IAEA interlaboratory studies in service of analytical quality of Member States' Laboratories

A. Shakhashiro, U. Sansone, A. Fajgelj

6th EURACHEM Workshop on Proficiency Testing
6-7 October 2008 Rome, Italy

IAEA
International Atomic Energy Agency

Content

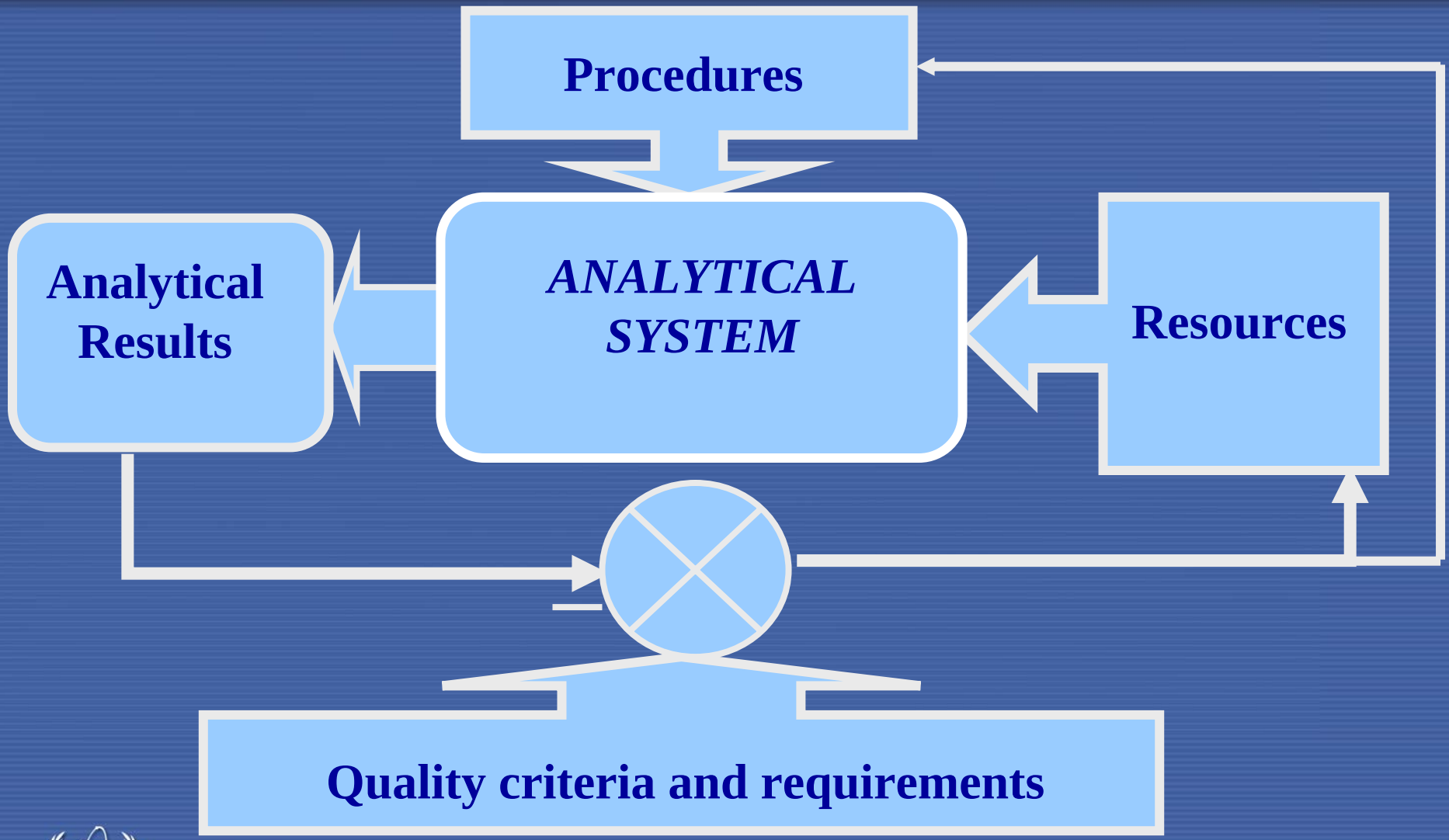
- Overall view of the IAEA laboratories;
- Recent developments related to the IAEA interlaboratory studies;
- Interlaboratory studies 2005-2008;
- Planned interlaboratory studies 2009-210;
- Know-how transfer and training to the Member States

The IAEA Laboratories Mission

Contribute to the implementation of the Agency's programmes in the field of:

- Food and agriculture
- Human health
- Physical and chemical sciences
- Environmental management
- Radiation protection
- Safeguards verification

Introduction



Introduction

http://curem.iaea.org/catalogue/

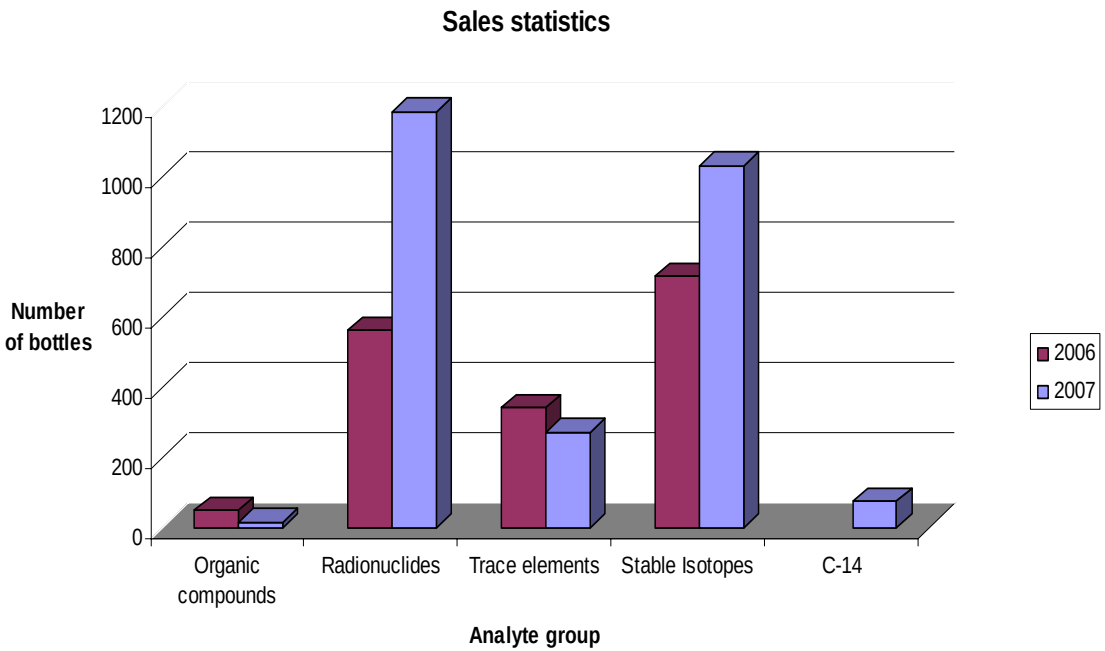
IAEA Reference Materials Catalogue and Documents

- Radionuclides
- Trace Elements & Methylmercury
- Organic Contaminants
- Stable Isotopes and Carbon-14
- Ordering Information
- Miscellaneous Documents

Revision 1.0
issued in Apr. 2007

Introduction

Preparation, certification and distribution of Reference Materials (RMs)



Organization

Department of Nuclear Sciences & Application

- Seibersdorf & Vienna Laboratories
- Physics, Chemistry & Instrumentation
  - Chemistry Unit → RMs of terrestrial origin
  - Isotope Hydrology Unit → RMs in the field of isotope hydrology
- FAO-IAEA Agriculture & Biotechnology
  - Soil Science Unit → N-15 & C-13 labelled plant PTMs
- Marine Laboratory Monaco → RMs of marine origin

Background

From the overall picture regarding the requests from the IAEA Member States (MS) it can be conclude that:

- The demand on PTs and RMs is increasing,
  - 670 laboratories from 94 different MS participated in CU PTs 2005-2007
- Customers' awareness is increasing,
- Higher quality criteria of PTs and RMs,
- Pressure of accreditation bodies to obtain services by accredited providers,

Recent developments

The Programme Performance Assessment Process in 1997 recommended to the Agency:

- Produce future reference materials in accordance with the ISO Guidelines 34 and 35.
- Implement a quality system at the Agency's Laboratories.
- Disseminate guidelines and standard operating procedures for analytical measurements

A step forward has been made

Recent developments

- In 2005 the IAEA NEtwork of Laboratories for Terrestrial Environment Reference Materials (NELTERM network) was formed

•production of matrix RM;

•Participation in characterization of matrix RM;

•Participation in the IAEA reference materials certification committee activities,

NELTERM 2nd meeting 3-5 September 2008

Recent developments

- in 2006 the IAEA RM Certification Committee was formed;
- in 2008 a Quality Manual was drafted according to ISO Guide 34 and ISO/IEC 17025;
- accreditation of reference material production according to combined ISO/IEC 17025 and ISO Guide 34 is planned for 2011;
- analytical procedures used for characterization of RM will be accredited.

Recent developments

- New reference materials storage facility

Recent developments

- Laboratory of Reference material preparation.

Recent developments

	Group of expert laboratories
Property value	Derived as consensus value
Type of Reference Material	Certified Reference Material
	Quality Control PT Material
Intended use	All Quality Assurance aspects

Recent developments

	Group of expert laboratories	1 expert lab & confirmation by 2 or more expert labs
Property value	Derived as consensus value	One value
Type of Reference Material	Certified Reference Material	Certified Reference Material
	Quality Control PT Material	Quality Control PT Material
Intended use	All Quality Assurance aspects	All Quality Assurance aspects

Recent developments

	Group of expert laboratories	1 expert lab & confirmation by 2 or more expert labs	Formulation
Property value	Derived as consensus value	One value	Value calculated from calibrant & confirmed from 1 or more labs.
Type of Reference Material	Certified Reference Material	Certified Reference Material	Certified Reference Material
	Quality Control PT Material	Quality Control PT Material	Quality Control PT Material
Intended use	All Quality Assurance aspects	All Quality Assurance aspects	All Quality Assurance aspects

Recent developments

	Group of expert laboratories	1 expert lab & confirmation by 2 or more expert labs	Formulation	Interlaboratory comparison study
Property value	Derived as consensus value	One value	Value calculated from calibrant & confirmed from 1 or more labs.	Derived as consensus value
Type of Reference Material	Certified Reference Material	Certified Reference Material	Certified Reference Material	Quality Control Material
	Quality Control PT Material	Quality Control PT Material	Quality Control PT Material	
Intended use	All Quality Assurance aspects	All Quality Assurance aspects	All Quality Assurance aspects	For precision & for control charts

Interlaboratory studies 2005-2008

ILs in support of Technical Cooperation Projects

- Total Arsenic in water for Bangladeshi laboratories — 2005
- RN and TE in soil and compost — 2006
- Major, minor and trace elements in Chinese ceramics — 2006
- ^{210}Pb and ^{137}Cs in soil for soil erosion studies research — 2006
- RN in sea water — 2007
- RN in air filters in cooperation with DOE — 2008

IAEA

Interlaboratory studies 2005-2008

ILs in support of Technical Cooperation Projects

Interlaboratory studies 2005-2008

World wide open PTs (WWOPT)

Gamma emitting radionuclides in water, soil and grass.

alpha, beta and gamma emitting radionuclides in water, soil and spinach.

Po-210 determination in water.

Naturally occurring radionuclides in phosphogypsum

2006 2007 2007 2008

Interlaboratory studies 2005-2008

ALMERA network PTs

Soil sampling exercise, Trieste, Italy.

Gamma emitting radionuclides in water, soil and grass.

Alpha, beta and gamma emitting radionuclides in water, soil and spinach.

Po-210 in water.

Naturally occurring radionuclides in phosphogypsum

2005 2006 2007 2007 2008

Interlaboratory studies 2005-2008

Interlaboratory comparisons

- CCRI(II) S4 -BIPM supplementary comparison on gamma emitting radionuclides, 2006
- CCRI(II) S5 -BIPM supplementary comparison on naturally occurring radionuclides, 2007
- CCQM-Pilot study P-104, trace elements in phosphogypsum, 20087.

PT on the determination of Cs-137 and Pb-210 in spiked soil

Interlaboratory studies 2005-2008

Cs-137 and Pb-210 for soil erosion studies

19 laboratories from

- Argentina, Australia, Brazil, Canada, China, Chile, Japan, Morocco, Pakistan, Poland, Romania, Russian Federation, Turkey,

United Kingdom, United States of America and Vietnam.

PT samples:

- one soil sample (blank soil),
- two spiked soil samples activity level of Cs-137 is ~ 10 times of the blank; and for Pb-210 is ~ 5 times.
- two spiked soil samples, activity level of Cs-137 and Pb-210 are ~ 2 times of previous samples,

Interlaboratory studies 2005-2008

- Spiking procedure for Cs-137 and Pb-210 in soil was developed;
- A Chinese soil was used a a raw material to prepare spiked sources;

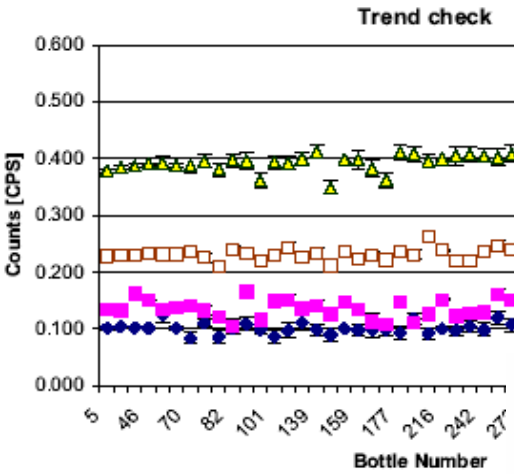


Fig. 2 Gamma-spectrometry measureme soil bottles (^{210}Pb, ^{109}Cd, ^{241}Am, ^{134}Cs)

Anal Bioanal Chem
DOI 10.1007/s00216-006-0772-z

ORIGINAL PAPER

Matrix materials for proficiency testing: optimization of a procedure for spiking soil with gamma-emitting radionuclides

A. Shakhashiro · A. M. Gondin da Fonseca Azeredo · U. Sansone · A. Fajgelj

Received: 2 June 2006 /Revised: 3 August 2006 /Accepted: 10 August 2006

Interlaboratory studies 2005-2008

the result passes trueness criteria if:

$$A1 < A2$$

$$A1 = \left| Value_{IAEA} - Value_{Laboratory} \right|$$

$$A2 = 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

the result passes precision criteria if

$$P \leq LAP_{max}(\%)$$

$$P = \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab.}}\right)^2} X100\%$$

T	P	MAB	Final score
A	A	-	A
A	N	A	W
N	A	A	W
N	A	N	N
A	N	N	N
N	N	-	N

T-trueness criteria

P- precision criteria

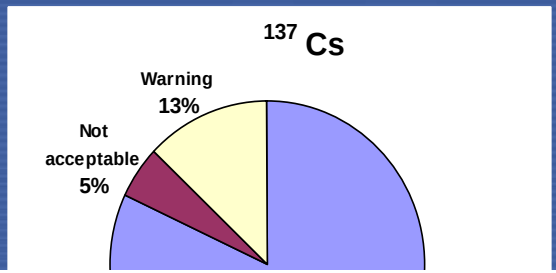
MAB-maximum acceptable bias

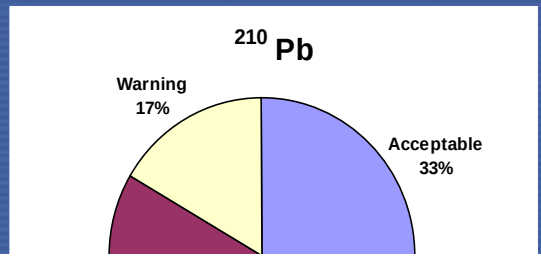
A-acceptable, N- not acceptable

W-warning

Interlaboratory studies 2005-2008

- The analytical data evaluation of this proficiency test indicates that 82% of the laboratories reported "acceptable" results for the Cs-137 and 33% for Pb-210.





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Results of an IAEA inter-comparison exercise to assess ^{137}Cs and total ^{210}Pb analytical performance in soil

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Interlaboratory studies 2005-2008

ALMERA - Analytical Laboratories for the Measurement of Environmental RAdioactivity

PTs 2006, 2007

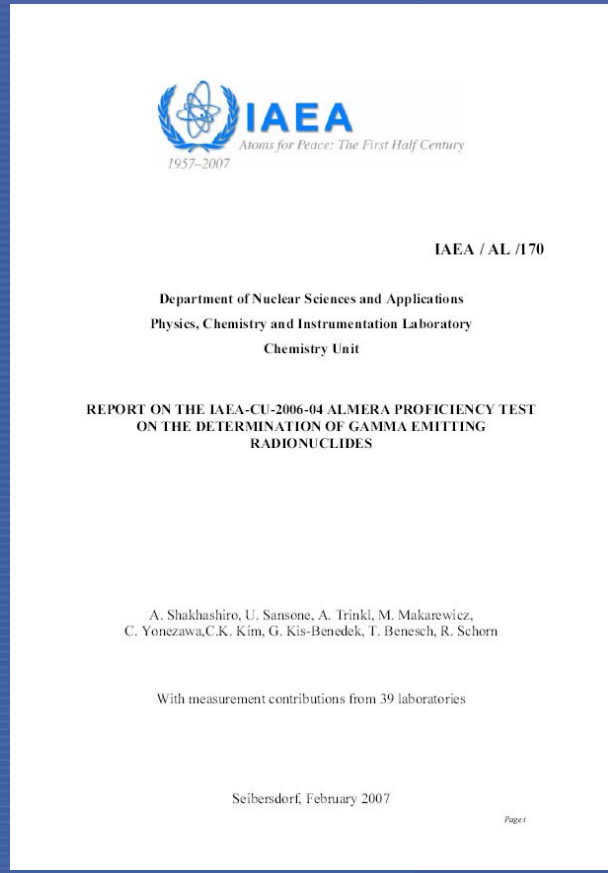
Interlaboratory studies 2005-2008

- 1995, ALMERA network was established, (53 laboratories from 26 different countries)
- 2008, 134 laboratories from 76 different countries)

- 3 working days reporting time for gamma emitting RN;

- Performance evaluation results are not anonymous;

- In the ALMERA 2006 Proficiency Test 195 PT samples (soil, grass, water) were distributed in June 2006;



IAEA
Atoms for Peace: The First Half Century
1957–2007

IAEA / AL /170

Department of Nuclear Sciences and Applications
Physics, Chemistry and Instrumentation Laboratory
Chemistry Unit

REPORT ON THE IAEA-CU-2006-04 ALMERA PROFICIENCY TEST ON THE DETERMINATION OF GAMMA EMITTING RADIONUCLIDES

A. Shakhashiro, U. Sansone, A. Trinkl, M. Makarewicz, C. Yonezawa,C.K. Kim, G. Kis-Benedek, T. Benesch, R. Schorn

With measurement contributions from 39 laboratories

Seibersdorf, February 2007

Page i

Interlaboratory studies 2005-2008

Participants:

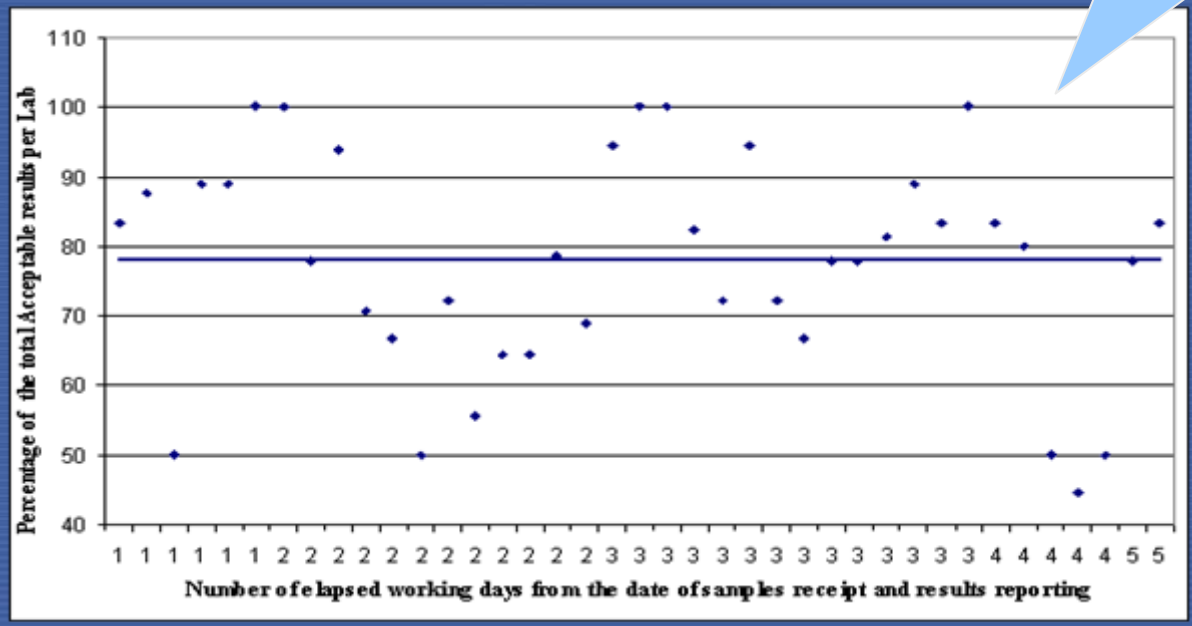
ALMERA: **65 laboratories** from **43 countries registered and received PT materials,** 39 Laboratories from 28 countries reported their results.

PT ALMERA Meeting November 2007

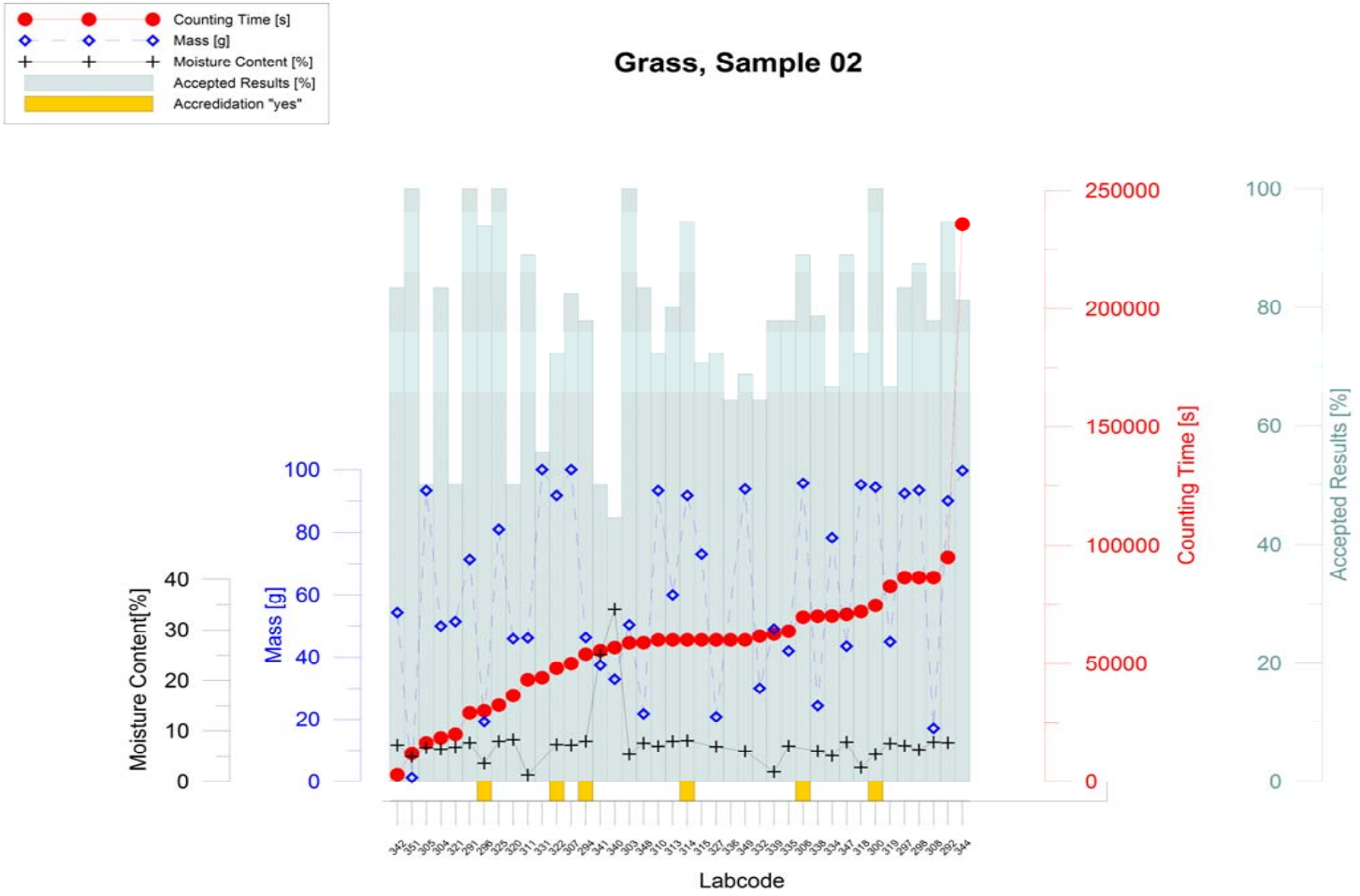
Interlaboratory studies 2005-2008

Analytical performance level vs. number of elapsed working days from the date of samples receipt and results reporting. The line represents the average in percentage of the total acceptable results

3 days reporting time



Interlaboratory studies 2005-2008



Interlaboratory studies 2005-2008

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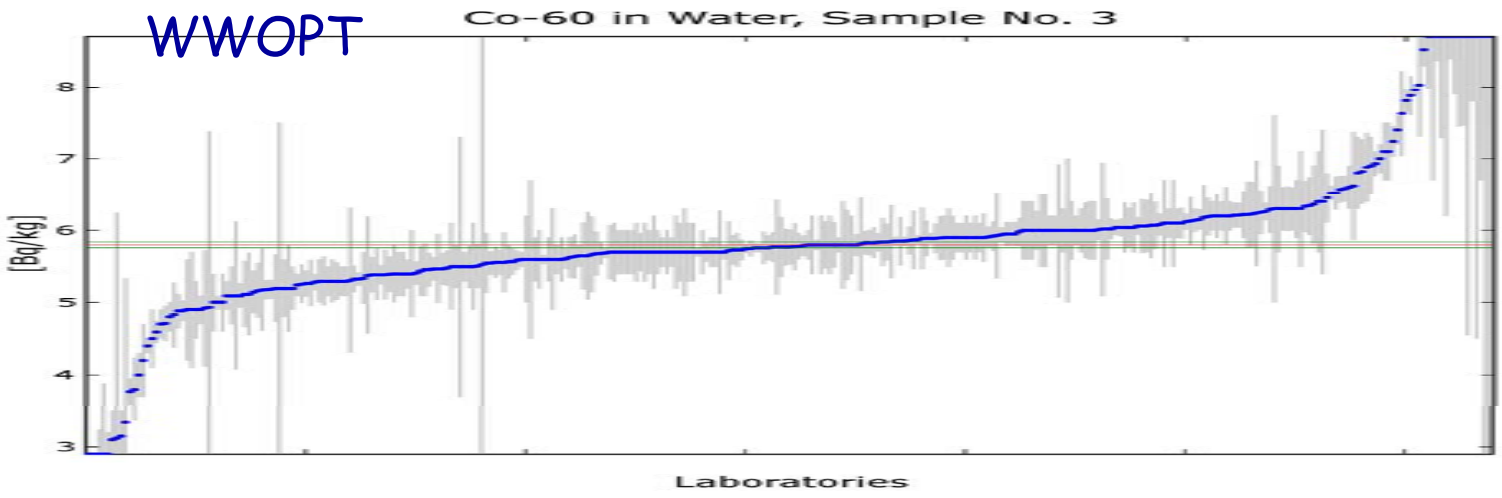
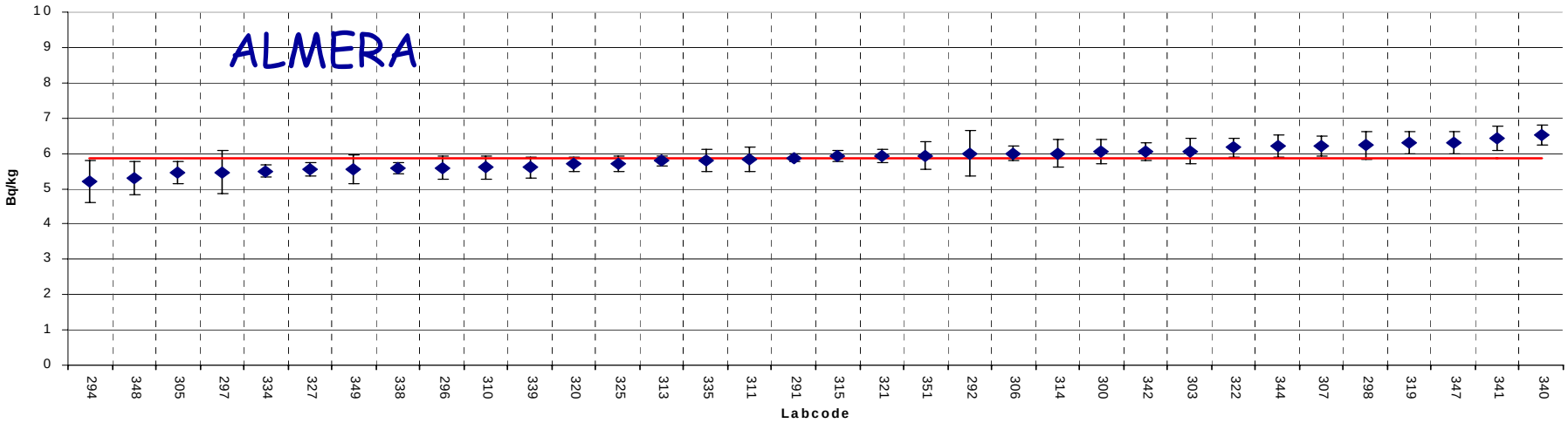


ALMERA



Interlaboratory studies 2005-2008

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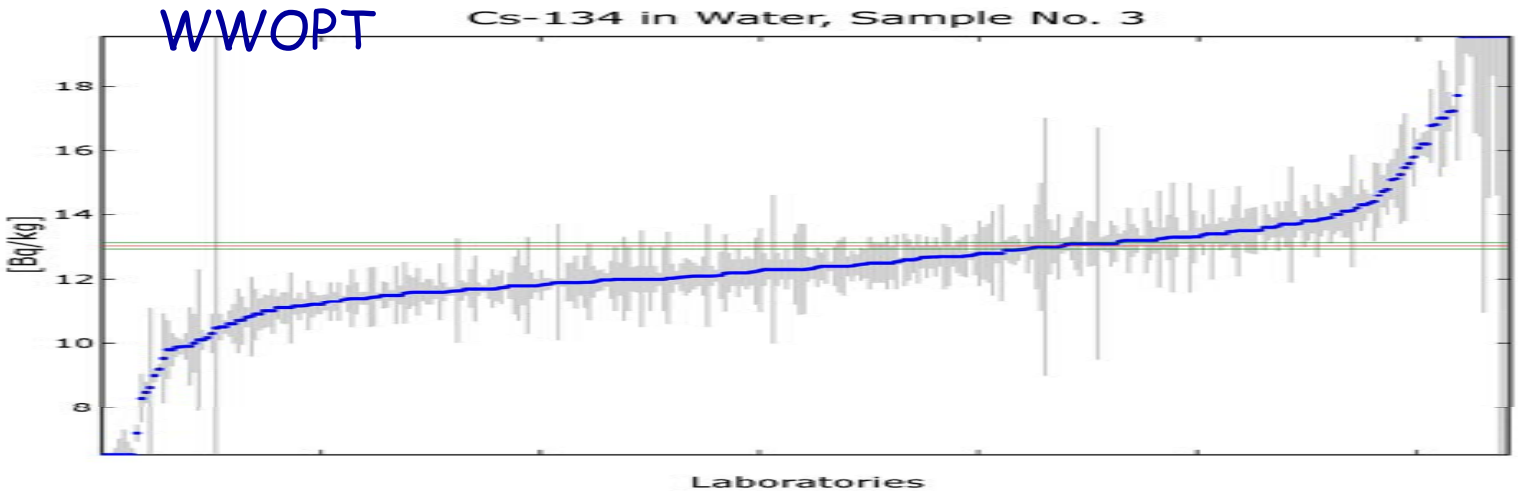
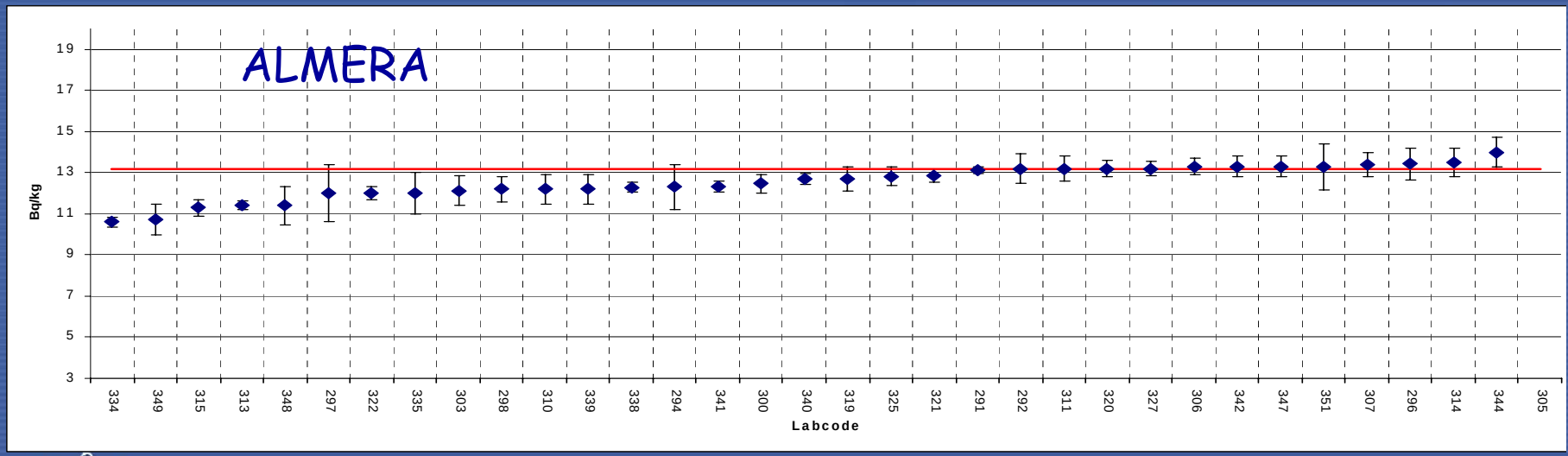


ALMERA



Interlaboratory studies 2005-2008

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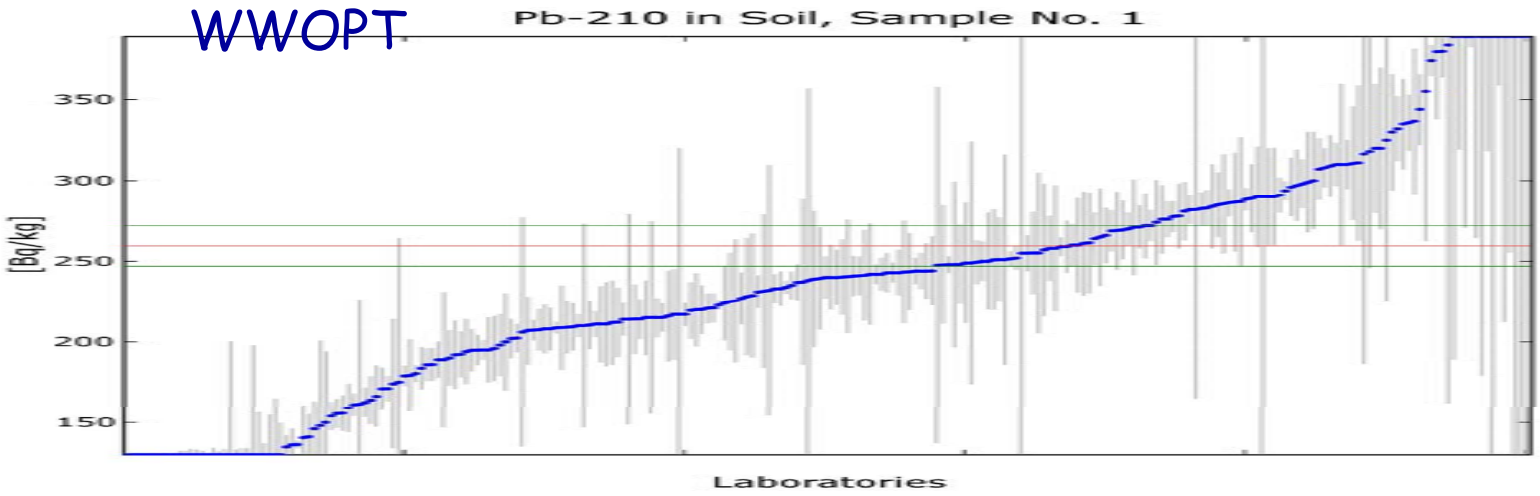
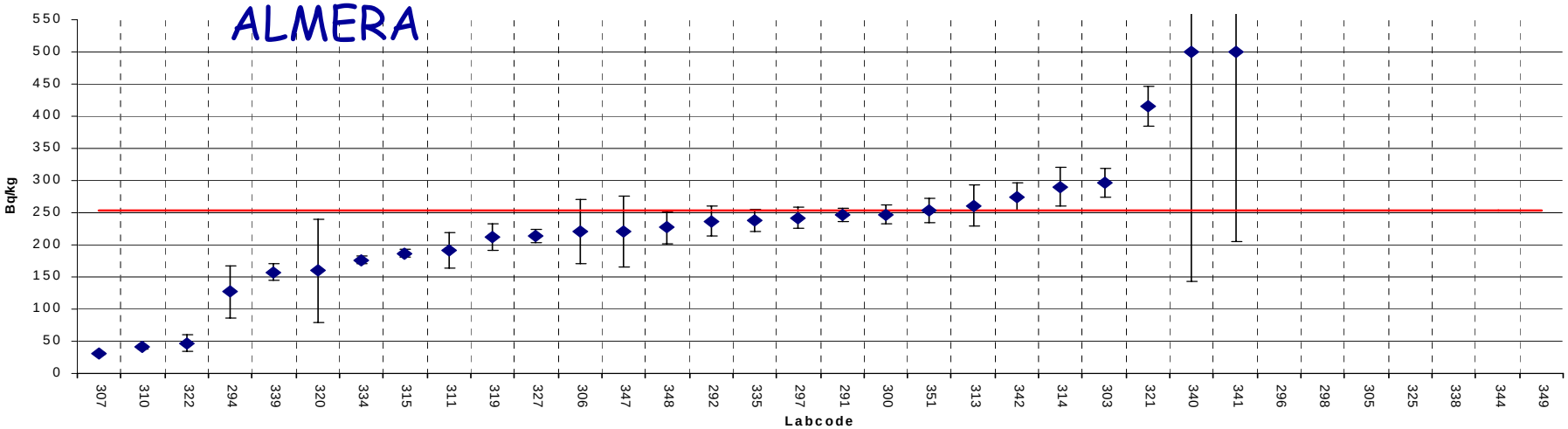
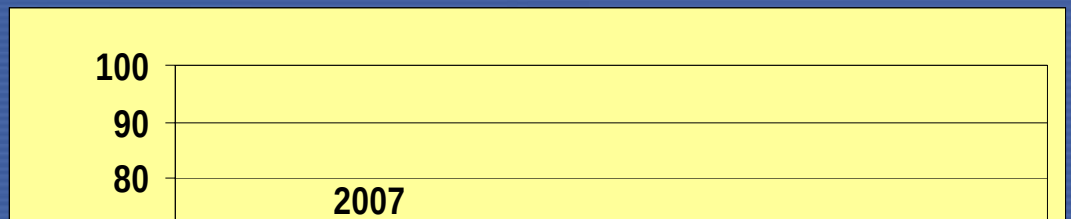


ALMERA



Interlaboratory studies 2005-2008

ALMERA Results (Pb-210)



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The IAEA's 'ALMERA Network' proficiency test on the determination of gamma-emitting radionuclides: A test of results comparability

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ARTICLE INFO

Keywords:
Proficiency testing
Gamma-emitting radionuclides
Water
Soil
Grass

ABSTRACT

The International Atomic Energy Agency (IAEA) coordinates the work of a world-wide network of analytical laboratories, the Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA) network. A proficiency test for ALMERA members was organized in 2006 based on the determination of gamma-emitting radionuclides (^{54}Mn, ^{60}Co, ^{65}Zn, ^{109}Cd, ^{134}Cs, ^{137}Cs, ^{241}Am and ^{210}Pb) in three matrices: water, soil and grass. This paper presents the methodology applied in this proficiency test and discusses the results of the analytical performance evaluation for 38 participating laboratories. The paper also addresses some technical root causes, which could explain low performances in the determination of ^{109}Cd and ^{210}Pb.

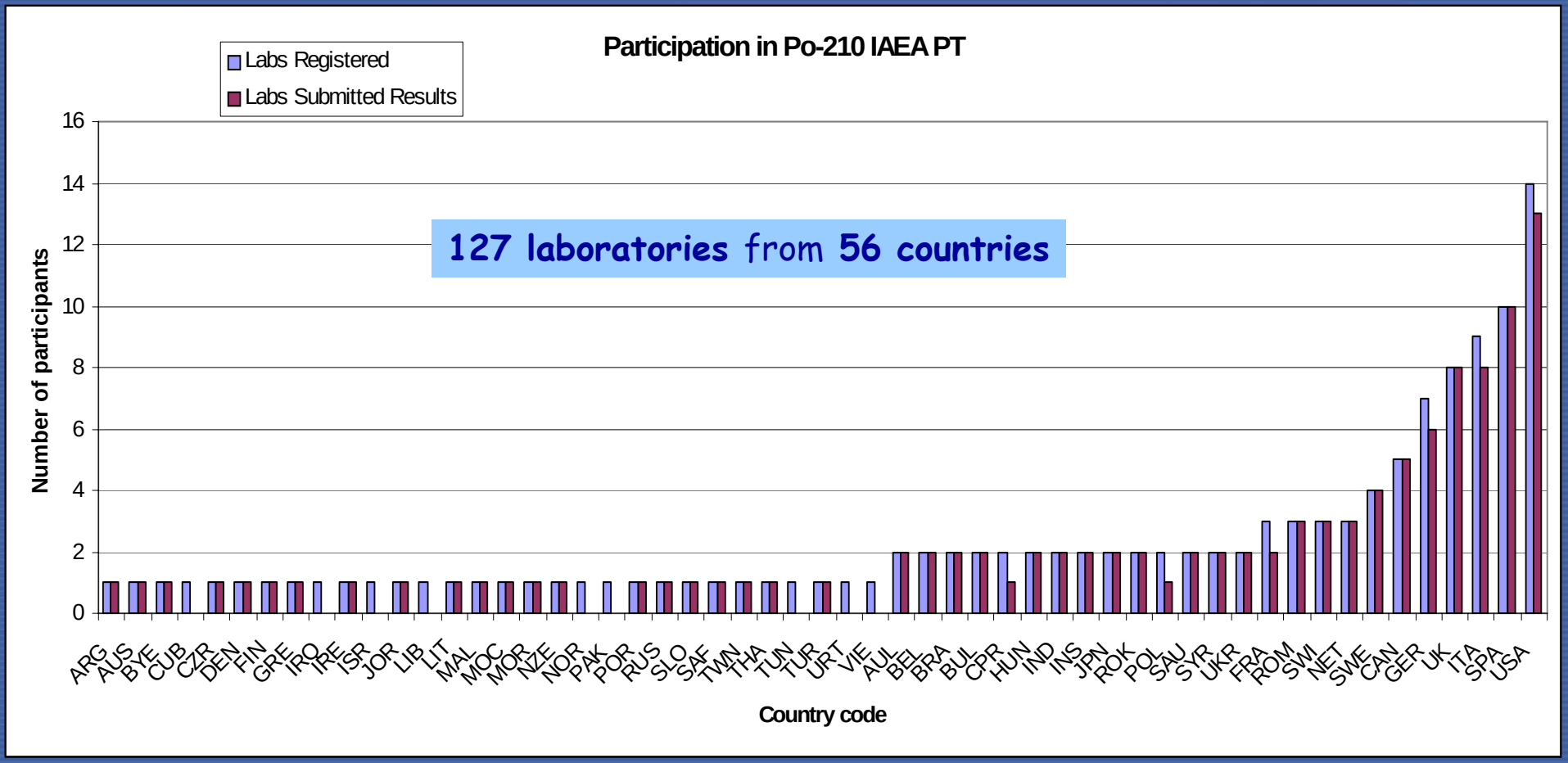
Interlaboratory studies 2005-2008

IAEA-CU-2007-09 ALMERA and world wide Proficiency Test on the determination of Po-210 in water

Interlaboratory studies 2005-2008

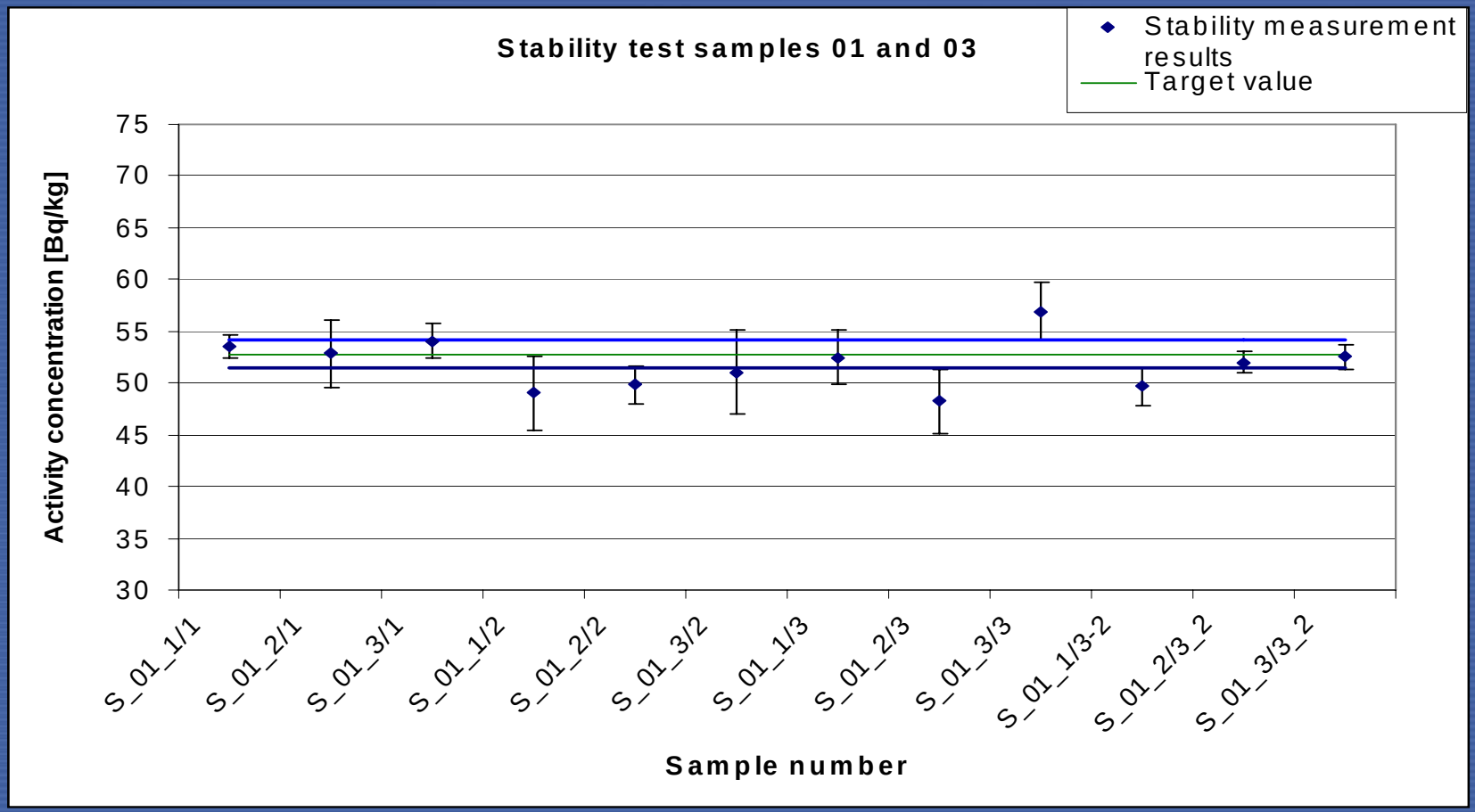
- To gather information on the current state of practice for Po-210 measurements at different activity levels in aqueous samples
- To check false positive reporting
- To explore options for method development.

Interlaboratory studies 2005-2008

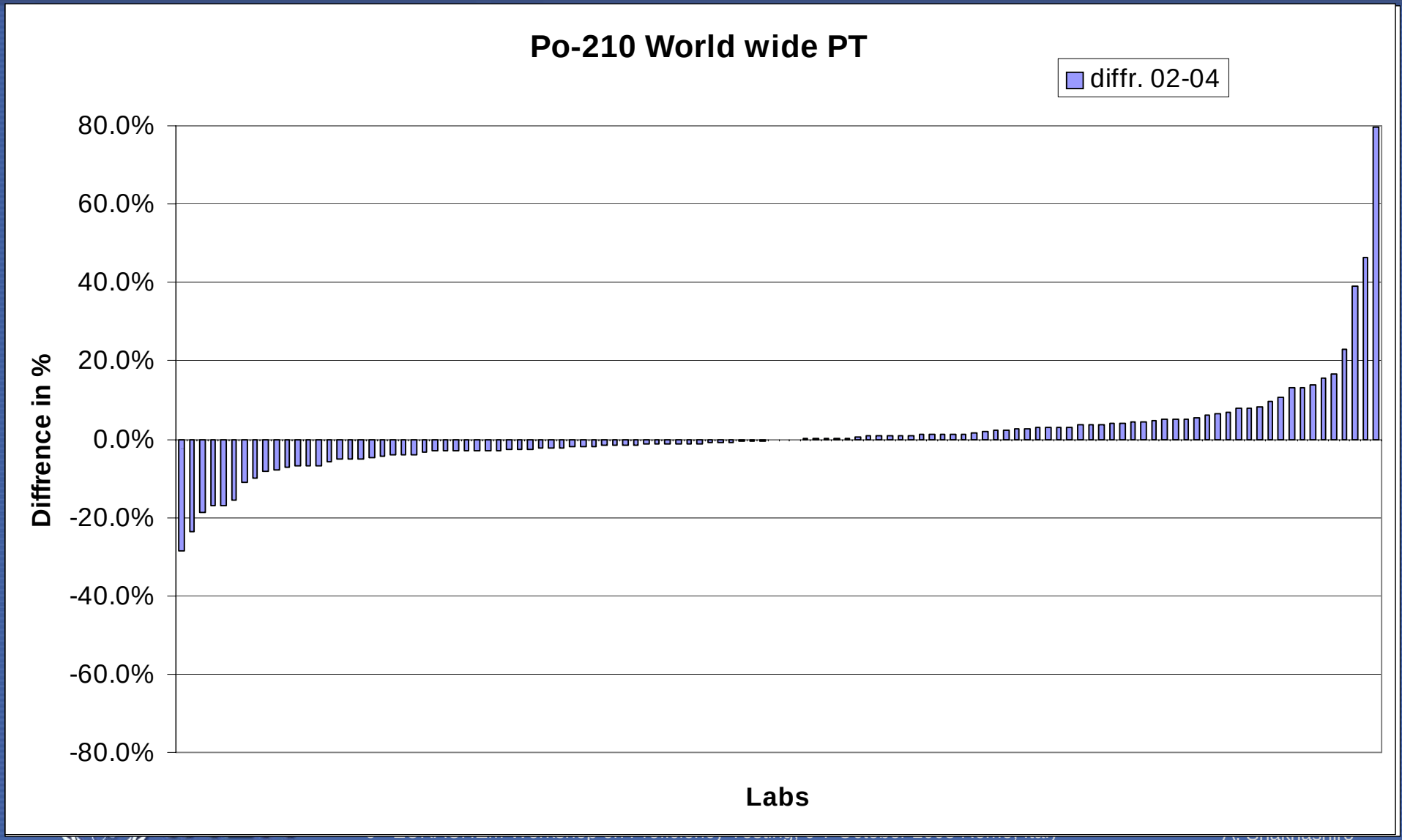


Interlaboratory studies 2005-2008

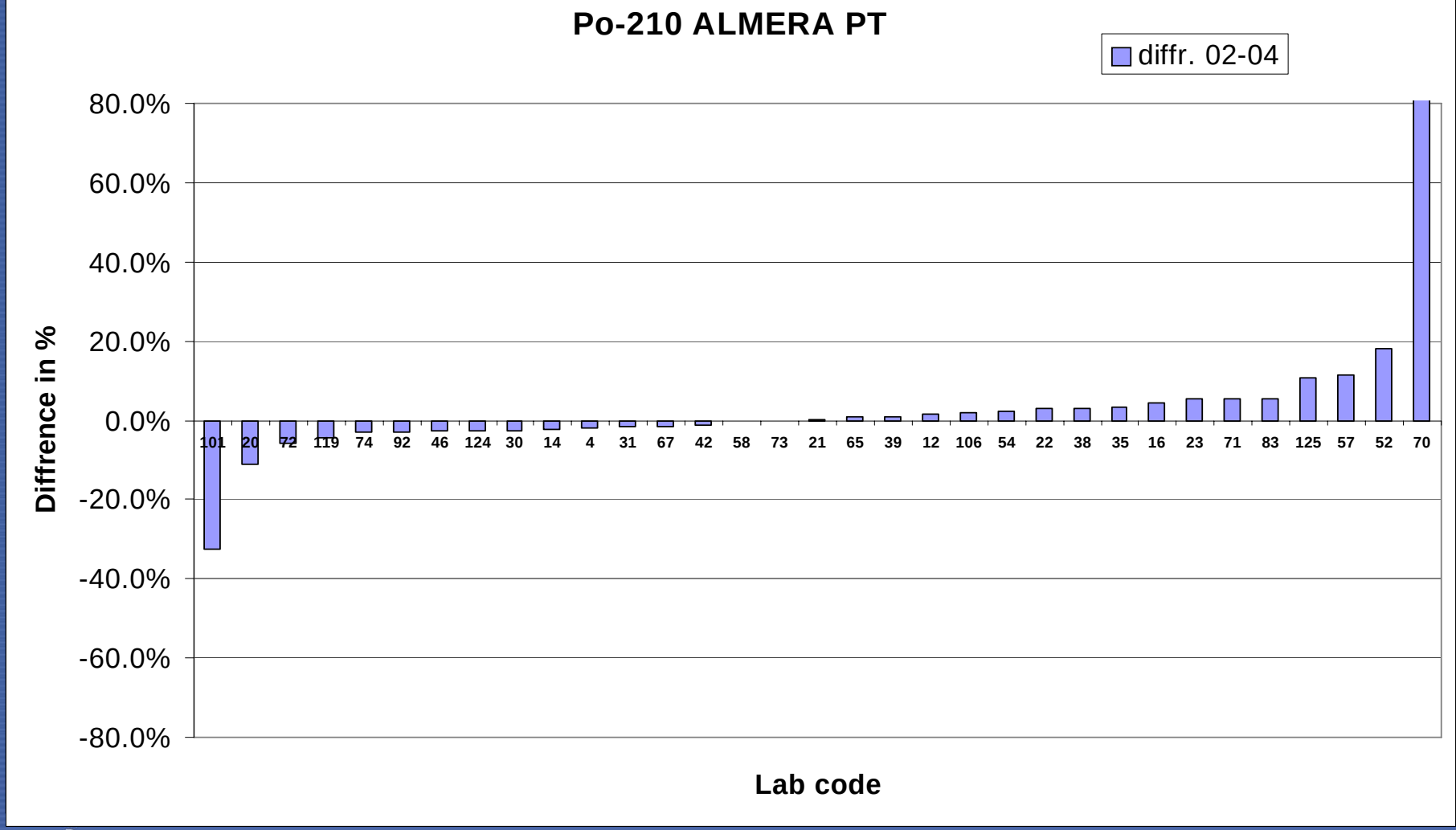
Stability test: from 19 March to 7 May 2007



Interlaboratory studies 2005-2008

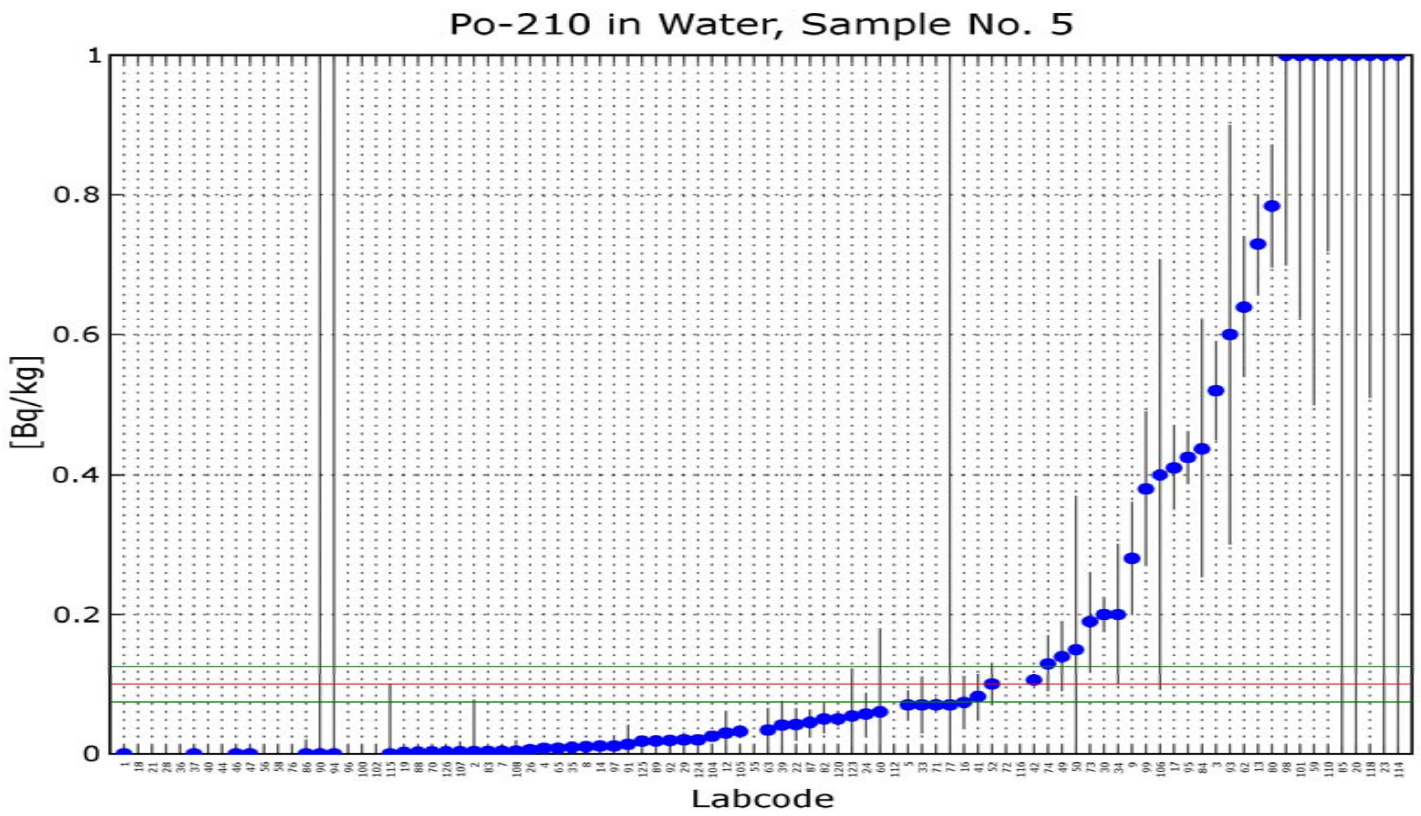


Interlaboratory studies 2005-2008



Interlaboratory studies 2005-2008

Blank sample results



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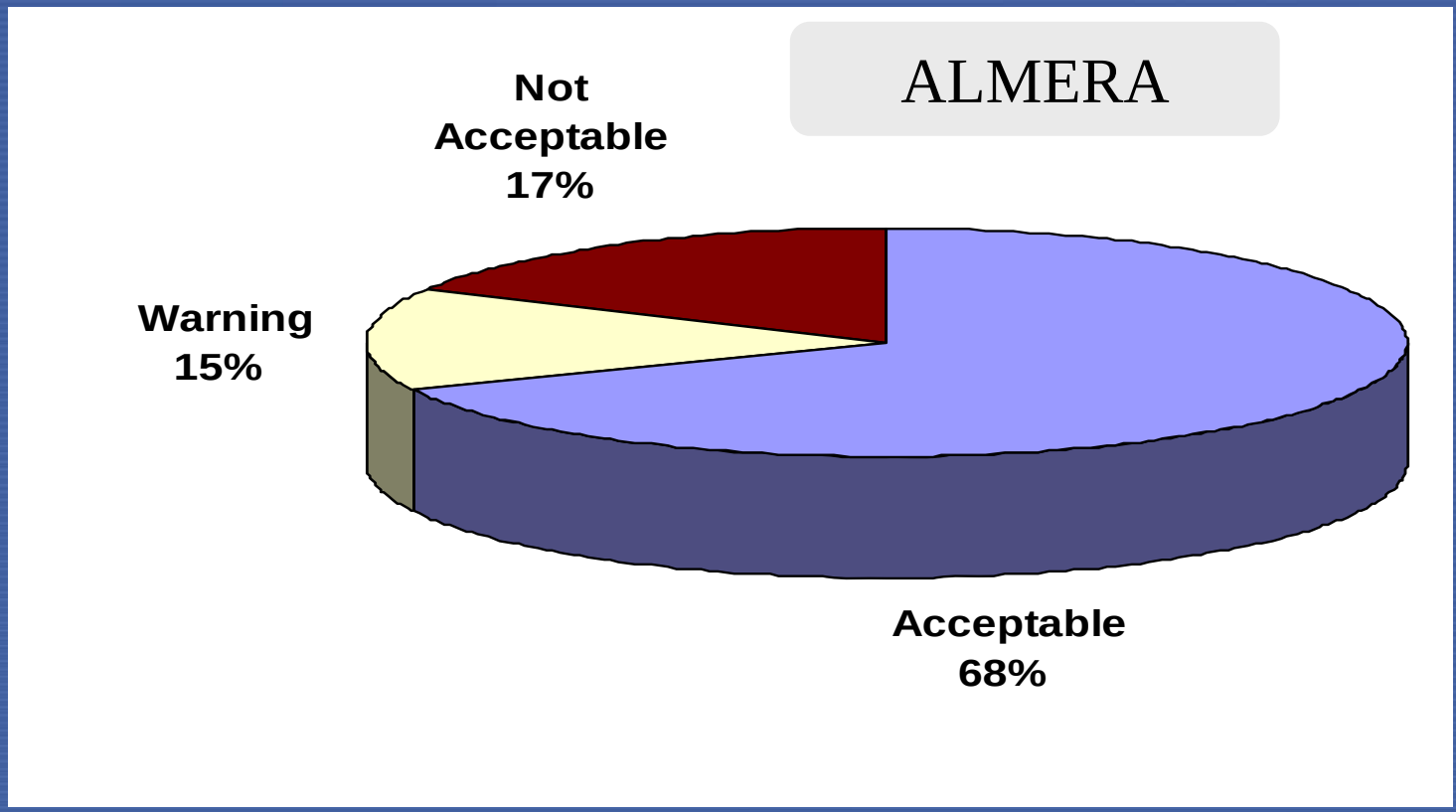
Interlaboratory studies 2005-2008



Final report was issued and could be downloaded at URL:
http://www.iaea.org/programmes/aqcs/icpt/icpt_po210.html

Interlaboratory studies 2005-2008

Proficiency Test

Gamma emitters in soil, water and grass

IAEA-CU-2006-03 (World Wide Open)

IAEA-372 Grass CRM

Interlaboratory studies 2005-2008

- The following proficiency test design was applied:
  - one spiked soil sample (200g)
  - one natural grass sample (100 g)
  - one spiked water sample (500 ml)

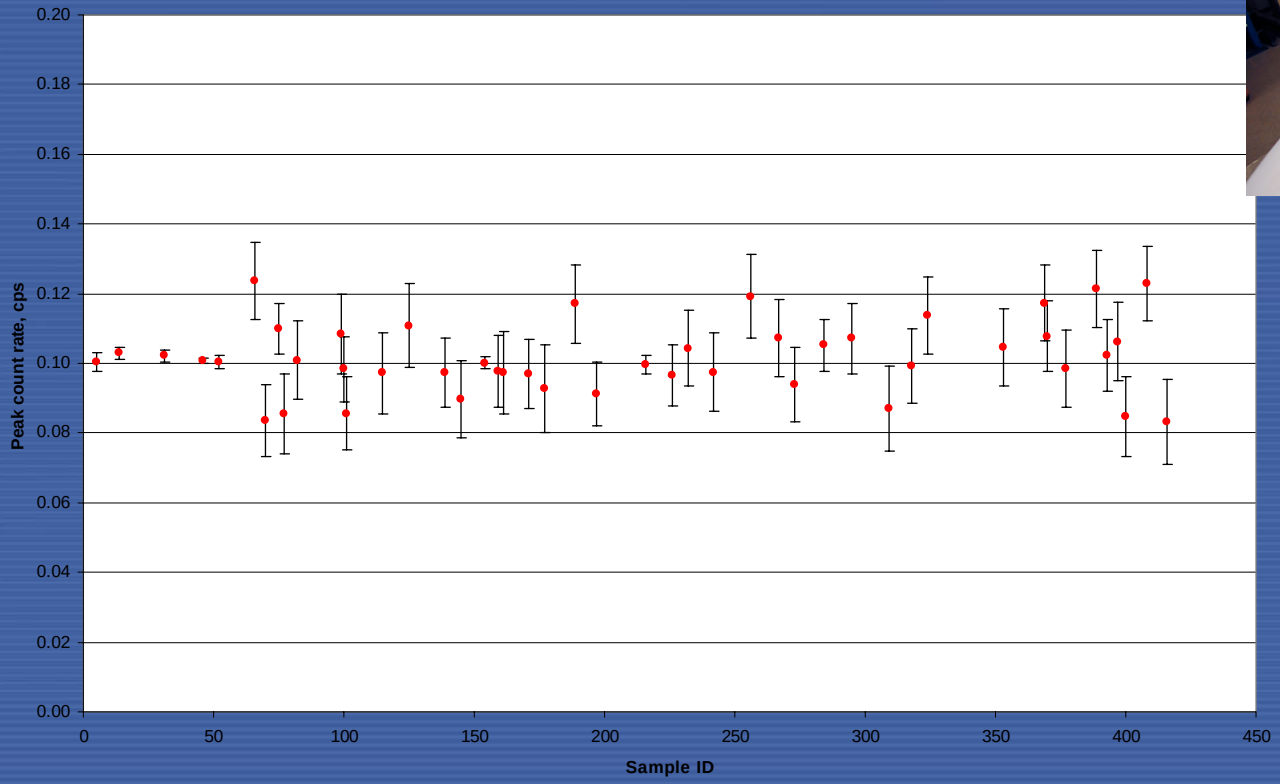
Participants:
605 applications received
401 laboratories from 85 countries registered and received PT materials,

327 Laboratories from 78 countries reported their results.

Interlaboratory studies 2005-2008

Trend check

46 keV (Pb-210) peak count rate
(counting time: 3600-53000 sec)



Interlaboratory studies 2005-2008

Trend check

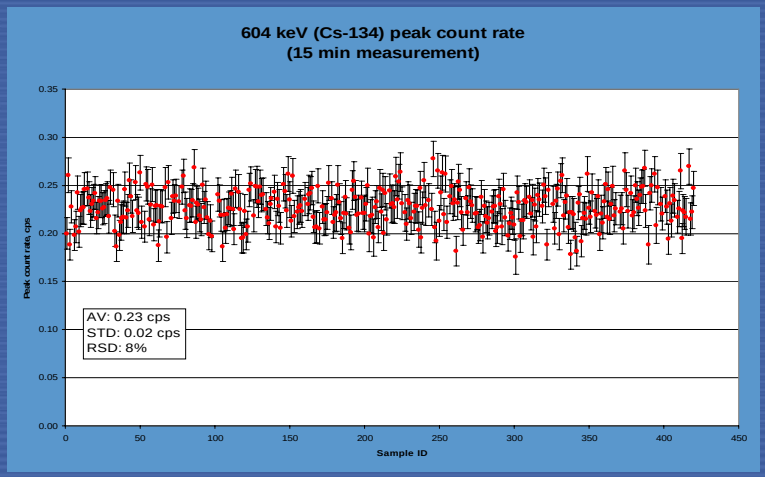
	^{210}Pb	^{241}Am	^{109}Cd	^{134}Cs	^{137}Cs	^{54}Mn	^{65}Zn	^{60}Co
Slope	1.303 E-05	3.667 E-05	-1.110 E-05	3.196 E-06	-9.595 E-06	-3.755 E-06	-1.336 E-05	7.996 E-06
u-Slope	1.230 E-05	2.045 E-05	2.295 E-05	1.122 E-05	9.391 E-06	9.891 E-06	1.073 E-05	7.121 E-06
df	44	44	44	44	44	44	44	44
Slope/u	1.058	1.792	-0.484	-0.323	-1.021	-0.379	-1.224	1.122
T-critical (0.05, 44)	2.01	2.01	2.01	2.01	2.01	2.01	2.01	2.01

Interlaboratory studies 2005-2008

Uncertainty associated with between bottles heterogeneity

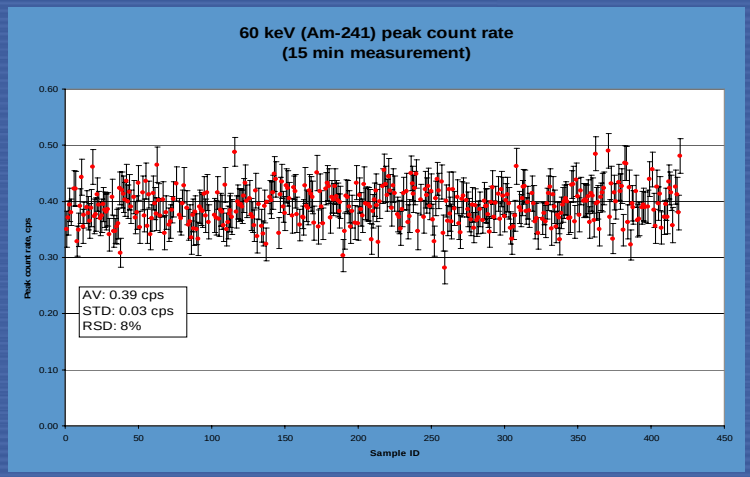
RN	^{210}Pb	^{241}Am	^{109}Cd	^{134}Cs	^{137}Cs	^{54}Mn	^{65}Zn	^{60}Co
U_{bb} (%)	0.60	0.30	1.22	0.24	0.36	0.29	0.43	0.50

- 401 samples were measured to detect any gross error during the preparation.



Peak count rate. cps

Bottle number



Bottle number

Interlaboratory studies 2005-2008

Assignment of the property values of the IAEA-372

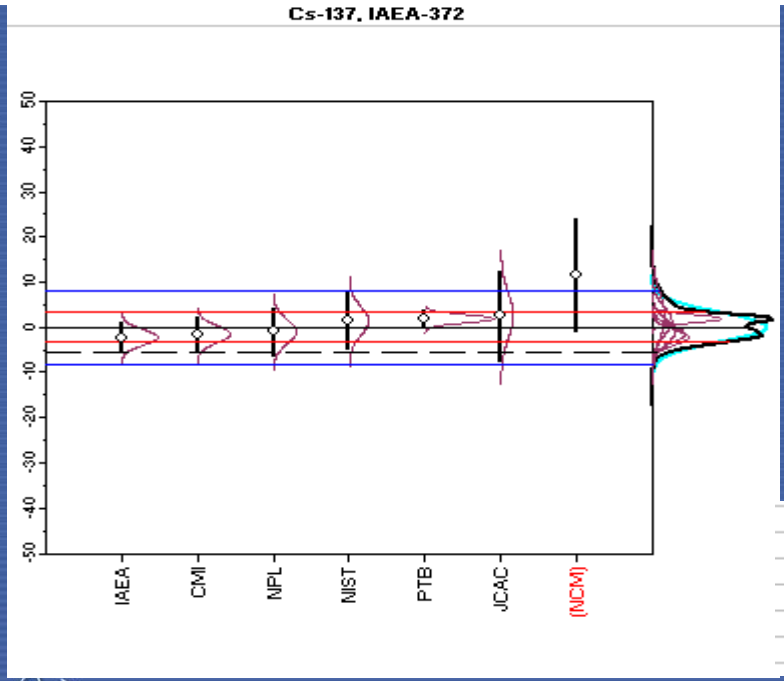
- The material was bottled at 100 g in 500 ml HPE containers,
- After bottling 10 bottles from 750 were selected randomly and 10 sub-samples were measured at 5 g for homogeneity test;

	5 g (10 ccm) 63x63 -mm well type det. (NaI(Tl)) [counts]									
Bott.#	24	86	178	243	310	392	470	574	648	701
	23480	23230	22720	23290	23400	23650	23510	24180	23780	23400
	23520	23300	23290	22330	23480	23900	23470	24320	23280	22820
	22410	22880	23140	23160	23300	23850	23820	23960	24540	23550
	23250	23230	23410	23210	23570	23190	22350	23570	24070	23140
	23280	23590	23100	22860	22700	23580	23180	24640	23240	23250
	23720	23000	23670	23050	22910	23040	23680	24490	24030	23450
	22690	23550	23260	22780	22650	23910	23360	24770	23710	22970
	22780	23900	23830	22820	23690	24110	23303	24010	23740	22640
	22550	24270	22700	23130	23460	23600	23030	23610	23040	23500
	23910	23210	22730	22990	23450	24330	23200	24020	23260	23160
average	23159	23416	23185	22962	23261	23716	23290	24157	23669	23188
sd	520	421	392	281	370	393	407	404	468	304
rel. Sd	2.24	1.80	1.69	1.22	1.59	1.66	1.75	1.67	1.98	1.31

Interlaboratory studies 2005-2008

Code	Value (Bq kg^{-1})	Uncertainty		Laboratory result/MM-median value
		Bq kg^{-1}	%	
NIST	11470	356	3.10	1.01
CMI	11139	210	1.89	0.98
JCAC	11600	560	4.83	1.02
PTB	11526	100	0.87	1.02
IAEA	11060	190	1.72	0.98
NPL	11200	300	2.68	0.99
NCM	12618	702	5.56	1.11

Assignment of the property values of the IAEA-372



* Ref. D. Duewer (NIST)

Interlaboratory studies 2005-2008

Assignment of the property values of the IAEA-372

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The new IAEA-372 grass-certified reference material for ^{40}K and ^{137}Cs

A. Shakhashiro [a,*], U. Sansone [a], D. Arnold [b], P. Dryak [c], Jerome J. La Rosa [d], S.M. Jerome [e], M. Makarewicz [a], J. Mentcheva [f], K. Sato [g], S. Tarjan [h]

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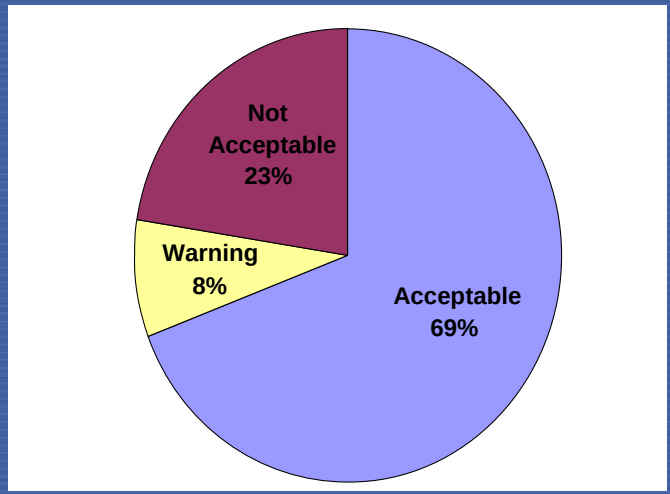
Interlaboratory studies 2005-2008

Participants:

401 laboratories from **85 countries registered and received PT materials,** 327 Laboratories from 78 countries reported their results.

Summary evaluation of all reported data
4850 measurement results:



Planned interlaboratory studies 2008-2010

- ALMERA and world wide open PT on the determination of naturally occurring radionuclides in phosphogypsum, 2008.
- Latin America regional PT on the determination of radionuclides and trace elements in water, soil and sediment, technical cooperation,2009.
- ALMERA and World wide open PT on the determination of radionuclides in water and soil, 2009.
- ALMERA and World wide open PT on the determination of radionuclides in water and soil, 2010.
- World wide open PT on the determination of trace elements and Platinum in Algae, 2010.
- West Asia regional PT on the determination of trace elements in sludge, 2010.

Planned reference materials 2008-2011

IAEA-344 RN in sediment;

IAEA-360 RN in soil;

IAEA-377 RN in soil;

IAEA-447 RN in moss-soil;

IAEA-448 Ra-226 from in soil oil field;

IAEA-451 Ra-226 in water oil field;

IAEA-450 Pt and trace elements in Algae,

IAEA-452 Trace elements in sludge.

http://www.iaea.org/programmes/aqcs/interlab_studies.shtml

Training and fellowships

- Fellows training on PT and RM,
- Interregional TC projects on PT and RM INT-054,
- Latin America regional TC on QA/PT/RM,

The Agency's Laboratories Seibersdorf

http://www.iaea.org/programmes/aqcs/interlab_studies.shtml

IAEA
International Atomic Energy Agency