



*The Abdus Salam
International Centre for Theoretical Physics*



1833-1

**Workshop on Understanding and Evaluating Radioanalytical
Measurement Uncertainty**

5 - 16 November 2007

An introduction to the IAEA Terrestrial Environment Programme

Paul MARTIN
*International Atomic Energy Agency IAEA
Agency's Laboratories Seibersdorf
Chemistry Unit, A-2444 Seibersdorf
AUSTRIA*

An introduction to the IAEA Terrestrial Environment Programme

Paul Martin
IAEA Laboratories Seibersdorf



IAEA

Atoms for Peace: The First Half Century
1957–2007

ALMERA, ICTP, Trieste
Nov. 2007

CONTENTS

- A quick introduction to the IAEA programmatic structure
- Some IAEA Environment Programme activities in 2006/07 and 2008/09

Major Programmes

1. Nuclear power, fuel cycle & nuclear science
2. Nuclear techniques for development and environmental protection
3. Nuclear safety and security
4. Verification
5. Information support
6. Management of Technical Cooperation
7. Policy and general management

Major Programme 2

E. Food and Agriculture

F. Human Health

G. Water Resources

H. **Environment**

I. Physical and Chemical Applications

IAEA Environment Programme

- Marine env. & radiological assessment
- Coastal marine problems
- Ocean climate coupling

- Supporting analytical lab. performance
- Sustainable management of the terrestrial environment

IAEA Environment Programme

Marine Environmental Laboratory, Monaco

- Marine env. & radiological assessment
- Coastal marine problems
- Ocean climate coupling

Seibersdorf Laboratories, Austria

4. Supporting analytical lab. performance
5. Sustainable management of the terrestrial environment

Supporting analytical laboratory performance

- **Project 1**
 - *Laboratory Quality Management Activities and Metrology*
- **Project 2**
 - *IAEA Reference Materials*
- **Project 3**
 - *Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA)*

Sustainable management of the terrestrial environment

- **Project 1**
 - *Terrestrial Radioecology*
- **Project 2**
 - *Ecotoxicology*
- **Project 3**
 - *Remediation Strategies*

SOME IAEA ENVIRONMENT PROGRAMME ACTIVITIES IN 2006/07



IAEA

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

Development of analytical methods for use by Member State laboratories

Po-210 method review plus a laboratory comparison of selected methods

Pu isotopes by ICP-MS method review



Available online at www.sciencedirect.com

ScienceDirect

Applied Radiation and Isotopes 65 (2007) 267–279

Applied Radiation and Isotopes

www.elsevier.com/locate/apradiso

Review

Determination of ^{210}Po in environmental materials: A review of analytical methodology

K. Murray Matthews^{a,*}, Chang-Kyu Kim^b, Paul Martin^{b,*}

^aRadioactivity Specialists Ltd, Christchurch, New Zealand
^bAgency's Laboratories, Seibersdorf, International Atomic Energy Agency, A-1400 Vienna, Austria
Received 8 June 2006; received in revised form 7 August 2006; accepted 19 September 2006

Abstract

Polonium-210 (^{210}Po) is analysed for a variety of purposes, including for radiological impact assessment, as a tracer of environmental processes, and as an indirect measure of its progenitor lead-210 (^{210}Pb). Losses of polonium may occur at temperatures above 100 °C, depending on conditions, requiring particular care in sample preparation and treatment. In spite of this problem, the analysis of ^{210}Po is relatively straightforward, due to the ease of source preparation through spontaneous auto-deposition onto metal surfaces and the uncomplicated spectrum. Although several optimisation studies have been carried out, published source preparation methods remain remarkably diverse. Some areas where further study would be useful are identified.

© 2006 Elsevier Ltd. All rights reserved.

Keywords: Polonium; Environmental; Sample; Analysis; α -spectrometry; Review

Contents

1. Introduction	268
2. Sample preparation	270
2.1. Soils and sediments	270
2.2. Biological materials	270
2.3. Water	271
3. Extraction/separation	271
3.1. Solvent extraction	272
3.2. Ion-exchange chromatography	272
3.3. Extraction chromatography	272
4. Source preparation	273
5. Radioactivity measurements and calculations	274
5.1. α -spectrometry system	274
5.2. Spectrum analysis	274
5.3. Calculation of activity concentration in the sample	275
5.4. Detection limit	275
6. Conclusions	276
References	277

*Corresponding author. Tel.: +431 260028207; fax: +431 260028222.
E-mail address: p.martin@iaea.org (P. Martin).

0969-8043/\$ - see front matter © 2006 Elsevier Ltd. All rights reserved.
doi:10.1016/j.apradiso.2006.09.005

CRITICAL REVIEW

www.rsc.org/jaas | Journal of Analytical Atomic Spectrometry

Determination of Pu isotope concentrations and isotope ratio by inductively coupled plasma mass spectrometry: a review of analytical methodology

Cheol-Su Kim,^{a,*} Chang-Kyu Kim,^b Paul Martin^b and Umberto Sansone^b

Received 1st December 2006, Accepted 14th March 2007
First published as an Advance Article on the web 29th March 2007
DOI: 10.1039/b617588f

A number of analytical methods for Pu isotopes based on ICP-MS measurement have been developed and applied to the measurement of ultra-trace levels of Pu in nuclear fuel samples and environmental samples. The endeavour to improve the detection limit to sub- 10^{-11} g (g) mL⁻¹ has been continued by using efficient sample introduction systems. This review summarizes and critically discusses various types of ICP-MS, sample introduction systems and separation methods applied to the determination of Pu in many sample types. Together with the separation method, the adjustment of the Pu oxidation state in the loading solution preparation step and Pu elution step is summarized. The interference problem from polyatomic ions in ICP-MS coupled with various sample introduction systems is described, especially focused on the UH⁺ interference. Several factors affecting the trueness and precision of Pu results are discussed.

1. Introduction

From the viewpoint of radio-toxicity and long-term radiation effects to humans, plutonium (Pu) is by far the most important of the transuranic elements that have been released into the environment. It is present in the environment mainly as a result of nuclear weapon tests, nuclear accidents, and reprocessing of nuclear fuels. Approximately 15 TBq of $^{239+240}\text{Pu}$ have been released into the environment from atmospheric weapon tests carried out in the 1950s and 1960s and a few GBq of Pu have been released from fuel reprocessing plants.¹ The isotopes present in the environment from these sources are ^{239}Pu , ^{240}Pu , ^{241}Pu and ^{242}Pu , of which ^{239}Pu and ^{240}Pu are the most important due to their extremely long half-lives and high abundance. Pu isotopes are analysed mainly for the purposes of monitoring of the environment around nuclear facilities such as nuclear power plants, nuclear fuel reprocessing plants and nuclear waste storage sites,^{2–4} as well as for surveys of the contaminated areas resulting from nuclear weapons tests,^{5–8} nuclear accidents,^{9,10} and the discharge of nuclear waste.^{11–12} Moreover, Pu has been a major target element in the areas of radio-bioassay,^{13–15} tracing of nuclear weapon material,^{16,17} burn-up measurement of nuclear fuel^{18–24} and public and occupational health safety,²⁵ as well as a tracer in the study of radioecology,¹⁴ geochemistry^{26,28} and the marine environment.^{26–31}

Besides the concentration of Pu isotopes, the Pu isotope ratio ($^{240}\text{Pu}/^{239}\text{Pu}$ isotope ratio) is of great interest because this ratio is used as a fingerprint for different sources and in the study of the transport behaviour of Pu in ecosystems. This is

due to the dominance of the two isotopes in environmental Pu and the well-characterized ratios for various sources. For reactor grade Pu, this ratio can vary from 0.23 to 0.65 according to the irradiation conditions of the fuel.^{32,33} While Pu isotope ratios in fallout, which mainly originate from weapons testing, are characterized by the particular weapon's design and explosion yield.^{33,34} The average Pu isotope ratio observed in global fallout was 0.176 ± 0.014,³⁵ while the ratios in the marine samples of the Northwest Pacific and its marginal seas ranged from 0.18 to 0.34, which is a little higher than that of uncontaminated terrestrial sample.^{36,37}

There are several different techniques used for the determination of Pu isotopes, including alpha-spectrometry,^{38–40} liquid scintillation counting (LSC),^{40–42} fission track,^{43–46} Lx-alpha ray measurement,^{47,48} and mass spectrometry (MS). Of these, alpha-spectrometry is the most widely used analytical technique by virtue of its easy application and relatively low instrumentation cost. However, alpha-spectrometry is not a particularly sensitive technique for the determination of low levels of Pu, sometimes requiring from days to several weeks counting time for environmental samples. Moreover, it is difficult to distinguish ^{239}Pu and ^{240}Pu due to the small difference in their alpha particle energies, restricting the use of this technique in the determination of the Pu isotope ratio.

By contrast, mass spectrometry is an atom counting technique with several advantages over decay counting techniques for the determination of Pu isotopes. It enables determination of low levels of Pu with a low detection limit, while requiring only a short detection time. In addition, it enables the accurate determination of ^{239}Pu and ^{240}Pu , and hence their isotope ratio. So far, various types of mass spectrometric methods have been applied to the determination of concentration and isotope ratios of Pu isotopes, such as thermal ionization mass spectrometry (TIMS),^{49,50,52} accelerator mass spectrometry (AMS),^{53,54} secondary ion mass spectrometry (SIMS),^{55,56}

^aKorea Institute of Nuclear Safety (KINS), P.O. Box 114, Yuseong, Daejeon 305-380, Korea. E-mail: cskim@kins.go.kr; +82-42-82-860-0156; Tel: +82-42-868-0264
^bAgency's Laboratories, International Atomic Energy Agency, Seibersdorf, A1400 Vienna, Austria



IAEA

Atoms for Peace: The First Half Century

1957–2007

IAEA Conference

“Environmental Radioactivity:
From Measurements and
Assessments to Regulation”

256 participants from 56
countries

Vienna

23 – 27 April 2007



Reference Materials

- Move to greater harmonization of reference material production and distribution in the IAEA
- New reference material storage facility



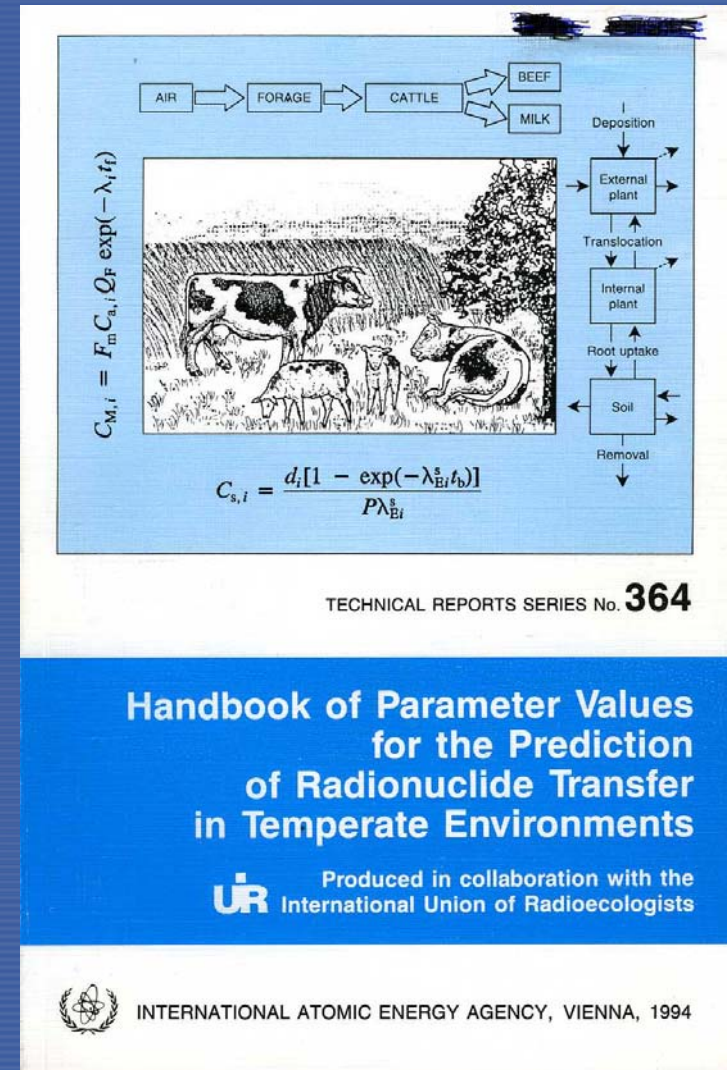
Proficiency Tests – 2007

- **Po-210 in water samples**
- **World-wide PT for gamma emitting radionuclides**
- **ALMERA PT**



Update of TRS364

- Technical Report Series (TRS) 364 on model parameter values will be replaced by:
 - a new TRS with reference information, and
 - a TECDOC with detailed radioecological model information



SOME IAEA ENVIRONMENT PROGRAMME ACTIVITIES IN 2008/09



IAEA

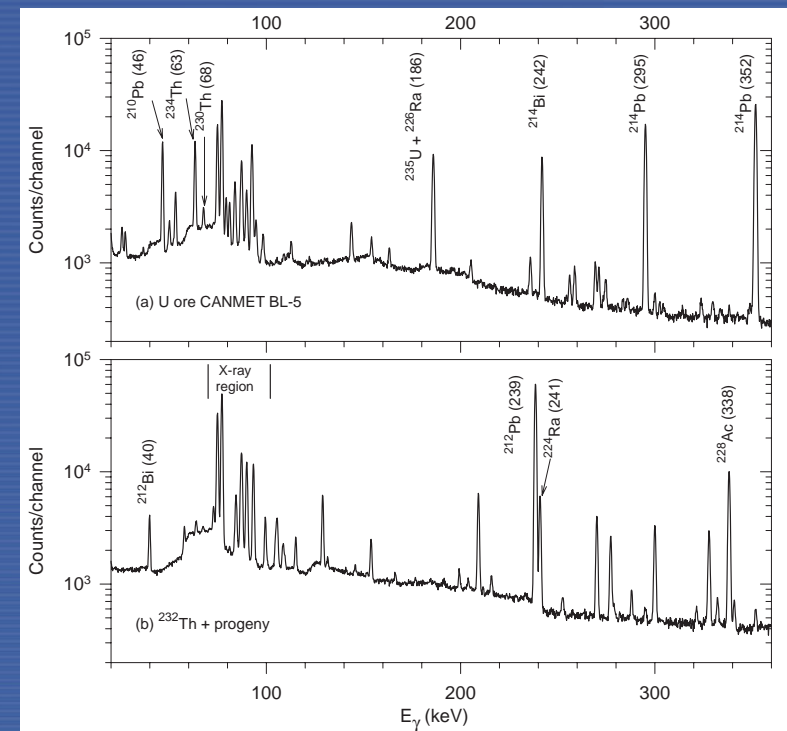
Atoms for Peace: The First Half Century
1957–2007

Coordinated Research Project – I

“Benchmarking Calibration for
Low-level Gamma Spectrometric
Measurements of
Environmental Samples”

Jointly conducted with the IAEA
Marine Environment Laboratory
(Monaco)

To begin in 2008

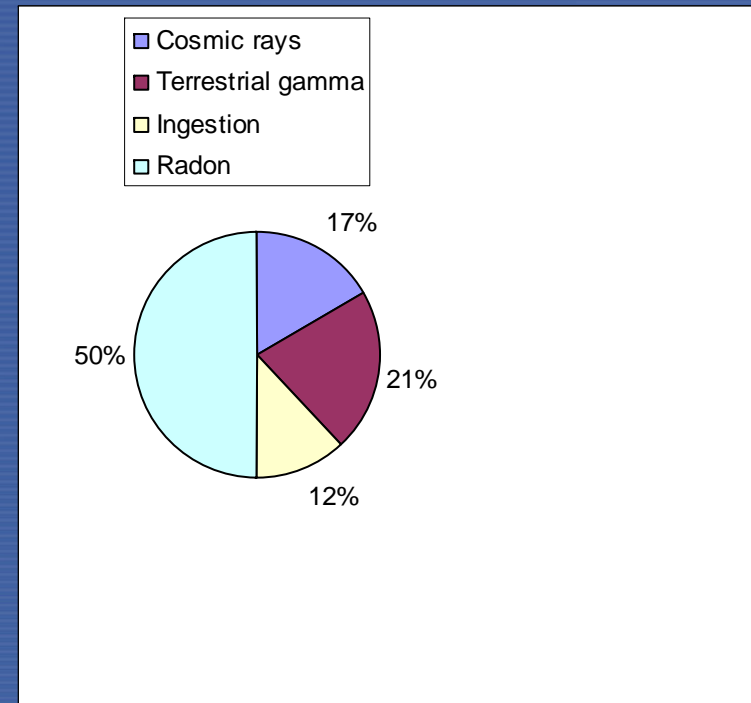


Coordinated Research Project – II

“Development of Methodologies for Radon Surveys”

In cooperation with UNSCEAR

To begin in 2009



Coordinated Research Project – III

“Impact of Radioactive
Particles on Man and
Non-human Species in the
Environment”

To begin in 2009



Isotopic methods for air quality

- 2nd meeting on sources and measurements of natural radionuclides (with WMO)
- Meeting on use of isotopes for the tracing of air pollution

WORLD METEOROLOGICAL ORGANIZATION
GLOBAL ATMOSPHERE WATCH



No. 155

1st INTERNATIONAL EXPERT MEETING
on SOURCES and MEASUREMENTS of NATURAL
RADIONUCLIDES APPLIED to CLIMATE and
AIR QUALITY STUDIES

Sponsored by WMO/IAEA/CNRS

Gif sur Yvette, France, 3- 5 June 2003



CENTRE NATIONAL
DE LA RECHERCHE
SCIENTIFIQUE



WMO TD No. 1201

Thank you for your attention



IAEA

Atoms for Peace: The First Half Century

1957-2007