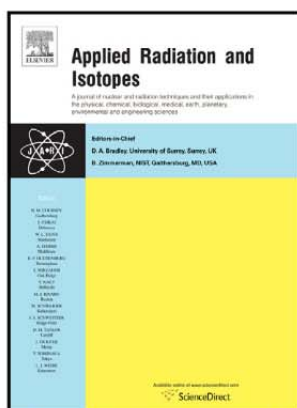


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www.elsevier.com/locate/apradiso

PII: S0969-8043(13)00142-5
DOI: <http://dx.doi.org/10.1016/j.apradiso.2013.03.052>
Reference: ARI6180

To appear in: *Applied Radiation and Isotopes*

Cite this article as: D. Calmet, S. Jerome, R. Ameon, A. Bombard, M. Forte, M. Fournier, M. Herranz, P. Kwakman, M. Llaurodo, S. Tokonami, ISO standards on test methods for water radioactivity monitoring, *Applied Radiation and Isotopes*, <http://dx.doi.org/10.1016/j.apradiso.2013.03.052>

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ISO STANDARDS ON TEST METHODS FOR WATER RADIOACTIVITY MONITORING

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Abstract

Water is vital to humans and each of us needs at least 1.5 litres of safe water a day to drink. Beginning as long ago as 1958 the World Health Organization (WHO) has published guidelines to help ensure water is safe to drink. Focused from the start on monitoring radionuclides in water, and continually cooperating with WHO, the International Standardization Organization (ISO) has been publishing standards on radioactivity test methods since 1978. As reliable, comparable and 'fit for purpose' results are an essential requirement for any public health decision based on radioactivity measurements, international standards of tested and validated radionuclide test methods are an important tool for production of such measurements.

This paper presents the ISO standards already published that could be used as normative references by testing laboratories in charge of radioactivity monitoring of drinking water as

well as those currently under drafting and the prospect of standardized fast test methods in response to a nuclear accident.

Introduction

In July 2010, the United Nations General Assembly expressed concern that approximately 884 million people lack access to improved water sources (UN General Assembly, 2010) and that in 2032 there may be a billion people more on this planet among whom water resources will have to be shared. To preserve water quality, therefore, water resources will have to be managed better at the planetary scale taking into account their potential conflicting uses. To ensure water is potable, its monitoring through chemical and bacteriological analysis is carried out regularly from its source to the tap all over the planet. This is done to evaluate its quality parameters in order to assess the impacts of human activities such as agriculture, industry, tourism on water resources to ultimately evaluate the consequences on health of the ingestion of drinking of water potentially contaminated or polluted. In 2011, WHO published the fourth edition of its guidelines for drinking-water quality (WHO, 2011). This builds on over 50 years of WHO guidance on drinking-water quality, starting with the publication of the first International Standards for drinking water in 1958 (WHO, 1958). The WHO guidelines are based on the assumption that monitoring environmental quality and the protection of human health are inseparable. They are considered an authoritative basis for the setting of national regulations and standards for water safety in support of public health, including protection against ionizing radiation. These latest guidelines are in line with the system of radiological protection, progressively developed by the International Commission on Radiological Protection (ICRP) with the increasing use of nuclear energy. This system is based on the assumption that any exposure to radiation involves some level of risk, and recognizes as well the link between environmental

radioactivity and public health. ICRP and WHO recommend a dose limit for the ingestion of drinking water with derived activity concentration limits for radionuclides that are referred to national regulations. National stakeholders on nuclear issues, such as industry, control authorities, local associations and public information commissions, are also linked with international stakeholders. Legal instruments require stakeholders to be informed of radioactivity levels in emissions (IAEA, 2002) and to furthermore check the radioactivity level for internationally traded foods (WHO, 2006). In Europe States must inform the European Commission at appropriate intervals about the measures they take and the radioactivity levels they have measured (Council Decision 87/600/Euratom, 1987). Health protection is a matter of concern and countries are more likely to place their trust in the quality of radioactivity data exchanged since they mutually recognize the services performed by accredited laboratories using common standards. Thus testing laboratories that carry out radionuclide activity measurements required by national authorities must obtain specific accreditation for radioactivity measurement on food and/or drinking-water samples and use International Standards to do so. This paper presents the ISO standards that give guidance to testing laboratories at the different stages from the sampling planning to the transmission of the test report to their customers.

Data quality objective

The international approach proposed by WHO to assess the safety of drinking-water with respect to its radionuclide content is to set up radionuclide screening levels and guidance levels, expressed as activity concentration, based on an annual dose derived from an annual risk of cancer. Such a dose of one-tenth of the dose limit for an individual of the general public not exposed to radiation in the workplace recommended by the ICRP (ICRP, 2000) was considered as a very low level of risk that is not expected to give rise to any detectable adverse health effect. This value of 0.1 mSv/y for one year's consumption of 2 L/d of

drinking water, regardless of the origin of the radionuclides, natural or man-made, is referred to as the individual dose criterion (IDC) also called the Total Indicative Dose (TID) in the EC Drinking Water Directive (EC, 2012). In the USA, in the same approach, the Code of Federal Regulations stipulates that if two or more radionuclides are present in drinking water, the sum of their annual dose equivalent to the total body or to any organ shall not exceed 0.04 mSv/y (4 mRem/y in the USA, Code of Federal Regulations, section 40 § 141.16.).

In most drinking water, since the usual activity concentrations of individual radionuclides are low and their determination is time consuming, detailed analysis is normally not justified for routine monitoring. WHO therefore recommends the use of a preliminary screening procedure to determine the gross (total) alpha and beta radiation emitted by all radionuclides in water before identifying any specific radionuclide. When test results are below the screening levels of 0.5 Bq/L for gross alpha activity and 1 Bq/L for gross beta activity, there is no need for specific radionuclide measurement as such concentrations will result in an individual dose well under 0.1 mSv/y. If either of the screening levels is exceeded, then the specific radionuclides producing this activity should be identified and compared to their respective guidance levels (GL) provided in the WHO guidelines. Depending on State regulations in force, these guidance levels are also called reference concentrations (RF) (EC, 1998), Maximum contaminant level (MCL in the USA, Code of Federal Regulations, section 40 CFR § 141.2) and Maximum acceptable concentrations (MAC in Health Canada, 2009). The GL are derived activity concentrations computed by WHO using the latest dose coefficient published by ICRP (ICRP, 1996) or previous ICRP publications by the USA (USA, 1959). Examples of the GL, RC and MCL for the radionuclides most commonly detected in drinking water are presented in the following Table 1.

The WHO guidance levels for radionuclides in drinking-water varies from 0.1 Bq/L for ^{210}Po to 10 000 Bq/L for ^3H . These values can be modified by national legislation of countries with

nuclear facilities (industrial, medical, research and military), usually lowered, such as in the case of tritium for which the reference level is set up at 740 Bq/L in the USA or at 100 Bq/L by the EC. The range of the different permissible activity concentration places constraints on the selection of measurement procedures and equipment for testing laboratories in charge of the radioactivity monitoring of drinking water.

This context justifies a performance-based approach with analytical protocols that are selected based on their ability to detect the radionuclides and quantify their activity concentrations above the GL that can also be considered as “action levels” for decision-maker. Thus, the decision-maker in comparing the test results with the reference concentration or maximum contaminant level will decide that, either the drinking water is fit for consumption from a radiological viewpoint or that there is a need to implement remedial measures or to place some restriction on the use of the water supply for drinking purposes.

As decisions are to be made about individual drinking water samples, the detection capability of the test protocol is an important method performance characteristic expressed in the Measurement Quality Objectives (MQO) as a required detection limit (DL). DL are thus established, either by the national authority or by the testing laboratory in charge of the measurement to ensure that the DL will be always be less than the radionuclide GL usually with a confidence level of 95% that the radionuclide is not present at a concentration greater than its DL. As it often occurs that drinking water contains several natural radionuclides, such as those belonging to uranium, thorium and actinium series, likely to be present at concentrations approaching their respective GL (such as inter alia ^{210}Po) and contributing simultaneously to the IDC, some states such as Canada, considered that any testing procedure should aim to achieve an DL lower than 20% of the GL of any radionuclide likely to be present in the water. The EC recommends that the DL for gross alpha and gross beta activities are 40% of the screening values of 0.1 and 1.0 Bq/L respectively, for radon and for

tritium is 10% of its parametric value of 100 Bq/L, and that the DL for the first check for ^{228}Ra shall be 0.02 Bq/L. The low value of the DL for ^{238}U of 0.02 Bq/L is justified by the chemotoxicity of uranium (EC, 2012).

DL is used initially for the selection of test methods by testing laboratory in charge of radioactivity monitoring of water and thus was also a key element in selecting the test methods to be published as an ISO standard for monitoring the radioactivity in water.

Standards as reference documents

Standards on test methods for radionuclides are reference documents and meet the technical concerns that arise repeatedly in the relations between economic, scientific, technical and social stakeholders, both nationally and internationally. During a controversial situation on the impact assessment of a nuclear plant, stakeholders are likely to carry out measurements on samples collected from the same sites. During an accidental situation, stakeholders that are the countries exporting and importing foods such as beverages could carry out measurements on the same cargo samples to check the radioactivity level (WHO, 2006). It is essential that stakeholders use agreed and appropriate methods and procedures for the sampling, handling, transport, storage and preparation of test samples, the test method, and for calculating measurement uncertainty. In this framework, the normative approach based on international standards aims to ensure the accuracy or validity of the test result through calibrations and measurements traceable to the International System of Units. This approach guarantees that radioactivity test results on the same types of samples are comparable over time and between different test laboratories.

In the large set of standards that can be used by testing laboratories, there are horizontal standards of generic interest for metrology considered at large and those on characterizing

the matrix measured: the water: These two sub-sets of specific standards on nuclear metrology and on the radioactivity test methods for water samples have to be considered by the laboratory specifically in charge of the monitoring of drinking waters.

Generic Standards on Metrology

Regarding the generic standards that will help testing laboratory manage the measurement process, there is ISO 10012 (2003) that specifies generic requirements and provides guidance for the management of measurement processes and metrological confirmation of measuring equipment used to support and demonstrate compliance with metrological requirements and ISO/IEC 17025 (2005) that specifies the general requirements for the qualification to carry out tests and/or calibrations, including sampling, both companion standards of the ISO 9001 (2008). The additional requirements in ISO 17025, as opposed to ISO 9001, include participation in proficiency testing (see ISO 17043 (2010)), adherence to documented, validated, methodology and specification of technical competence, especially on the part of operating laboratory personnel. It also requires that the test laboratory establishes traceability of its own measurement methods and measuring instruments to the International System of Units (SI for *Système international d'unités*) by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. There is also a difference in the method of scrutiny of laboratories under ISO 9001 as compared to ISO 17025 assessments (UNIDO, 2009). One standard of importance is ISO/IEC Guide 98 (2008) drafted by the Joint Committee for Guides in Metrology (JCGM), that establishes general rules for evaluating and expressing uncertainty in measurement that can be followed at various levels of accuracy and applied with the related 5 parts of ISO 5725 (1994) that provides the general principles necessary to accurately assess

measurement methods, results, applications and practical estimations. JCGM also published the International vocabulary of metrology (ISO/IEC Guide 99, 2012) that has to be referred to by test laboratory.

In the regulatory sector, government authorities implement laws covering the approval of drinking water systems mainly for reasons of public health and thus required conformity of drinking water to specified requirements such as GL. Conformity assessment bodies can objectively state such conformity and perform conformity assessment activities including certification, inspection and testing in line with ISO 17011 (2004) that gives a functional approach to conformity assessment and specifies the general requirements for accreditation bodies. With all accredited testing laboratories on an equal footing, recognized as such by all States, trade of any product, accepted formally in one economy, is then free to circulate in other economies without having to undergo extensive re-testing, re-inspection, re-certification, etc.

At the international level, in response to a growing need for an open, transparent and comprehensive scheme to give users reliable quantitative information on the comparability of national metrology services and to provide the technical basis for wider agreements negotiated for international trade, the Mutual Recognition Arrangement (MRA) was signed in October 1999 by leading National Metrology Institutes (NMI) of 38 Member States of the Mètre Convention and by two International Organizations under the auspices of the Comité International des Poids et Mesures (CIPM), and coordinated by the BIPM. The MRA objective is to establish the degree of equivalence of the national measurement standards maintained by the NMIs, to provide for the mutual recognition of the calibration and measurement certificates issued by the NMIs and provide thus to the Governments and other parties a solid technical foundation for other more extended agreements in connection with international trade, and regulation activities. The CIPM

MRA has now been signed by the representatives of 89 institutes from 51 Member States, 35 Associates of the CIPM, and 3 international organizations (IAEA in Vienna, the World Meteorological Organization in Geneva and the Institute for Reference Materials and Measurements in Geel) and covers a further 143 institutes designated by the signatory bodies (see <http://www.bipm.org/en/cipm-mra>).

Standards on Nuclear Metrology

Concerning standards for testing laboratories most specifically in charge of radioactivity measurement, ISO 80000-10 (2010) should be mentioned that gives the names, symbols, and definitions for quantities and units used in atomic and nuclear physics and IEC 60050 part 393 (2003), that supplements the VIM for the terminology used in nuclear instrumentation. The various parts of ISO 11843 can be used by testing laboratories to assess the capability of detection of their radionuclide measurement methods applying the generic approach specified in ISO Guide 30 (1995) and in ISO 11095 (1996).

Concerning radioactivity measurement, it is Sub-Committee 2 on Radiological protection of the Technical Committee 85, Nuclear Energy, Nuclear Technologies, and Radiological Protection that is in charge of producing standards in the field of the protection of individuals and the environment, with the exception of water, against all sources of ionizing radiations. Its terms of reference include the metrology of radiation and therefore various key standards for testing laboratories have been published. Thus, following the ISO/IEC Guide 98 requirement, ISO TC85/SC2 drafted ISO 11929 (2010) that specifies the procedure, in the field of ionizing radiation metrology, for the calculation of the decision threshold, the detection limit and the limits of the confidence interval that must be applied to establish the characteristic limits of any test method.

Standards on water radioactivity monitoring

Concerning water, it is the TC147 that is in charge specifically of standardization in the field of Water Quality, including definition of terms, sampling of waters, measurement and reporting of water characteristics but excluding the limits of acceptability for water quality. This Committee produced ISO 5667 that in its Part 1 sets out the general principles for, and provides guidance on, the design of sampling programmes and sampling techniques for all aspects of the sampling of water. Detailed instructions for specific sampling situations are covered in the various other parts of ISO 5667. Concerning water sampling, Part 3 of ISO 5667 (2004) presents guidance on the preservation and handling of water samples and part 20 (2008) provides guidance on the use of sampling data for decision making - compliance with thresholds and classification systems.

Since 1978, Sub-Committee 3 Radioactivity measurement of ISO/TC 147 has developed standards on test methods used for the monitoring of waters for regulatory purposes, research, etc. The SC3 terms of reference cover laboratory test methods, in situ and on-line measurements, of any individual radionuclide of natural or artificial origin, as well as radionuclide nonspecific parameters such as gross alpha activity and gross beta activity. The first edition of the WHO guidelines for drinking-water quality was published in 1984 and SC 3 published its first standard on the determination of tritium activity concentration using a liquid scintillation counting method in 1989 (ISO 9698, 2010). Three others standards were subsequently published, on the measurement of gross alpha activity (ISO 9696, 2007) and gross beta activity (ISO 9697, 2008) in 1992, and on gamma-ray spectrometry (ISO 10703, 2007) in 1997. The latter is related to IEC 1452 (1995) that establishes methods for the calibration and use of germanium spectrometers for the measurement of gamma-ray energies and emission rates and the calculation of source activities from these measurements.

These standard test methods form the basis for the monitoring of drinking water in line with the approach recommended by WHO since the first edition of its guidelines. The ensuing periodic revision of these standards demonstrated the need for reviews due to the availability of new equipment and the request to decrease the detection limit of many radionuclides in drinking water. The revised versions of ISO 9696, 9697, 10703 were subsequently published in 2008 and in 2011 for ISO 9696. Two alternative test methods to determine the gross alpha and gross beta activities were also published with a new sample preparation stage by direct evaporation to dryness described in ISO 10704 (2009) and with a different type of measuring equipment by liquid scintillation counting detailed in ISO 11704 (2010). In 2008, it was agreed to draft a new set of standards on test methods on ^{90}Sr (ISO 13160, 2012), ^{210}Po by alpha-particle spectrometry (ISO 13161, 2011) and ^{14}C by liquid scintillation (ISO 13162, 2012) that were subsequently published this year.

As naturally occurring radionuclides belonging to the U and Th series present in drinking water usually give radiation doses higher than those provided by man-made radionuclides, and are therefore of greater concern, in 2009 a new set of supplementary standards was proposed on test methods for ^{226}Ra , ^{210}Pb both using liquid scintillation counting, ^{222}Rn using gamma-ray spectrometry and an emanometric method (Calmet *et al.*, 2011), and for uranium radioisotopes. These are currently being drafted and at stages close to publication.

Discussion and Conclusion

Hundreds of thousands of radioactivity measurements are performed yearly by testing laboratories on environmental samples and reported to national authorities for regulatory purposes and public information. Increasing regulatory pressure on the monitoring of drinking water has already dramatically increased analytical needs of testing laboratories in terms of improved detection limit and quality program as national authorities seeks to lower

any expected risk to public health. This trend will certainly be accentuated in the future as countries, as already done the USA, could set a maximum contaminant level goal¹ for radionuclides in drinking water to zero, giving the authority a large margin of safety (EPA, 1993).

Thus the use of international standards on test methods for radionuclides in water samples is justified for laboratories carrying out these tests as they must obtain specific accreditation for these measurements on drinking water samples. In this context, the ISO strategic plan on this area aims to focus on priorities resulting from the latest WHO recommendations, taking into account the technical consequences of regulatory changes in drinking-water quality control.

The set of ISO standards on measurement and radionuclide test methods address the needs of test laboratories as they adhere to WHO and ICRP recommendations on assessing drinking-water safety with respect to naturally occurring and artificial radionuclides. The above-mentioned ISO standards rely on the trust built up between the two international organizations and experts representing monitoring laboratories in 23 countries. In the latest (2011) edition of the WHO guidelines, they are listed as references for the measurement of radionuclides in water in chapter 9 on radiological aspects. These standards can also be used to select the appropriate test methods needed that will provide 'fit for purpose' results for identifying spatial and/or temporal trends in the radiological characteristics of the water source, required to ensure adequate water management of the water quality for other uses such as crop irrigation and freshwater fish farming. EC Regulation No 178/2002 (2002) laying down procedures in matters of food safety already mentioned that where international standards exist or their completion is imminent, they shall be taken into

¹ *The maximum contaminant level goal (MCLG) is defined by the EPA as the level of a contaminant in drinking water below which there is no known or expected risk to health. MCLGs allow for a margin of safety and are non-enforceable public health goals.*

consideration in the development or adaptation of food law.

The Fukushima accident dramatically recalled the need to rapidly check the radionuclide activity concentration in drinking water to ensure the radiological protection of the public. In this situation, the major constraint for a testing laboratory during an accidental situation is less the accuracy of the result than the rapidity of the data availability, as water is needed for many uses. Following the accident, the Japanese Ministry of Health, Labour, and Welfare (2011) set provisional index values for radioactive iodine activity concentration in drinking water to 300 Bq/kg for adult and 100 Bq/kg for infants and of 200 Bq/kg for the sum of the activity concentration of ^{134}Cs and ^{137}Cs . These values are orders of magnitude higher than the level monitored in normal situations. ISOTC147 members has started to review the existing test methods and standards that could be applied in nuclear emergency situations and that could be implemented in emergency preparedness plans of radioactivity testing laboratories.

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Table Caption

Table 1: Reference activity concentrations and limit of detection for radioactivity in drinking water.

Table 1: Reference activity concentrations and limit of detection for radioactivity in drinking water.

Radionuclide	WHO	EC		USA	
	Guidance Level (Bq/L) ¹	Reference conc. ² (Bq/L)	Detection limit ³ (Bq/L)	Max Cont.Level ⁴ (Bq/L)	Detection limit ⁵ (Bq/L)
Gross alpha	0.5	0.1	0.04	0.555 ⁶	0.111
Gross beta	1	1	0.4 ⁷	1.85 ⁸	0.148
³ H(HTO)	10 000	100	10	740	37
Natural					
²²⁶ Ra	1	0.5	0.04	0.185	0.037
²²⁸ Ra	0.1	0.2	0.08	0.185	0.037
¹⁴ C	100	240	20	111.0	11.1
Artificial					
⁹⁰ Sr	10	4.9	0.4	0.296	0.074
⁶⁰ Co	100	40.0	0.5	7.4	0.7
¹³⁴ Cs	10	7.2	0.5	2.96	0.37
¹³⁷ Cs	10	11.0	0.5	7.4	0.7
¹³¹ I	10	6.2	0.5	0.111	0.037

¹WHO, 2011, Annex 6.

²EC, 1998.

³EC, 2012.

⁴USA, CFR 40§ 141.66(d)Maximum contaminant levels for radionuclides;

⁵ USA, 40 CFR 141.25 - Analytical methods for radioactivity: Detection Limits for the Various Radiochemical Contaminants table IV-2A; Other Radionuclides and Photon/Gamma Emitters 1/10th of the applicable MCL;

⁶ including radium-226 but excluding radon and uranium;

⁷ Excluding the contribution of tritium, potassium-40, radon and radon decay products;

⁸ calculations based on 40 CFR/141.16. Derived Concentrations of Beta and photon emitters in drinking water yielding a dose of 4 mRem/y to the total body or to any critical organ as defined in NBS Handbook 69.

Accepted manuscript