

# Implementation of ISO/IEC 17025 in a low level liquid scintillation tritium laboratory

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*Ll Pujot, M.E. Pérez-Zabaketa  
CEDEX – Alfonso XII, 3, 28014 Madrid, Spain*

**CEDEX**

Centro de Estudios y Experimentación de Obras Públicas

# Summary

- Introduction to ISO/IEC 17025.
- Quality system of the CEDEX.
- Method for tritium enrichment.
- Validation of the tritium method.
- How to apply for accreditation.
- Internal quality control.
- Conclusions.

# Introduction to ISO/IEC 17025



## What is ISO/IEC 17025?

The practical experience on the implementation of **ISO/IEC 17025** quality system in a low level tritium laboratory of **CEDEX** (Madrid, Spain) is described.

The ISO/IEC 17025 is the main standard used by testing and calibration laboratories.

Laboratories use this standard to implement a quality system aimed at improving their ability to produce **valid results**.

It is also the basis for accreditation from an Accreditation Body.

This standard comprises two aspects:

- **Management Requirements**
- **Technical Requirements.**

# Management Requirements of ISO/IEC 17025



- 4.1. Organization.
- 4.2. Quality system.
- 4.3. Document control.
- 4.4. Review of requests, tenders and controls.
- 4.5. Subcontracting of tests and calibrations.
- 4.6. Purchasing services and supplies.
- 4.7. Service to the client.
- 4.8. Complaints.
- 4.9. Control of non-conforming testing and/or calibration work.
- 4.10. Corrective actions.
- 4.11. Preventive actions.
- 4.12. Control of records.
- 4.13. Internal audits.
- 4.14. Management reviews.

# Technical Requirements of ISO/IEC 17025



- 5.1. General.
- 5.2. Personnel.
- 5.3. Accommodation and environmental conditions.
- 5.4. Test and calibration methods and method validation.
- 5.5. Equipment.
- 5.6. Measurement traceability.
- 5.7. Sampling.
- 5.8. Handling of test and calibration items.
- 5.9. Assuring the quality of test and calibration results.
- 5.10. Reporting the results.



# Quality system of the CEDEX

**CEDEX**

Centro de Estudios y Experimentación de Obras Públicas

# What is CEDEX?

The Centro de Estudios y Experimentación de Obras Públicas (CEDEX) was created in 1957. At present, it is ascribed to the Ministry of Public Works of Spain.

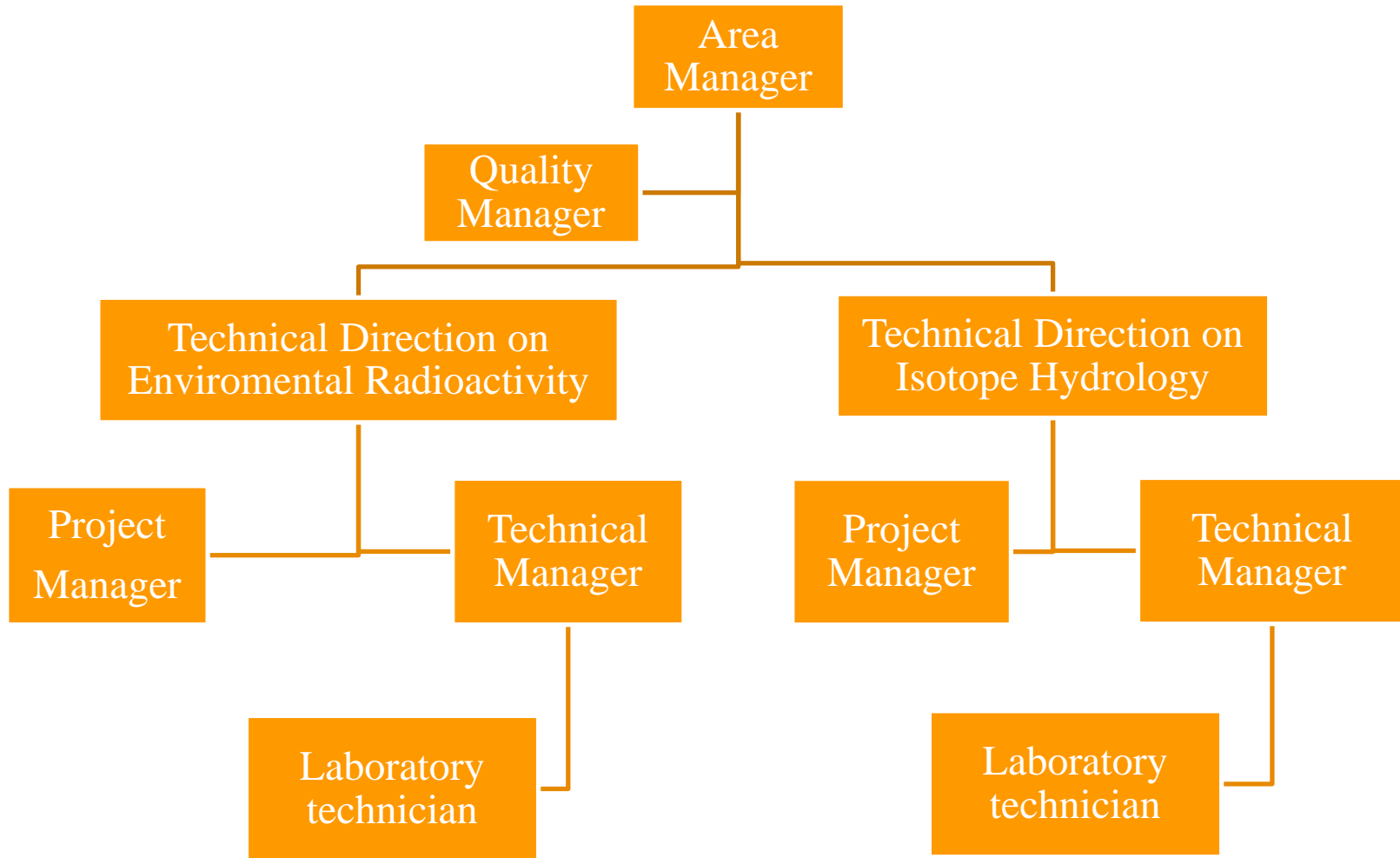


CEDEX is an institution that provides multidisciplinary support in civil engineering technologies, construction and associated environment, giving assistance to various administrations, public institutions and private companies.

**CEDEX**

Centro de Estudios y Experimentación de Obras Públicas

# Laboratory Organization Chart of Isotope Applications Area of CEDEX





# Main stages of the accreditation process in the CEDEX Laboratory

## ✓ **Quality system implementation.**

- Quality policy and quality manual.
- **17 Quality procedures (management and technical requirements).**
- Technical Instructions.

# Main stages of the accreditation process in the CEDEX Laboratory

## ✓ 17 Quality procedures (QP).

- QP-1: Independence, impartiality, integrity, confidentiality and security of information.
- QP-2: Document control.
- QP-3: Review of requests, tenders and contracts. Service to the customer and complaints
- QP-4: Purchasing services and supplies.
- QP-5: Control of nonconforming, corrective and preventive actions.
- QP-6: Control of records.
- QP-7: Internal audits and management reviews.
- QP-8: Recruitment, training and qualification for technical activities.
- QP-9: Accommodation and environmental conditions.
- QP-10: Validation of methods.
- QP-11: Estimation of uncertainty of measurement.
- QP-12: Assuring the quality of test.
- QP-13: Equipment and measurement traceability.
- QP-14: Handling of Environmental Radioactivity tests.
- QP-15: Reporting the results.
- QP-16: Notification of changes to ENAC.
- QP-17: Handling of Isotope Hydrology tests.

# Main stages of the accreditation process in the CEDEX Laboratory

## ✓ **Quality system implementation.**

- Quality policy and quality manual.
- 17 Quality procedures (management and technical requirements).
- **Technical Instructions.**

# Main stages of the accreditation process in the CEDEX Laboratory

Code	Title	Rev.	Date
<b>ITE-1006</b>	<b>Tritium activity by electrolytic enrichment and liquid scintillation counting</b>	<b>3</b>	<b>May 2015</b>
F-ITE-100601	Template for sample preparation control by electrolytic enrichment	3	May 2015
ITV-1301	Verification of liquid scintillation Quantulus 1220	3	May 2015
ITV-1302	Tritium-free background management for low-level counting	1	June 2015
ITV-1303	Reference material preparation for tritium determination by liquid scintillation counting	1	June 2015

# Main stages of the accreditation process in the CEDEX Laboratory

## ✓ **Quality system implementation.**

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- Technical Instructions.

## ✓ **Method validation.**

- Validation parameters.
- Statement of fitness-for-purpose.

# Main stages of the accreditation process in the CEDEX Laboratory

## ✓ **Quality system implementation.**

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## ✓ **Method validation.**

- Validation parameters.
- Statement of fitness-for-purpose.

## ✓ **Formal accreditation.**

- Apply for the accreditation.
- Initial audit.

# Main stages of the accreditation process in the CEDEX Laboratory

## ✓ **Quality system implementation.**

- Quality policy and quality manual.
- 17 Quality procedures (management and technical requirements).
- Technical Instructions.

## ✓ **Method validation.**

- Validation parameters.
- Statement of fitness-for-purpose.

## ✓ **Formal accreditation.**

- Apply for the accreditation.
- Initial audit.

## ✓ **Maintenance of accreditation.**

- Internal quality control (& others requirements)



# Method for tritium enrichment



# Procedure for tritium electrolytic enrichment in the CEDEX Laboratory



**Initial  
Distillation**

A batch of 9 water samples of 500 ml are distilled once. Also, one control sample ("spiked" water) is prepared with 490 ml of tritium-free water and 10 ml of a tritium standard.

# Procedure for $^3\text{H}$ electrolytic enrichment in the CEDEX Laboratory



**Initial  
Distillation**



**$\text{Na}_2\text{O}_2$   
addition**

Samples and spike are poured into the cells and 2 g of  $\text{Na}_2\text{O}_2$  are added.

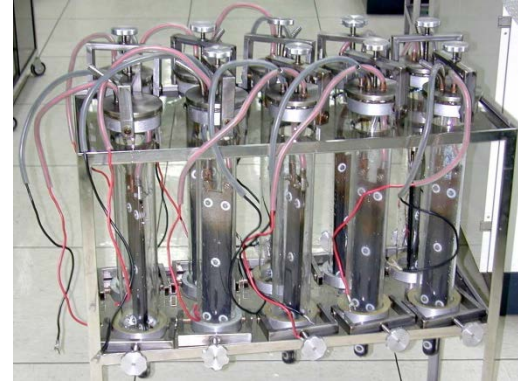
# Procedure for $^3\text{H}$ electrolytic enrichment in the CEDEX Laboratory



**Initial  
Distillation**



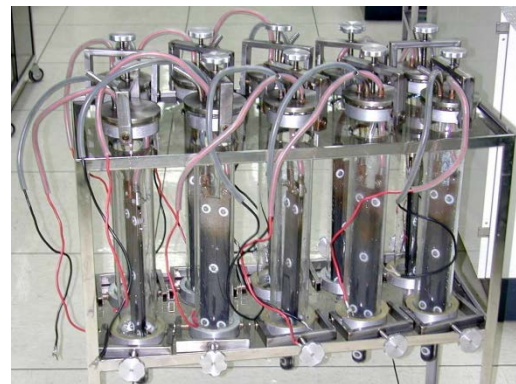
**$\text{Na}_2\text{O}_2$   
addition**



**9 samples  
& 1 spike  
of 500 ml**

The equipment used for the electrolytic enrichment consists of 10 electrolytic cells.

# Procedure for $^3\text{H}$ electrolytic enrichment in the CEDEX Laboratory



9 samples & 1 spike of 500 ml

**Initial Distillation**

**Na<sub>2</sub>O<sub>2</sub> addition**



Cells are placed in a refrigerating container that vary between 0.5 and 2 °C.  
**Process last 8 days.**



**Refrigerating container for electrolytic enrichment**

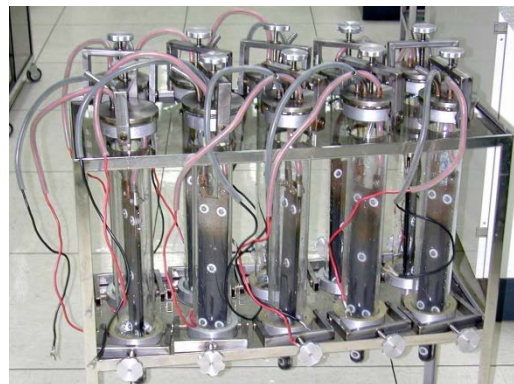
# Procedure for <sup>3</sup>H electrolytic enrichment in the CEDEX Laboratory



**Initial Distillation**



**Na<sub>2</sub>O<sub>2</sub> addition**



**9 samples & 1 spike of 500 ml**



**Refrigerating container for electrolytic enrichment**



**Final Distillation**

At the end of each electrolysis run, the enriched water is neutralized by adding PbCl<sub>2</sub> before the final distillation.

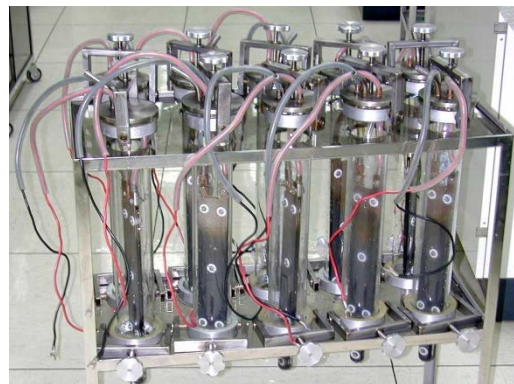
# Procedure for $^3\text{H}$ electrolytic enrichment in the CEDEX Laboratory



**Initial Distillation**



**Na<sub>2</sub>O<sub>2</sub> addition**



**9 samples & 1 spike of 500 ml**



**Refrigerating container for electrolytic enrichment**



**Final Distillation**



**Liquid scintillation counting**

# Expression used for tritium concentration determination at CEDEX laboratory



$$A_T = \frac{N_{SA} \cdot A_{ST}}{N_{SP} \cdot V \cdot D}$$

It must be remarked that in our methodology, we assume that the behaviour of all electrolytic cells is the same to that of the spike.

$A_T$  is the tritium activity concentration,

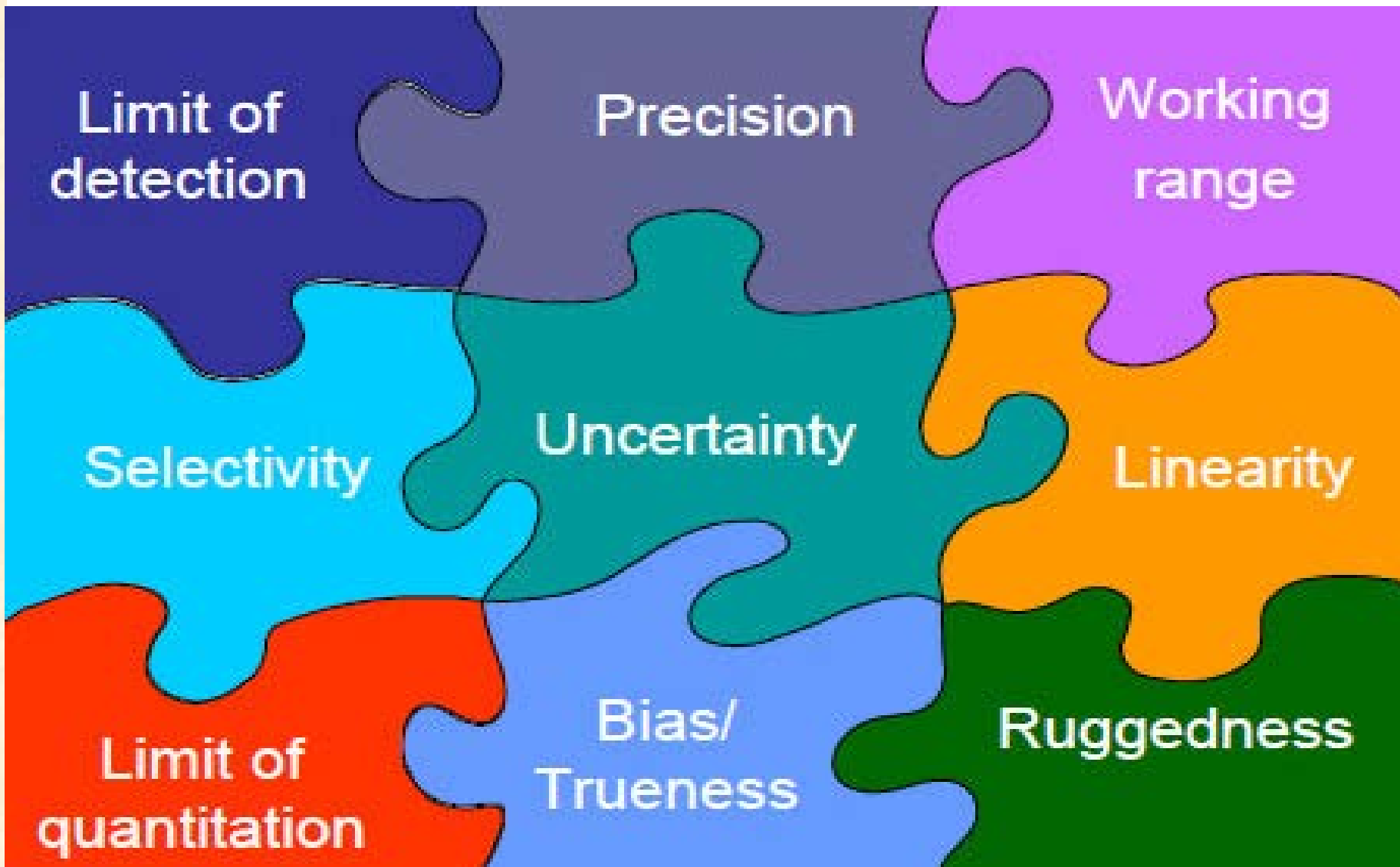
$N_{SA}$  is the net count rate of the sample (cpm),

$A_{ST}$  is the activity concentration of the standard (Bq),

$N_{SP}$  is the net count rate of the spike (cpm),

$V$  is the volume of the sample (l),

$D$  is the factor taking into account decay of tritium in the sample from the date of measurement to the date of sampling.



# *Validation of the tritium method*



# Validation parameters for electrolytic enrichment

Although several validation parameters are applicable for all types of test, **the validation parameters considered were:**

- ✓ **Precision.** Closeness of agreement between indications or measured quantity values obtained by replicate measurements on the same or similar objects under specified conditions (VIM, 2012).
- ✓ **Accuracy.** Closeness of agreement between a measured quantity value and a true quantity value of a measurand (VIM, 2012).
- ✓ **Detection limit.** Measured quantity value, obtained by a given measurement procedure, for which the probability of falsely claiming the absence of a component in a material is  $\beta$ , given a probability  $\alpha$  of falsely claiming its presence (VIM, 2012).
- ✓ **Uncertainty.** Non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used (VIM, 2012).

# Validation parameters equations

Parameter	Equation	Definitions
Precision	$RSD(\%) = 100 \cdot \frac{\sigma}{\mu}$	<p><math>\sigma</math> is the standard deviation</p> <p><math>\mu</math> is the mean</p> <p><i>RSD</i> is the rel. standard deviation</p>
Accuracy	$RB(\%) = 100 \cdot \frac{ \mu - X_{ref} }{X_{ref}}$	<p><math>\mu</math> is the measured value</p> <p><math>X_{ref}</math> is the reference value</p> <p><i>RB</i> is the relative bias</p>
Detection limit	$MDA = \frac{3.29 \cdot A_{ST}}{D \cdot V \cdot N_{SP}} \cdot \sqrt{\frac{N_b}{t_m} + \frac{N_b}{t_{mb}}}$	<p><math>A_{ST}</math> is the activity of standard (Bq)</p> <p><math>D</math> is the decay correction factor</p> <p><math>V</math> is the volume of the sample (l)</p> <p><math>N_{SP}</math> is the net count rate of spike (cpm)</p> <p><math>N_b</math> is the background count rate (cpm)</p> <p><math>t_m</math> is the sample counting time (min)</p> <p><math>t_{mb}</math> is the backgr. counting time (min)</p>

# Validation parameters equations

Parameter	Equation and definitions
Uncertainty	$u(A_T) = A_T \cdot \sqrt{\left(\frac{u(N_{SA})}{N_{SA}}\right)^2 + \left(\frac{u(A_{ST})}{A_{ST}}\right)^2 + \left(\frac{u(N_{SP})}{N_{SP}}\right)^2 + \left(\frac{u(V)}{V}\right)^2 + \left(\frac{u(D)}{D}\right)^2}$ <p> <math>u(A_T)</math> is the uncertainty of the tritium activity concentration.  <math>u(N_{SA})/N_{SA}</math> is the relative standard uncertainty (RSU) of the <b>net count rate of the sample</b> (cpm).  <math>u(A_{ST})/A_{ST}</math> is the RSU of the <b>activity concentration of the standard</b> (Bq).  <math>u(N_{SP})/N_{SP}</math> is the RSU of the <b>net count rate of the spike</b> (cpm).  <math>u(V)/V</math> is the RSU of the <b>volume of the sample</b> (l).  <math>u(D)/D</math> is the RSU of the <b>decay correction factor</b>.         </p>

The combined uncertainty is calculated from the relative standard uncertainties of the components of the activity expression.

## Method validation: precision (drinking & inland water)

Precision was carried out with **tritium-free background water spiked** with 0.1 ml (level 1) and with 1 ml (level 2) of tritium standard dilution.

Level 1 (MDA - 10 T.U.) run: B2378		Level 2 (10 T.U. – 100 T.U.) run: B2379		Level 2 (10 T.U. – 100 T.U.) run: B2380	
Sample Number	Activity (T.U.)	Sample Number	Activity (T.U.)	Sample Number	Activity (T.U.)
15103	9.93 ± 0.51	15112	86.3 ± 2.6	15121	84.7 ± 2.5
15104	7.92 ± 0.45	15113	87.6 ± 2.6	15122	84.6 ± 2.5
15105	7.87 ± 0.45	15114	86.6 ± 2.6	15123	86.8 ± 2.5
15106	7.93 ± 0.45	15115	90.1 ± 2.6	15124	86.2 ± 2.5
15107	7.40 ± 0.43	15116	93.1 ± 2.7	15125	81.4 ± 2.4
15108	8.26 ± 0.46	15117	86.1 ± 2.6	15126	82.6 ± 2.4
15109	8.13 ± 0.45	15118	91.7 ± 2.7	15127	80.5 ± 2.4
15110	8.02 ± 0.45	15119	90.4 ± 2.7	15128	87.5 ± 2.6
15111	9.42 ± 0.49	15120	93.6 ± 2.7	15129	84.5 ± 2.5
<b>RSD (%)</b>	<b>9.8</b>	<b>RSD (%)</b>	<b>3.3</b>	<b>RSD (%)</b>	<b>2.9</b>

## Method validation: precision (seawater)

Precision in seawater was carried out with **seawater spiked** with 0.1 ml (level 1) and with 1 ml (level 2) of tritium standard dilution.

Level	Run	Sample Number	Activity (Bq/l)	Activity (Bq/l)	RSD (%)
MDA - 1 Bq/l (level 1)	B2381 (TT730)	30677	1.02 ± 0.06	1.06 ± 0.06	2.72
		30678	1.06 ± 0.06	1.14 ± 0.06	5.14
		30679	1.07 ± 0.06	1.00 ± 0.06	1.04
1 Bq/l – 10 Bq/l (level 2)	B2375 (TT711)	30552	11.1 ± 0.3	11.2 ± 0.3	0.95
		30553	10.1 ± 0.3	10.4 ± 0.3	2.14
		30554	10.6 ± 0.3	10.2 ± 0.3	3.06
1 Bq/l – 10 Bq/l (level 2)	B2376 (TT713)	30549	10.7 ± 0.3	10.6 ± 0.3	0.93
		30550	11.3 ± 0.3	11.8 ± 0.3	2.69
		30551	10.7 ± 0.3	11.1 ± 0.3	2.59

## Accuracy (drinking & inland and water seawater)

Accuracy was carried out with the same previously tritium-free background water spiked with tritium standard dilution used for precision.

Level 1 (MDA - 10 T.U.) run: B2378		Level 2 (10 T.U. – 100 T.U.) run: B2379		Level 2 (10 T.U. – 100 T.U.) run: B2380	
Sample Number	Activity (T.U.)	Sample Number	Activity (T.U.)	Sample Number	Activity (T.U.)
15103	9.93 ± 0.51	15112	86.3 ± 2.6	15121	84.7 ± 2.5
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15107	7.40 ± 0.43	15116	93.1 ± 2.7	15125	81.4 ± 2.4
15108	8.26 ± 0.46	15117	86.1 ± 2.6	15126	82.6 ± 2.4
15109	8.13 ± 0.45	15118	91.7 ± 2.7	15127	80.5 ± 2.4
15110	8.02 ± 0.45	15119	90.4 ± 2.7	15128	87.5 ± 2.6
15111	9.42 ± 0.49	15120	93.6 ± 2.7	15129	84.5 ± 2.5
<b>Mean</b>	<b>8.32</b>	<b>Mean</b>	<b>89.5</b>	<b>Mean</b>	<b>84.3</b>
$X_{ref}$	<b>8.29</b>	$X_{ref}$	<b>83.0</b>	$X_{ref}$	<b>83.0</b>
<b>RB (%)</b>	<b>0.36</b>	<b>RB (%)</b>	<b>7.9</b>	<b>RB (%)</b>	<b>1.6</b>

# Uncertainty estimation



Uncertainty sources were evaluated before to the method validation and presented in International Symposium on Quality Assurance for Analytical Methods in Isotope Hydrology in Vienna in August 2004, in the paper "Assessment of the low-level liquid scintillation tritium laboratory at CEDEX, Madrid" (IAEA-CN-119/11).

The uncertainty of the individual parameters was evaluated according to recommendations of the IAEA and EURACHEM. Results showed that for samples with low tritium concentration, the uncertainty of the net count rate of the sample was the dominating component.

The expanded uncertainty for  $k=2$  and for 1 TU was  $\pm 0.43$  TU.  
The expanded uncertainty for  $k=2$  and for 10 TU was  $\pm 0.7$  TU.

# Statement of fitness-for-purpose for method validation

 Centro de Estudios y Experimentación de Obras Públicas	DECLARACIÓN DE MÉTODO VALIDADO	Cod: F-PC-1001
		Rev.: 02
		Fecha: Mayo 2012

MÉTODO DE ENSAYO: (se adjunta)	ITE-1006.-Determinación de la actividad de tritio por concentración electrolítica en muestras de agua
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Parámetro a determinar: Concentración de la actividad de tritio  
 Unidades: Bq/l y UT

	Precisión	Exactitud	AMD	U Expandida
Matriz	CV (%)	Error relativo (%)	Bq/l y UT	U (k= 2)
Aguas de mar	Nivel 1	15%	0,04 Bq/l	0,43 - 2,74
	Nivel 2	10%		
Aguas continentales y de consumo	Nivel1	15%	0,31 -0,35 UT	0,43 - 2,74
	Nivel 2	10%		

CRITERIOS DE ACEPTACIÓN / RECHAZO (para aplicación del Aseguramiento de la calidad):

Se diferencian por matrices y se aceptarán los valores de CV(%) con valores inferiores a los indicados en la tabla siguiente:

Matriz	Actividad (Bq/l)	CV (%)
Aguas de mar	AMD - 1 Bq/l	15%
Aguas continentales y de consumo	1 Bq/l - 10 Bq/l	10%

En el caso de la exactitud, se aceptarán para las dos matrices valores de ER (%) inferiores al 15% para el nivel 1 y del 10% para el nivel 2.

El presente método queda validado para su uso en el AAI por las personas cualificadas para ello de acuerdo con las características que se detallan en esta hoja.

Director Técnico  
 Fdo.:  Fecha: 27-8-2014

SE ADJUNTA EL INFORME DE VALIDACIÓN IDENTIFICADO EN EL ENCABEZAMIENTO

5.4.5.2 (ISO/IEC 17025).

The laboratory shall record the results obtained, the procedure used for the validation, and a **statement** as to whether the method is fit for the intended use.







# *How to apply for accreditation*

## *How we applied for accreditation*

- ✓ We completed an application form, which contained a series of legal commitments, along with a number of annexes detailing the information of the laboratory.
- ✓ We defined the scope of the accreditation.
- ✓ The Spanish Accreditation Body (ENAC) revised all this documentation.
- ✓ If everything was correct, the laboratory received an estimated quotation of the cost of the process.

## Scope of Accreditation to ISO/IEC 17025:2005 of CEDEX

Matrix	Test	Standard/ Instruction
Drinking waters and inland waters	Gross alpha activity by ZnS(Ag) scintillation detector ( $> 0.03$ Bq/l)	ITE-1005
	Gross beta activity by proportional counter ( $> 0.04$ Bq/l)	UNE 73311-4: 2002
	Residual beta activity by proportional counter ( $> 0.04$ Bq/l)	UNE 73340-2: 2003
	Tritium activity by LSC ( $> 3$ Bq/l)	ITE-1003
	<b>Tritium activity by electrolytic enrichment and liquid scintillation counting (<math>&gt; 0.05</math> Bq/l)</b>	<b>ITE-1006</b>
Sea waters	Gross beta activity by proportional counter ( $> 0.70$ Bq/l)	UNE 73311-4: 2002
	Residual beta activity by proportional counter ( $> 0.70$ Bq/l)	UNE 73340-2: 2003
	<b>Tritium activity by electrolytic enrichment and liquid scintillation counting (<math>&gt; 0.05</math> Bq/l)</b>	<b>ITE-1006</b>

## Initial Audit

- ✓ Initial audit is conducted by an audit team made up of a **Chief Auditor** and a **senior Technical Expert**. The duration of the visit will depend on the requested scope.
- ✓ At the end of the audit, a **report** is submitted with a summary of the assessment and the deviations detected, if any.
- ✓ If deviations were detected, you will need to respond with a **plan of action** to address the problems identified.
- ✓ Accreditation body assess whether the corrective action plan has resolved the issues identified.
- ✓ Accreditation is granted by Accreditation Committee.

# Certificate of Accreditation of CEDEX

Acreditación



**ACREDITACIÓN**  
82/LE1955

a la entidad técnica / to the technical entity

**CENTRO DE ESTUDIOS Y EXPERIMENTACION  
DE OBRAS PÚBLICAS (CEDEX)**  
**Centro de Estudios de Técnicas Aplicadas  
(CETA)**

Según criterios recogidos en la Norma UNE-EN ISO/IEC 17025, para la realización de ENSAYOS de PROTECCIÓN RADIOLÓGICA definidos en el ANEXO TÉCNICO adjunto.

According to the criteria in UNE-EN ISO/IEC 17025 for the performance of Test of Radiological Protection as defined in the attached Technical Annex.

Fecha de entrada en vigor / Coming into effect: 04/05/2012



D. Antonio Muñoz Muñoz  
Presidente  
4 de mayo de 2012

La acreditación mantiene su vigencia hasta notificación en contra. Este documento no tiene validez sin su correspondiente anexo técnico, cuyo número coincide con el de la acreditación. La presente acreditación y su anexo técnico están sujetos a modificaciones, suspensiones temporales y retirada. Su vigencia puede confirmarse en [www.enac.es](http://www.enac.es).

The accreditation maintains its validity unless otherwise stated. The present accreditation is not valid without its corresponding technical annex, which number coincides with the accreditation. This accreditation and its technical annex could be reduced, temporarily suspended and withdrawn. The state of validity of it can be confirmed at [www.enac.es](http://www.enac.es).

ENAC es firmante del Acuerdo Europeo de Reconocimiento Mutuo firmado entre Organismos Nacionales de Acreditación ([www.european-accreditation.org](http://www.european-accreditation.org)).

ENAC is signatory of the European Recognition Agreement signed among National Accreditation Bodies ([www.european-accreditation.org](http://www.european-accreditation.org)).

Ref.: CLE/5233

Código Validación Electrónica: TJs0g0PvSw79m8HdV3

La vigencia de la acreditación y del presente certificado puede confirmarse en <http://www.enac.es/web/enac/validacion-electronica> o haciendo clic aquí

## First cycle (four years)

- ✓ Initial audit: March 2012.
  - Gross beta.
- ✓ First follow-up : June 2013.
  - Direct tritium.
  - Residual beta.
- ✓ Second follow-up: Dec. 2014.
  - Gross alpha.
  - **Electrolytic enrichment.**
- ✓ Re-assessed: May 2016.

## Second cycle (five years)

- ✓ Next December 2017.

**CEDEX**

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# *Internal quality control*

## Internal quality control carried out by CEDEX

1 Control chart for tritium background. Each run.

2 Control chart for tritium efficiency. Each run.

3

Characteristic	Periodicity	Matrix	Level	RSD (%)
Precision	Half yearly	Seawater	MDA - 1 Bq/l	< 15
			> 1 Bq/l - 10 Bq/l	< 10
		Drinking and inland water	MDA - 1 Bq/l	< 15
			> 1 Bq/l - 10 Bq/l	< 10

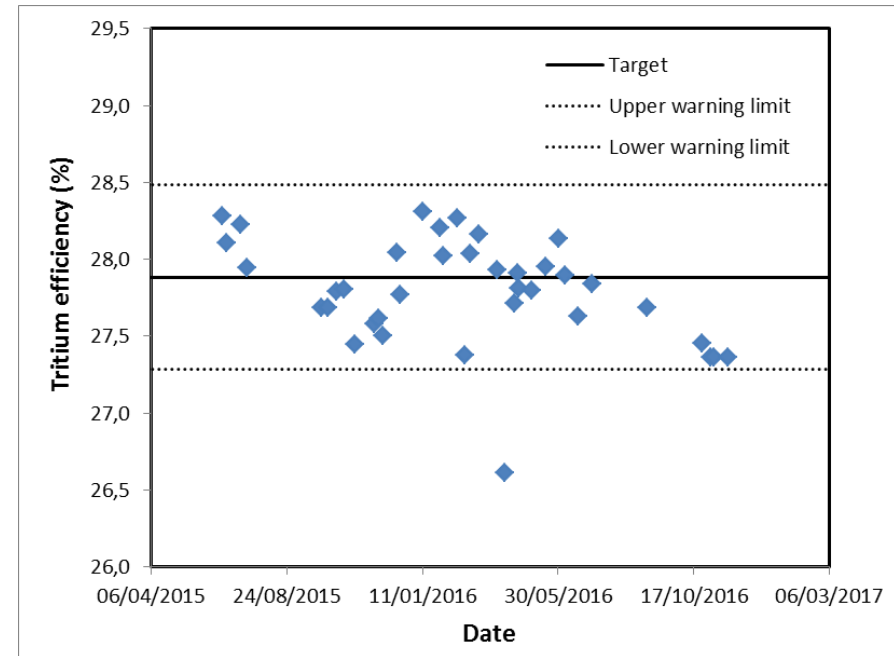
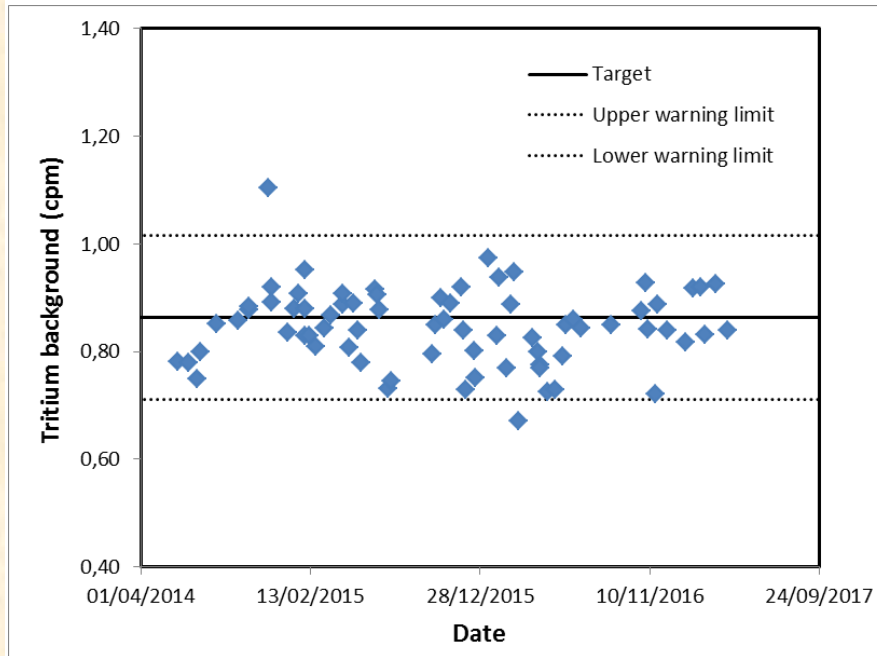
4

Characteristic	Periodicity	Matrix	Level	RB (%)
Accuracy	Half yearly	Seawater, drinking and inland water	MDA - 1 Bq/l	< 15
			> 1 Bq/l - 10 Bq/l	< 10

5 Control chart for enrichment factor (Z) and retention factor (RF). Each run.

1 &amp; 2

## Control charts for tritium background and for tritium efficiency



The assay performance is controlled by checking the long-term trends of the background and the efficiency. Typical background for Quantulus 1220 is below 1 cpm and typical efficiency around 28%.



3 &amp; 4

## Internal quality control: precision & accuracy (2<sup>nd</sup> half 2016)

### Internal Quality Control: Precision

Matrix	Level	Run	TT	Sample Number	Activity (Bq/l)			Activity (Bq/l)			RSD (%)
Seawater	MDA - 1 Bq/l	B2445	TT932	32579	1,04	±	0,07	0,94	±	0,07	10,3
	1 Bq/l – 10 Bq/l	B2445	TT932	32580	9,54	±	0,30	9,24	±	0,30	3,3
Drinking & inland water	MDA - 1 Bq/l	B2448	TT941	32581	0,33	±	0,05	0,34	±	0,05	2,9
	1 Bq/l – 10 Bq/l	B2448	TT941	32582	5,47	±	0,18	5,68	±	0,20	3,9

### Internal Quality Control: Accuracy

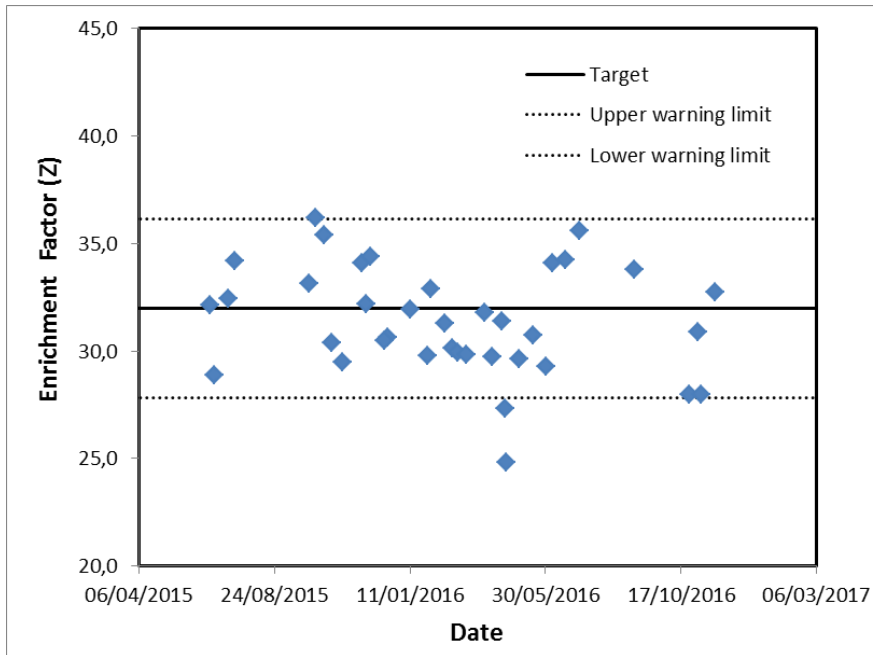
Matrix	Level	Run	TT	Sample Number	Activity (Bq/l)			Activity (Bq/l)			RB (%)
Drinking & inland water and Seawater	MDA - 1 Bq/l	B2448	TT941	32583	0,97	±	0,07	0,98	±	0,07	6,4
	1 Bq/l – 10 Bq/l	B2445	TT932	32584	3,08	±	0,14	3,28	±	0,13	6,8

Precision control value:  $RSD < 15\%$  (level 1) and  $RSD < 10\%$  (level 2).

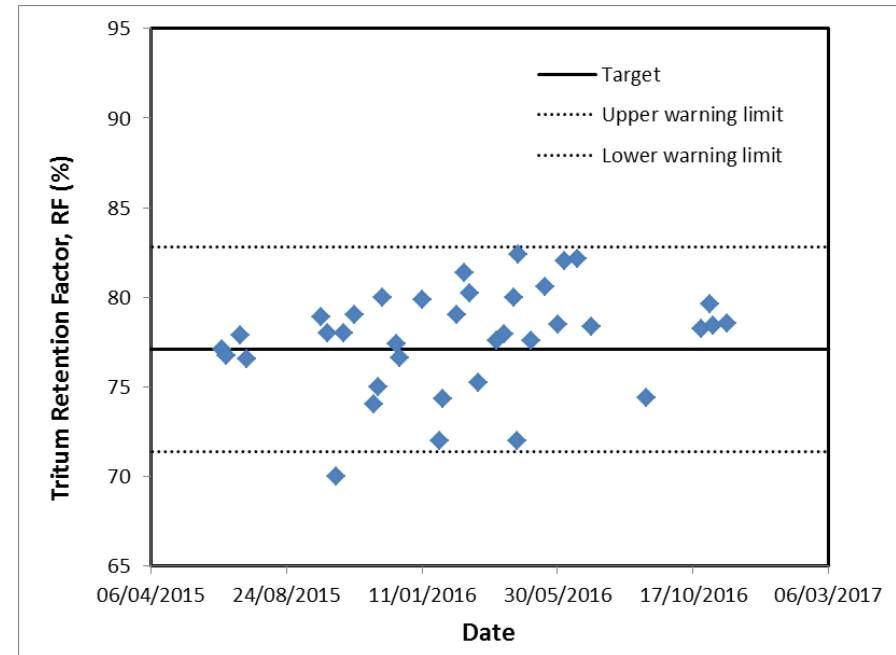
Accuracy control value:  $RB < 15\%$  (level 1) and  $RB < 10\%$  (level 2).

5

## Control charts for enrichment factor (Z) and for tritium retention factor (RF)



Control value for  $Z \geq 25$ .



Control value for  $RF \geq 70\%$ .

# Conclusions



## Comments

- This presentation shows **my personnel point of view** of the implementation of a quality system in the Isotope Applications Laboratory of CEDEX.
- **Not all the topics** related with the technical requirements such as equipment, personnel qualification for technical activities, interlaboratory comparison exercises and so on, have been presented in this work, **only internal quality control**.
- Several technical instructions for tritium electrolytic enrichment analysis were revised in order to adapt the technical aspects to the requirements of ISO/IEC 17025.
- Method validation seems to be the cornerstone of the technical requirements for a quality system according to ISO/IEC 17025.

## Recommendations

- Validation parameters considered for tritium analysis by electrolytic enrichment were: 1) precision, 2) accuracy, 3) detection limit, and 4) uncertainty estimation.
- Precision and accuracy parameters were determined into two different levels: 1) MDA – 1 Bq/l and 2) 1 Bq/l – 10 Bq/l.
- Internal quality control is an essential tool for assuring that analytical data produced from the laboratory are valid.
- Internal quality control carried out in our laboratory for tritium analysis by electrolytic enrichment was: 1) control charts for tritium background and tritium efficiency, 2) precision and accuracy determination half yearly into two different levels, and 3) control charts for tritium enrichment factor and tritium retention factor.

# Thank You For Your Attention...



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