-standard in position 2 of the sample cassette.
7. Make sure the instrument is in Normal Count Mode. The standards assay can only be done in normal count mode, even if you later want to use the low-level mode.
8. Consider other settings.
   - PSA level can be between 0-255.
   - Automatic mode will scan the entire range automatically, but this might require some time depending on the number of points.
   - Histrogram mode will be the fastest mode and the best way to further optimize performance. It only requires one measurement per standard sample and allows working with dual discriminators to optimize the spill.

9. Save the protocol and link it to a protocol flag.

After you have run the α/β-standards assay the QuantaSmart software will determine the optimum PSA value for the α/β-assays of unknown samples with the same tSIE value as the standard just measured. Typically, the PSA level for drinking water samples is between 70-150, which can be set when switching to manual mode, saving some time.

To run an α/β-assay

1. Define an α/β-nuclide in the α/β-nuclide library and choose the α/β-standards set from the α/β-standards library. This will ensure that the optimal PSA value determined in the standards assay will be used for the α/β-discrimination
2. Define the required parameters.

Note: In the Quantulus GCT, the normal count mode with activated GCT Low is typically more sensitive than the measurement with GCT off.

3. If you have not entered the activity of the standards in the α/β-standards assay protocol, measure the two standards to determine the counting efficiency for α- and β-nuclides in the chosen count mode. This will be used later to determine the activity concentration of the unknowns.

Note: If you already entered the activity of the standards in the α/β-standards assay protocol, efficiencies will be calculated automatically by the software (assuming QuantaSmart software version 5.2 or later).

Note: Typically, depending on window settings, the counting efficiencies are above 90%.

4. Distill sample with volumes of ~100-150 mL to almost dryness to eliminate Radon and Tritium.

Note: Volume range listed above is typical for drinking water samples.

Note: Avoid distilling to dryness because it can be difficult to solubilize the solidified material again.

Note: Depending on the amount of salt content in the water, the sample volume may be less than 150 mL to allow homogenous mixing with the cocktail. Too high salt contents can limit the uptake capacity of the cocktail and can result in phase separation which is sometimes difficult to see in plastic vials. In general samples with a salt content of more than 500 mg/L might need smaller sample volumes to prepare a homogeneous solution with the cocktail.

Tip: To allow a better visual inspection of the homogeneity of the sample, a test mixture can be prepared in a glass vial.

Note: If the salt content is unknown a sample might be distilled to dryness in a flask which has been weighed before to determine the amount of residue.

5. (Optional) If the ^{3}H activity will also be determined in these samples, store them in a dark and cool place.
6. Dissolve residue using 9 mL of nitric acid of pH 1.7 and transfer to a plastic scintillation vial.
7. Add 11 mL Ultima Gold LLT scintillation cocktail to the vial.

8. Perform measurement with α/β-discrimination immediately after sample preparation, ensuring an appropriate counting window is selected.

Note: A good counting window is the window which will result in the best detection limit for this nuclide. The detection limit should be calculated according to ISO 11929.[4] An Excel macro to do this calculation can be ordered from Revvity.

Note: The counting window for β-measurement is typically 10-600 keV but this can change depending on quench. The energy window must be relatively wide open if no information about the expected radionuclides is available but can be optimized using the calibrated standards if more information about potential nuclides is available.

Note: The α-counting window typically is between 50 – 600 keV to cover all possible α-nuclides.

Note: Up to 200 minutes counting time or more depending on the instrument are necessary to reach the required detection limit of 0.04 Bq/L for gross α-measurements with very sensitive instruments such as Quantulus GCT. With this counting time, reaching a detection limit of 0.4 Bq/L is not an issue for the gross β-measurements.

9. Determine the efficiencies for α- and β-nuclides using the following equation:

$$E_{\alpha\alpha} = \frac{R_{s\alpha\alpha} - R_{0\alpha}}{a_{s\alpha}} \qquad E_{\beta\beta} = \frac{R_{s\beta\beta} - R_{0\beta}}{a_{s\beta}}$$

$E_{\alpha\alpha}, E_{\beta\beta}$ = Efficiency of α-standard in α-channel and β-standard in β-channel.

$R_{s\alpha\alpha}, R_{s\beta\beta}$ = Brutto count rate of α- or β-standard 1/s.

$R_{0\alpha}, R_{0\beta}$ = Background count rate in α- or β-window in 1/s.

$a_{s\alpha}, a_{s\beta}$ = Activity of the used α- or β-standard in Bq.

10. Correct the count rates for spill from α- into the β-channel and vice versa. To do this, first define the spill factors then determine the spill corrected count rate according to ASTM D7283-13.
The following equations can be used:

$$X_\alpha = \frac{E_{\alpha\beta}}{E_{\alpha\alpha}} \quad X_\beta = \frac{E_{\beta\alpha}}{E_{\beta\beta}} \quad E_{\alpha\beta} = \frac{R_{s\beta} - R_{0\beta}}{c_\alpha} \quad E_{\beta\alpha} = \frac{R_{s\alpha} - R_{0\alpha}}{c_\beta}$$

$$R_{c\alpha} = \frac{R_\alpha - R_\beta \cdot X_\beta}{1 - X_\alpha \cdot X_\beta} \quad R_{c\beta} = \frac{R_\beta - R_\alpha \cdot X_\alpha}{1 - X_\alpha \cdot X_\beta}$$

With

$R_{c\alpha}$ = Background and spill corrected α-count rate in 1/s.

$R_{c\beta}$ = Background and spill corrected β-count rate in 1/s.

R_α = Background corrected α-count rate = $R_{s\alpha\alpha} - R_{0\alpha}$ in 1/s.

R_β = Background corrected β-count rate = $R_{s\beta\beta} - R_{0\beta}$ in 1/s.

X_α = α- to β-spill factor.

X_β = β- to α-spill factor.

$E_{\alpha\alpha}$ = Efficiency of the α-nuclide in the α-channel.

$E_{\alpha\beta}$ = Efficiency of the α-nuclide in the β-channel.

$E_{\beta\alpha}$ = Efficiency of the β-nuclide in the α-channel.

$E_{\beta\beta}$ = Efficiency of the β-nuclide in the β-channel.

c_α = Activity concentration of the gross α-nuclides in Bq/L.

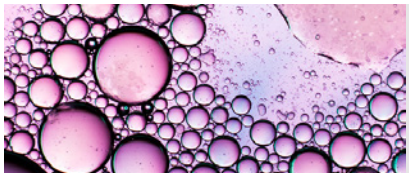
c_β = Activity concentration of the gross β-nuclides in Bq/L.

V = Sample volume in liter.

11. Determine the activity concentration for the gross α- and β-activity using the following equations:

$$c_\alpha = \frac{R_{c\alpha}}{E_{\alpha\alpha} \cdot V} \qquad c_\beta = \frac{R_{c\beta}}{E_{\beta\beta} \cdot V}$$

12. Report the results according to ISO 11929.



Example calculation of gross α/β-measurement in Drinking Water

Known data and data from the measurements

Volume of the sample	200 mL
Activity of α-standard	58408 DPM corresponding to 973.5 Bq
Activity of β-standard	56417.9 DPM corresponding to 940.3 Bq
Brutto count rate α-standard in α-channel	56132 CPM corresponding to 935.5 s^{-1}
Brutto count rate β-standard in β-channel	51345 CPM corresponding to 855.8 s^{-1}
Brutto count rate α-standard in β-channel	1348.1 CPM corresponding to 22.5 s^{-1}
Brutto count rate β-standard in α-channel	1412.3 CPM corresponding to 23.5 s^{-1}
Unknown brutto count rate β-nuclide	6.35 CPM corresponding to 0.106 s^{-1}
Background count rate in β-channel	4.72 corresponding to 0.079 s^{-1}
Unknown brutto count rate α-nuclide	5.42 CPM corresponding to 0.09 s^{-1}
Background count rate in α-channel	1.85 corresponding to 0.031 s^{-1}

Calculation of the efficiencies

$$E_{\alpha\alpha} = \frac{R_{s\alpha\alpha} - R_{0\alpha}}{a_{s\alpha}} = \frac{56132 - 1.85}{58408} = 0.961$$

$$E_{\beta\beta} = \frac{R_{s\beta\beta} - R_{0\beta}}{a_{s\beta}} = \frac{51345 - 4.72}{56417.9} = 0.910$$

$$E_{\alpha\beta} = \frac{R_{sa\beta} - R_{0\beta}}{c_{\alpha}} = \frac{1348.1 - 4.72}{58408} = 0.023$$

$$E_{\beta\alpha} = \frac{R_{s\beta\alpha} - R_{0\alpha}}{c_{\beta}} = \frac{1412.3 - 1.85}{56417.9} = 0.025$$

Calculation of the spill factors

$$X_{\alpha} = \frac{E_{\beta\alpha}}{E_{\alpha\alpha}} = \frac{0.023}{0.961} = 0.024$$

$$X_{\beta} = \frac{E_{\beta\alpha}}{E_{\beta\beta}} = \frac{0.025}{0.910} = 0.028$$

Calculation of spill corrected count rate of unknowns

$$R_{c\alpha} = \frac{R_{\alpha} - R_{\beta} \cdot X_{\beta}}{1 - X_{\alpha} \cdot X_{\beta}} = \frac{5.42 - 1.85 - (6.35 - 4.72) \cdot 0.028}{1 - 0.024 \cdot 0.028} = \frac{3.57 - 0.05}{0.9993} = 3.52\ CPM\ (0.059\ s^{-1})$$

$$R_{c\beta} = \frac{R_{\beta} - R_{\alpha} \cdot X_{\alpha}}{1 - X_{\alpha} \cdot X_{\beta}} = \frac{6.35 - 4.72 - (5.42 - 1.85) \cdot 0.024}{1 - 0.024 \cdot 0.028} = \frac{1.63 - 0.09}{0.9993} = 1.54\ CPM\ (0.026\ s^{-1})$$

Calculation of activity concentration of the unknown gross α- and gross β-activity:

$$c_{\alpha} = \frac{R_{c\alpha}}{E_{\alpha\alpha} \cdot V} = \frac{0.059}{0.961 \cdot 0.2} = 0.31\ Bq/L$$

$$c_{\beta} = \frac{R_{c\beta}}{E_{\beta\beta} \cdot V} = \frac{0.026}{0.91 \cdot 0.2} = 0.14\ Bq/L$$

Note: In some cases, above calculations are rounded below the exact report according to ISO 11929.

Reporting (according to ISO 11929)

The calculations of the characteristic parameters are based on a sample volume of 0.2 L, counting time 18000 s for sample and background, $k_{1-\alpha} = k_{1-\beta} = k_{1-\gamma} = 1.96$ relative uncertainty of standards and dilutions 0.02, uncertainty sample volume 0.0001 L.

Data for gross α-measurement	
Guideline value	0.04 Bq/L
α-activity concentration	0.303 Bq/L
Uncertainty of the gross α-activity	0.02 Bq/L
Decision threshold	0.019 Bq/L
Detection limit	0.039 Bq/L
Lower confidence limit	0.27 Bq/L
Upper confidence limit	0.33 Bq/L
Omega	1
Best estimate	0.303 Bq/L
Uncertainty of best estimate	0.02 Bq/L
Physical effect detected?	Yes
Is this a suitable method?	Yes

Data for gross β-measurement	
Guideline value	0.4 Bq/L
β-activity concentration	0.141 Bq/L
Uncertainty of the gross β-activity	0.02 Bq/L
Decision threshold	0.032 Bq/L
Detection limit	0.065 Bq/L
Lower confidence limit	0.11 Bq/L
Upper confidence limit	0.18 Bq/L
Omega	1
Best estimate	0.141 Bq/L
Uncertainty of best estimate	0.02 Bq/L
Physical effect detected?	Yes
Is this a suitable method?	Yes

Calibration and activity determination of the ^{222}Rn measurement[12]

Typically, this measurement is done in a simple CPM assay as described in the QuantaSmart reference manual. For ^{222}Rn measurements, no quench curve is required since the counting efficiency and calibration factor will be determined from a known activity of a ^{226}Ra source.

A background sample is required and should be processed using the same preparation steps as is used for the unknown sample. Should dead water not be available, a drinking water sample can be used which is free of Radon. Radon can be removed from the sample by heating the water for 10 minutes or by bubbling nitrogen through the background sample for 30 minutes.

It is advisable to measure both the sample and background multiple times to assure stable conditions. The measurement cycles should include the unknown and background sample in every cycle. In case you suspect the existence of other α-nuclides you can measure the sample again after three days and should see a decrease of the count rate according to the half-life of radon. All the following equations are based on the method using separation funnel and measurement with α/β-discrimination.

1. Use 15 mL of a ^{226}Ra source (for example NIST traceable standards or comparable sources) in a 20 mL glass scintillation vial or 20 mL Teflon coated plastic vial.
2. Add approximately 5 mL of scintillation cocktail Ultima Gold F to completely fill the vial.
3. Tightly close the vial and place it upside down (to avoid loss of ^{222}Rn) until equilibrium (30 days).
4. Vigorously shake the sample (time t_s).
5. Set the optimized window.

Note: Typically, an optimized window from 20 to 850 keV can be used for all nuclides, though this might vary depending on the quench.

Note: It is important that all samples (the ^{226}Ra standard, the background sample and the sample with unknowns) have the same tSIE value. The tSIE value of all samples can be determined with a short CPM measurement using tSIE as quench indicating parameter. Deviation of the quench level can be adjusted with a quenching agent such as nitromethane if necessary.

6. Measure the sample with a simple CPM assay

Note: It is recommended that ^{222}Rn be measured after three hours in order to have very high counting efficiency. Counting efficiency of up to more than 300% can result from the three fast in growing α-nuclides (^{222}Rn, ^{218}Po and ^{214}Po) and the two fast in growing β-nuclides (^{214}Pb and ^{214}Bi).

Note: During the three hours between shaking and counting, the sample should be kept in a cool and dark location. If you are unable to begin measurement after three hours, keep the sample cool and in the dark with the cap of the scintillation vial upside down.

7. Determine the calibration factor using the following equation:

$$\varphi_A = \frac{a_s \cdot V_s}{R_s - R_0} \cdot e^{-\lambda t_A} = \frac{a_s \cdot V_s}{R_s - R_0} \cdot e^{-ln2 \cdot \frac{t_A}{t}}$$

With:

φ_A = Calibration factor in Bq * s.

a_s = Activity concentration of the ^{226}Ra standard in Bq/L.

V_s = Volume of the used standard solution in L.

R_s = Brutto count rate of the standard solution in 1/s.

R_0 = Background count rate in 1/s.

λ = Decay constant of ^{222}Rn in 1/s.

t = Half-life of ^{222}Rn in s.

t_A = $t_m - t_s$ = time between shaking of the standard solution with cocktail and the end of the measurement t_m in s.

8. Calculate the activity concentration of ^{222}Rn using the following equation.

$$c_{Rn} = \frac{\varphi_A \cdot (R_b - R_0)}{V} \cdot e^{ln2 \cdot \frac{t_A}{t}} = \frac{\varphi_A \cdot (R_b - R_0)}{V} \cdot e^{\lambda \cdot t_A}$$

With:

c_{Rn} = Activity concentration of ^{222}Rn in Bq/L.

R_b = Brutto count rate unknown in 1/s.

R_0 = Background count rate in 1/s.

V = Volume of the sample in l.

t_A = Time between sampling and Measurement in s.

φ_A = Calibration factor in Bq * s.

λ = Decay constant of ^{222}Rn in 1/s.

t = Half-life of ^{222}Rn in s.

The detection limit according to the council directive for ^{222}Rn is 10 Bq/L. Typically the measurement time to reach the required detection limit is clearly below 30 minutes, in most cases close to 10 minutes in a Tri-Carb with α/β-discrimination and clearly below five minutes in a Quantulus GCT.

Example calculation for the determination of ^{222}Rn activity in Drinking Water

Calculation of the calibration factor

Activity of the ^{226}Ra standard	60,000 Bq/L
Volume of the standard used	0.015 L
Brutto count rate of the standard	3175 s^{-1}
Background count rate	0.070 s^{-1}
Time between shaking and end of measurement	12600 s (= 3.5 h)

$$\varphi_A = \frac{a_s \cdot V_s}{R_s - R_0} \cdot e^{-\lambda t_A} = \frac{60000 \cdot 0.015}{3175 - 0.07} \cdot e^{-2.1 \cdot 10^{-6} \cdot 1.26 \cdot 10^4} = \frac{900}{3174.93} \cdot e^{-0.026} = 0.276\ Bq \cdot s$$

Calculation of the activity concentration of ^{222}Rn in the unknown water sample

Brutto count rate unknown	0.71 s^{-1}
Volume of the sample	0.015 L
Background count rate	0.07 s^{-1}

R_0 = 0.07 s^{-1}
V = 0.015 L
λ = 2.1 · 10^{-6} s^{-1}
t = 3.3 · 10^5 s (= 3.82 d)
t_A = 2.45 · 10^5 s (= 2.8 d)
t_m = 600 s

$$C_{Rn} = \frac{\varphi_A \cdot (R_b - R_0)}{V} \cdot e^{\lambda \cdot t_A} = \frac{0.276 \cdot (0.71 - 0.07)}{0.015} \cdot e^{2.1 \cdot 10^{-6} \cdot 2.45 \cdot 10^5} = 11.778 \cdot e^{0.5145} = 19.70\ Bq/L$$

Reporting *(according to ISO 11929)*

The report according to ISO 11929 for the ^{222}Rn measurement is based on a counting time of 100 minutes for sample and background, a sample volume of 0.015 L, $k_{1-\alpha} = k_{1-\beta} = k_{1-\gamma} = 1.96$, relative uncertainty of the ^{226}Ra standard = 0.02, uncertainty of the sample volume 0.001 L but an estimated uncertainty of 20% regarding possible deviations due to loss of ^{222}Rn from the average of triplicate measurements.

Data for ^{222}Rn-measurement	
Guideline value	10.0 Bq/L
^{222}Rn activity concentration	19.704 Bq/L
Uncertainty of the gross α-activity	4.351 Bq/L
Decision threshold	0.291 Bq/L
Detection limit	0.740 Bq/L
Lower confidence limit	11.176 Bq/L
Upper confidence limit	28.231 Bq/L
Omega	1
Best estimate	19.704 Bq/L
Uncertainty of best estimate	4.351 Bq/L
Physical effect detected?	Yes
Is this a suitable method?	Yes

Calibration and activity determination of the ^{3}H measurement

1. The unknown water sample should be distilled and requires at least 10 mL of sample.
   - 100 - 200 mL in case gross α/β-measurements have to be done later.
   - To eliminate all the radon dissolved in water. Other α- and β-nuclides should stay in the residue and ^{3}H should be in the distillate after the distillation.
   - In case ^{222}Rn has to be determined in the same sample, an extraction of Rn with an extracting cocktail such as Ultima Gold F has to be done before the distillation as described above.
   - In case you expect elemental iodine isotopes in the water sample, take an aliquot of the water sample (10 mL) and add 0.2 g of sodium thiosulfate ($Na^{2}S^{2}O^{3}$) to reduce elemental iodine to the iodide. The iodide will stay in the residue and will not be distillated.
2. Take 9 mL of the distillate and transfer it to a 20 mL plastic scintillation vial.
3. Add 11 mL of Ultima Gold LLT cocktail.
4. Shake the sample vigorously to ensure a homogenous solution.
5. Keep the sample cool and in the dark for some time prior to counting. It is recommended to keep the samples overnight before starting the counting process.
6. Count using one of the two options detailed below.

Option 1: Using a quench curve

The utilization of a quench curve is the preferred method to measure ^{3}H activity even though many water samples have very similar quench levels (same tSIE) corresponding to a constant efficiency. By using a DPM protocol with quench curve, the fully automatic determination of the ^{3}H activity, including reporting according to ISO 11929, can be accomplished.

Quench curve: ^{3}H quenched sets are commercially-available from Revvity or can be prepared by the customer based on calibrated and certified standards (for example NIST traceable standards or comparable sources) with known precision and standard errors. A quench standards assay is performed using the commercially-available or self-made standards to prepare a quench curve with a graph counting efficiency versus tSIE value.

DPM assay: Once the quench curve is saved, the unknown ^{3}H samples can be measured in a single DPM assay using both the saved quench curve and the tSIE quench indicating parameter in a fully automatic protocol. The QuantaSmart software will automatically report the activity of ^{3}H in DPM or Bq. If the instrument used is equipped with the ISO 11929 macro and Excel, an automatic report according to ISO 11929 is possible. This report contains the activity, the uncertainty of the activity, decision threshold, detection limit, best estimate, uncertainty of best estimate, and confidence limits of the sample. If the ISO 11929 macro is used, it is important to carefully read the requirements of this macro and the settings necessary in the QuantaSmart Software in the separate ISO 11929 macro manual.

Option 2: Internal standard method

This method does not require the preparation of quench curves; however, it also does not allow fully automatic reporting with all the documentation mentioned for the use of quench curves. Instead, the activity concentration must be determined using the equations outlined later within this guide. Calculation and reporting with the Revvity Excel macro according to ISO 11929 is possible- but this is because the macro allows manual import of data.

If the distilled water samples have tSIE values in a small tSIE range, one can assume constant quench. When this is the case, ^{3}H efficiency can be determined once with the internal standard method and the determined efficiency can be used for all other distilled water samples to calculate the activity of ^{3}H. When this is the case, it is required that the tSIE of the sample is always documented to assure constant quench. If the tSIE has large variations (> 5%), a quench curve is the preferred alternative.

When using the internal standard method, only a simple CPM assay is required:

1. Transfer 9 mL of distilled unknown water sample to a vial.
2. Add 11 mL Ultima Gold LLT to vial.
3. Measure (CPM_s).
4. Add a known amount of activity (DPM_{st}) to the unknown sample.

Note: To avoid changing the quench of the sample, the addition of the ^{3}H water sample with known activity should be a small volume (in the µL range), as larger volumes of water can have an influence on the quench level in the sample.

5. Measure (CPM_{s+st}).

Note: Using these two measurements we can determine the ^{3}H efficiency and the activity of the unknown sample.

6. Calculate efficiency using the following formula: 7.

$$E_{3_H} = \frac{CPM_{s+st} - CPM_s}{DPM_{st}}$$

Where:

E_{3_H} = ^{3}H efficiency.

CPM_s = First background-corrected count rate of the unknown sample.

CPM_{s+st} = Second background-corrected count rate of sample plus added standard.

DPM_{st} = Known decay rate of the added standard.

7. Calculate ^{3}H activity using the following formula:

$$DPM_s = \frac{CPM_s}{E_{3_H}}$$

Where:

E_{3_H} = ^{3}H efficiency.

CPM_s = First background-corrected count rate of the unknown sample.

DPM_s = Decay rate of the unknown ^{3}H water sample.

Example calculation for the determination of ^{3}H activity in Drinking Water

Calculation for the determination of ^{3}H activity in Drinking Water

Sample volume	0.008 L
Background count rate	0.95 CPM corresponding to 0.01583 s^{-1}
Brutto count rate sample	4.45 CPM corresponding to 0.07417 s^{-1}
Brutto count rate sample + standard	117.95 CPM corresponding to 1,966 s^{-1}
Decay rate of the added standard	410 DPM corresponding to 6.83 Bq

$$E_{^3H} = \frac{CPM_{s+st} - CPM_s}{DPM_{st}} = \frac{117.0 - 3.5}{410} = 0.277 \text{ or } 27.7\%$$

$$DPM_s = \frac{CPM_s}{E_{^3H}} = \frac{3.5}{0.277} = 12.64 \frac{DPM}{8} \text{ mL or } 0.211 \text{ Bq/8 mL corresponding to } 26.32 \text{ Bq/L}$$

Reporting *(according to ISO 11929)*

The following report according to ISO 11929 is based on a sample volume of 0.008 L, a counting time of 6000 s for unknown sample and background, $k_{1-\alpha} = k_{1-\beta} = k_{1-\gamma} = 1.96$, (coefficients for the quantiles of the normal distribution), relative uncertainty of standards and dilutions 0.02, uncertainty of the sample volume 0.0001 L, relative uncertainty of quench curve 5%, and relative uncertainty of quench parameter 5%.

Data for ^{3}H_measurement	
Guideline value	10.0 Bq/L
^{3}H activity concentration	26.324 Bq/L
Uncertainty of the gross ^{3}H-activity	2.680 Bq/L
Decision threshold	0.2032 Bq/L
Detection limit	4.455 Bq/L
Lower confidence limit	21.071 Bq/L
Upper confidence limit	31.576 Bq/L
Omega	1
Best estimate	26.324 Bq/L
Uncertainty of best estimate	2.680 Bq/L
Physical effect detected?	Yes
Is this a suitable method?	Yes

Materials needed for gross α/β- measurements, ^{3}H and ^{222}Rn analysis

- Ultima Gold F, Revvity, 6013171
- Ultima Gold LLT, Revvity, 6013371
- Dispensette for cocktail volumes 1-10 mL, Revvity, 6005362
- Dead water (or Rn free drinking water), Eckert and Ziegler, BGW-1LB
- ^{226}Ra standard, PTB, Braunschweig, Germany, (https://www.ptb.de/cms/ptb/fachabteilungen/abt6/fb-61/611-aktivitaetseinheit/katalog-aktivitaetsnormale.html)
- ^{90}Sr PTB, Braunschweig, Germany
- ^{241}Am standard PTB, Braunschweig, Germany
- ^{3}H internal standard, Revvity, 6004052
- ^{3}H quenched, low level Ultima Gold standards, 20 mL, Revvity, 6010704
- ^{3}H quenched Ultima Gold standards, 20 mL, Revvity, 6007600
- Quantulus GCT 6220 liquid scintillation counter or at least a Tri-Carb with Ultra Low Level Count mode, Revvity
- ISO 11929 Macro for QuantaSmart, Revvity, 7005435
- MS Excel (only if ISO 11929 macro is required), Microsoft
- Low potassium 20 mL glass vials with foil-lined cap, Revvity, 6000128
- Teflon-coated plastic vials, Revvity, 6000477
- Caps for septum (for glass vial 6000128), Revvity, 6000239
- Rubber septum (for glass vial 6000128), Revvity, 6000240
- Separation funnel (50 mL, 250 mL)
- Analytical balance (precision 0.1 mg)
- Distillation apparatus
- Injection needles, pipettes (µL and 15 mL volume)
- Nitric acid p. A. (HNO_3)
- Sodiumthiosulfate ($Na_2S_2O_3$) in case Iodine might be in the ^{3}H-sample
- pH meter
- Standard laboratory equipment
- Nitromethane as for preparation of quenched curves or to adjust quench levels
- On-line training support (two hours), Revvity, ED023967
- On-site training & support, Revvity, N0236524, N0236525, N0236526, N0236527, N0236528 (depending on the time required for the training)

Abbreviations and definitions

BGO Bismuth germanate ($Bi_4Ge_3O_{12}$), material with crystal structure and scintillator properties used as guard detector in the Quantulus GCT

Bq Decay per second, official SI unit for the activity in a sample
1 Bq = 60 DPM

Cocktail Mixture of chemicals used to convert the energy of a radionuclide into light photons

CPM Counts per minute, raw counts detected by the instrument

DPM Decay (Disintegrations) per minute. Background and efficiency-corrected CPM, giving the activity of the sample:

$$DPM = \frac{(CPM\text{-}Background)}{Efficiency}$$

Efficiency Portion of the Total Activity which could be detected by the instrument

GCT Guard Compensation Technology, a very efficient patented technology used to reduce the background in the Quantulus GCT. Most sensitive technology for the detection of radionuclides offered by Revvity

LSC Liquid Scintillation Counter (Counting)

MCA Multi-Channel Analyzer

PDA Pulse Decay Analysis

PSA Pulse Shape Analysis

SLLCM Super Low-Level Count Mode, special count mode for very sensitive measurements available on the Quantulus GCT

TR-LSC Time Resolved-Liquid Scintillation Counting

ULLCM Ultra Low-Level Count Mode for sensitive detection of radionuclides

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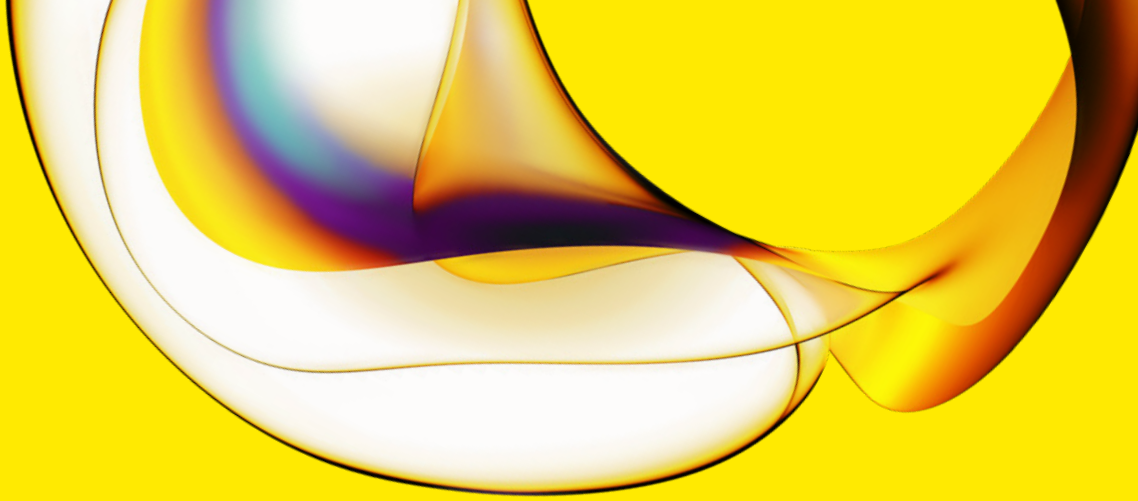

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