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Revision 0.1 (Water)
Revision 0 (Air Particulate Filters and Swipes)
Revision 0 (Phosphorous-32 in Water)
Revision 0 (Soil)
Revision 0 (Plutonium in Radioisotope Thermoelectric Generators)
Revision 0 (Concrete Building Materials)

Rapid Radiochemical Methods Applicable to Selected Radionuclides for Environmental Remediation Following Radiological Incidents

Water, Air Particulate Filters and Swipes, Soil, Building Materials, and Radioisotope Thermoelectric Generator (RTG) Material

Third Edition
(Online)

U.S. Environmental Protection Agency

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Office of Radiation and Indoor Air
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Methods Included in the Third Edition

Rapid Radiochemical Method for Americium-241 in Water for Environmental Remediation Following Radiological Incidents, Revision 0.1, 11-17-2011

Rapid Radiochemical Method for Plutonium-238 and Plutonium-239/240 in Water for Environmental Remediation Following Radiological Incidents, Revision 0.1, 11-17-2011

Rapid Radiochemical Method for Radium-226 in Water for Environmental Remediation Following Radiological Incidents, Revision 0.1, 11-17-2011

Rapid Radiochemical Method for Total Radiostrontium (Sr-90) in Water for Environmental Remediation Following Radiological Incidents, Revision 0.1, 11-17-2011

Rapid Radiochemical Method for Isotopic Uranium in Water for Environmental Remediation Following Radiological Incidents, Revision 0.1, 11-17-2011

Rapid Radiochemical Method for Phosphorus-32 in Water for Environmental Remediation Following Radiological Incidents, Revision 0, 11-17-2011

Rapid Method for Acid Digestion of Glass-Fiber and Organic/Polymeric Composition Filters and Swipes Prior to Isotopic Uranium, Plutonium, Americium, Strontium, and Radium Analyses for Environmental Remediation Following Radiological Incidents, Revision 0, 10-22-2012

Rapid Method for Sodium Carbonate Fusion of Glass-Fiber and Organic/Polymeric Composition Filters and Swipes Prior to Isotopic Uranium, Plutonium, Americium, Strontium, and Radium Analyses for Environmental Remediation Following Radiological Incidents, Revision 0, 10-22-2012

Rapid Method for Fusion of Soil and Soil-Related Matrices Prior to Americium, Plutonium, and Uranium Analyses for Environmental Remediation Following Radiological Incidents, Revision 0, 08-31-2012

Rapid Method for Radium in Soil Incorporating the Fusion of Soil and Soil-Related Matrices with the Radioanalytical Counting Method for Environmental Remediation Following Radiological Incidents, Revision 0, 08-31-2012

Rapid Method for Sodium Carbonate Fusion of Soil and Soil-Related Matrices Prior to Strontium-90 Analyses for Environmental Remediation Following Radiological Incidents, Revision 0, 08-31-2012

Rapid Method for Sodium Hydroxide/Sodium Peroxide Fusion of Radioisotope Thermoelectric Generator Materials in Water and Air Filter Matrices Prior to Plutonium Analyses for Environmental Remediation Following Radiological Incidents, Revision 0, 04-16-2014

Rapid Method for Sodium Hydroxide Fusion of Concrete and Brick Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses for Environmental Remediation Following Radiological Incidents, Revision 0, 04-16-2014

Rapid Radiochemical Method for Am-241 in Building Materials for Environmental Remediation Following Radiological Incidents, Revision 0, 04-16-2014

Rapid Radiochemical Method for Plutonium-238 and Plutonium-239/240 in Building Materials for Environmental Remediation Following Radiological Incidents, Revision 0, 04-16-2014

Rapid Radiochemical Method for Radium-226 in Building Materials for Environmental Remediation Following Radiological Incidents, Revision 0, 04-16-2014

Rapid Radiochemical Method for Total Radiostrontium (Sr-90) in Building Materials for Environmental Remediation Following Radiological Incidents, Revision 0, 04-16-2014

Rapid Radiochemical Method for Isotopic Uranium in Building Materials for Environmental Remediation Following Radiological Incidents, Revision 0, 04-16-2014

This report was prepared for the National Analytical Radiation Environmental Laboratory of the Office of Radiation and Indoor Air and the National Homeland Security Research Center of the Office of Research and Development, United States Environmental Protection Agency. It was prepared by Environmental Management Support, Inc., of Silver Spring, Maryland, under contract EP-W-07-037, work assignments B-41, I-41, and 2-43, managed by David Garman and Dan Askren, and contract EP-W-13-016, task order 014, managed by Dan Askren. This document has been reviewed in accordance with U.S. Environmental Protection Agency (EPA) policy and approved for publication. Note that approval does not signify that the contents necessarily reflect the views of the Agency. Mention of trade names, products, or services does not convey EPA approval, endorsement, or recommendation.

Preface to the Third Edition

This compendium provides rapid radioanalytical methods for selected radionuclides applicable to the intermediate and recovery phases of a nuclear or radiological incident requiring integrated laboratory response. These new methods were developed to expedite the analytical turnaround time while providing quantitative results that meet measurement quality objectives. It should be noted that these methods were not developed for compliance monitoring of drinking water samples, and they should not be construed to have EPA approval for that or for any other regulatory program.

Second Edition

The revisions in the Second Edition (July 2013) clarified and included editorial corrections of the rapid methods for amercium-241, plutonium-238 and plutonium-239/240, isotopic uranium, radiostrontium (strontium-90), and radium-226 in water initially published in 2010 (EPA 402-R-10-001). The changes do not impact the single-laboratory validations that were performed in accordance with the guidance in *Method Validation Guide for Qualifying Methods Used by Radiological Laboratories Participating in Incident Response Activities* and Chapter 6 of *Multi-Agency Radiological Laboratory Analytical Protocols Manual*. The Second Edition also included a new rapid method for the separation and determination of phosphorous-32 in water and new methods for the carbonate fusion and analysis of americium-241, plutonium-238/239, radium-226, strontium-90, and isotopic uranium in soil matrices — all of which also were single-laboratory validated in accordance with the guidance cited above.

The Second Edition also added rapid methods for acid digestion and carbonate fusion of air particulate filters and swipes. The five water methods above are used for chemical separation and analysis of glass-fiber and organic/polymeric air filters, and swipes after the samples have been solubilized using the "Method for Rapid Acid Digestion of Air Particulate Filters and Swipes Prior to Isotopic Uranium, Plutonium, Americium, Strontium, and Radium Analyses" or the alternate technique, "Method for Rapid Sodium Carbonate Fusion of Air Particulate Filters and Swipes Prior to Isotopic Uranium, Plutonium, Americium, Strontium, and Radium Analyses." The fusion method should be chosen particularly when refractory constituents are suspected in the sampled particulates or when the acidic digestion procedure is otherwise deemed to be ineffective, as may be the case with certain organic/polymeric filters, or with adhesive-backed swipes. Following the appropriate sample dissolution, these methods direct the laboratory to proceed with the appropriate step in the radionuclide-specific rapid method for water.

Third Edition

This Third Edition includes new rapid methods for sodium hydroxide/sodium peroxide fusion of radioisotope thermoelectric generator material in water and air filter matrices; and new methods for sodium hydroxide fusion of concrete and brick matrices together with specific chemical separation and analysis methods for americium-241, plutonium-238 and -239/240, radium-226, total radiostrontium, and isotopic uranium in concrete building materials. These too were single-laboratory validated in accordance with the guidance cited above.

The methods in this compendium all are capable of achieving a required relative method uncertainty of 13% at or above the analytical action level (AAL) stated in the method. The analytical action levels are based on risk or dose values that could typically be encountered during the intermediate and recovery phases of an incident. The methods have been tested to determine the time needed to analyze a batch of samples. Depending on the method and matrix, results for a batch of samples can be provided within six to eleven hours of the initiation of chemical separations (except for radium-226 which incorporates a 24-hour ingrowth step and thus requires about 38 hours). Dissolution of a batch of solid samples (i.e., digestion or fusion) requires from two to five hours depending on the technique and the matrix involved. These processing times compare with the days to weeks required by many previous methods.

EPA expects that additional methods and any subsequent revisions will be published online, and no paper reports will be distributed. Please continue to visit www.epa.gov/narel/rapid_methods.html for the most current versions of all rapid methods.

Prior to use at the laboratory, these methods must be validated according to the laboratory's quality manual and the procedures described in Chapter 6 of MARLAP (2004). The table below, adapted from MARLAP Table 6.1, summarizes the tiered approach to method validation for required method uncertainty. The method validation process is described in detail in the *Method Validation Guide for Qualifying Methods Used by Radiological Laboratories Participating in Incident Response Activities* (EPA 402-R-09-006, June 2009)

Validation Level	Application	Acceptance Criteria
A Without Additional Validation	Existing Validated Method	Method Previously Validated (By One of the Validation Levels B through E)
B	Same or Similar Matrix	Three samples at each of three levels (½ AAL, AAL and 3×AAL) Measured Value Within $\pm 2.8u_{MR}$ or $\pm 2.8\varphi_{MR}$ of Known Value
C	Similar Matrix/New Application	Five samples at each of three levels (½ AAL, AAL and 3×AAL) Measured Value Within $\pm 2.9\ u_{MR}$ or $\pm 2.9\varphi_{MR}$ of Known Value
D	Newly Developed or Adapted Method	Seven samples at each of three levels (½ AAL, AAL and 3×AAL) Measured Value Within $\pm 3.0\ u_{MR}$ or $\pm 3.0\varphi_{MR}$ of Known Value
E	Newly Developed or Adapted Method	Seven samples at each of three levels (½ AAL, AAL and 3×AAL) Measured Value Within $\pm 3.0\ u_{MR}$ or $\pm 3.0\varphi_{MR}$ of Known Value

The need to ensure adequate laboratory infrastructure to support response and recovery actions following a major radiological incident has been recognized by a number of federal agencies. The Integrated Consortium of Laboratory Networks (ICLN), created in 2005 by 10 federal agencies,[1] consists of existing laboratory networks across the federal government. The ICLN is designed to provide a national infrastructure with a coordinated and operational system of laboratory networks that provide timely, high-quality, and interpretable results for early detection and effective consequence management of acts of terrorism and other events requiring an integrated laboratory response. It also designates responsible federal agencies (RFAs) to provide laboratory support across response phases for chemical, biological, and radiological agents. To

[1] Departments of Agriculture, Commerce, Defense, Energy, Health and Human Services, Homeland Security, Interior, Justice, and State, and the U.S. Environmental Protection Agency.

meet its RFA responsibilities for environmental samples, EPA has established the Environmental Response Laboratory Network (ERLN) to address chemical, biological, and radiological threats. For radiological agents, EPA is the RFA for monitoring, surveillance, and remediation, and will share responsibility for overall incident response with the U.S. Department of Energy (DOE). As part of the ERLN, EPA's Office of Radiation and Indoor Air is leading an initiative to ensure that sufficient environmental radioanalytical capability and competency exist across a core set of laboratories to carry out EPA's designated RFA responsibilities.

EPA's responsibilities, as outlined in the *National Response Framework*, include response and recovery actions to detect and identify radioactive substances and to coordinate federal radiological monitoring and assessment activities. This document was developed to provide guidance to those radioanalytical laboratories that will support EPA's response and recovery actions following a radiological or nuclear incident of national significance.

As with any technical endeavor, actual radioanalytical projects may require particular methods or techniques to meet specific measurement quality objectives. Sampling and analysis following a radiological or nuclear incident will present new challenges in terms of types of matrices, sample representativeness, and homogeneity not experienced with routine samples. A major factor in establishing measurement quality objectives is to determine and maintain control the uncertainties associated with each aspect of the analytical process.

These methods supplement guidance in a planned series designed to present radioanalytical laboratory personnel, Incident Commanders (and their designees), and other field response personnel with key laboratory operational considerations and likely radioanalytical requirements, decision paths, and default data quality and measurement quality objectives for samples taken after a radiological or nuclear incident, including incidents caused by a terrorist attack. Additional rapid methods currently under development include:

- Rapid Radiochemical Method for Am-241 in Brick Matrices for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Plutonium-238 and Plutonium-239/240 in Brick Matrices for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Radium-226 in Brick Matrices for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Total Radiostrontium (Sr-90) In Brick Matrices for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Isotopic Uranium in Brick Matrices for Environmental Remediation Following Radiological Incidents
- Rapid Method for Sodium Hydroxide Fusion of Asphalt/Bitumen Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Am-241 in Asphalt/Bitumen Samples for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Plutonium-238 and Plutonium-239/240 in Asphalt/Bitumen Samples for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Radium-226 in Brick Matrices for Environmental Remediation Following Radiological Incidents

- Rapid Radiochemical Method for Total Radiostrontium (Sr-90) In Asphalt/Bitumen Samples for Environmental Remediation Following Radiological Incidents
- Rapid Radiochemical Method for Isotopic Uranium in Asphalt/Bitumen Samples for Environmental Remediation Following Radiological Incidents
- Rapid Method for Sodium Hydroxide Fusion of Stucco Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses for Environmental Remediation Following Radiological Incidents
- Rapid Method for Sodium Hydroxide Fusion of Limestone Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses for Environmental Remediation Following Radiological Incidents
- Rapid Method for Sodium Hydroxide Fusion of Asphalt Roofing Material Matrices Prior to Americium, Plutonium, Strontium, Radium, and Uranium Analyses for Environmental Remediation Following Radiological Incidents
- Analysis of Gross Alpha and Gross Beta Activity in Flowback and Produced Water from Hydraulic Fracturing Operations

Other related guidance documents include:

- *Radiological Laboratory Sample Analysis Guide for Incidents of National Significance – Radionuclides in Water* (EPA 402-R-07-007, January 2008)
- *Radiological Laboratory Sample Analysis Guide for Incidents of National Significance – Radionuclides in Air* (EPA 402-R-09-007, June 2009)
- *Radiological Laboratory Sample Screening Analysis Guide for Incidents of National Significance* (EPA 402-R-09-008, June 2009)
- *Method Validation Guide for Qualifying Methods Used by Radiological Laboratories Participating in Incident Response Activities* (EPA 402-R-09-006, June 2009)
- *Guide for Laboratories – Identification, Preparation, and Implementation of Core Operations for Radiological or Nuclear Incident Response* (EPA 402-R-10-002, June 2010)
- *A Performance-Based Approach to the Use of Swipe Samples in Response to a Radiological or Nuclear Incident* (EPA 600/R-11/122, October 2011)
- *Guide for Radiological Laboratories for the Control of Radioactive Contamination and Radiation Exposure* (EPA 402-R-12-005, August 2012)
- *Radiological Laboratory Sample Analysis Guide for Radiological or Nuclear Incidents – Radionuclides in Soil* (EPA 402-R-12-006, September 2012)
- *Uses of Field and Laboratory Measurements During a Radiological or Nuclear Incident* (EPA 402-R-12-007, August 2012)

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Acronyms, Abbreviations, Units, and Symbols

AAL ....analytical action level
ACS................American Chemical Society
ADL ....analytical decision level
APS ....analytical protocol specification
ATP................adenosine triphosphate
Bq...................becquerel
CL_{NA} ....critical net activity
CL_{NC}...............critical net concentration
CRM...............certified reference material (see also SRM)
CSU................combined standard uncertainty
Ci....................curie
d......................day
DL ....discrimination level
DRP................discrete radioactive particle
dpm ....disintegrations per minute
dps..................disintegrations per second
DW.................demineralized water
EPA................U.S. Environmental Protection Agency
FOM...............figure of merit
ft ....foot
FPWHFO ....Flowback and Produced Water from Hydraulic Fracturing Operations
FWHM...........full width at half maximum
g......................gram
gal...................gallon
GPC................gas-flow proportional counter
Gy...................gray
h......................hour
ICP-AES ....inductively coupled plasma – atomic emission spectrometry
ICP-MS ....inductively coupled plasma – mass spectrometry
ID ....[identifier] [identification number]
IND ....improvised nuclear device
INS ....incident of national significance
ISO ....International Organization for Standardization
IUPAC............International Union of Pure and Applied Chemistry
k......................coverage factor
kg....................kilogram (10^3 gram)
L ....liter
LCS ....laboratory control sample
LSC ....liquid scintillation counting [counter]
m ....meter
M....................molar
MARLAP.......*Multi-Agency Radiological Laboratory Analytical Protocols Manual*
MCL...............Maximum Contaminant Level
MDA ....minimum detectable activity
MDC ....minimum detectable concentration

MeV ....mega electron volts (10^6 electron volts)
min ....minute
MQO ....measurement quality objective
MV ....method validation
MVRM...........method validation reference material
μCi..................microcurie (10^{-6} curie)
NAREL ....EPA's National Analytical Radiation Environmental Laboratory, Montgomery, AL
NCRP ....National Council on Radiation Protection and Measurements
NHSRC ....EPA's National Homeland Security Research Center, Cincinnati, OH
NIST...............National Institute of Standards and Technology
ORD ...............U.S. EPA Office of Research and Development
ORIA.............U.S. EPA Office of Radiation and Indoor Air
φ_{MR}..........................required relative method uncertainty
pCi..................picocurie (10^{-12} curie)
PMT ....photomultiplier tube
PT...................proficiency test or performance test
QA..................quality assurance
QAPP ....quality assurance project plan
QC ....quality control
RDD ....radiological dispersal device
rem ....roentgen equivalent: man
ROI.................region of interest
RTG................radioisotope thermoelectric generator
s ....second
SI ....International System of Units
SRM ....standard reference material
STS.................sample test source
Sv ....sievert
TEVA.............tetravalent actinide resin
TRU................tansuranic resin
u_{MR} ....required method uncertainty
UTEVA..........uranium tetravalent resin
WCS...............working calibration source
y......................year

Radiometric and General Unit Conversions

To Convert	To	Multiply by	To Convert	To	Multiply by
years (y)	seconds (s) minutes (min) hours (h) days (d)	3.16×10^{7} 5.26×10^{5} 8.77×10^{3} 3.65×10^{2}	s min h d	y	3.17×10^{-8} 1.90×10^{-6} 1.14×10^{-4} 2.74×10^{-3}
disintegrations per second (dps)	becquerels (Bq)	1	Bq	dps	1
Bq Bq/kg Bq/m^3 Bq/m^3	picocuries (pCi) pCi/g pCi/L Bq/L	27.0 2.70×10^{-2} 2.70×10^{-2} 10^{-3}	pCi pCi/g pCi/L Bq/L	Bq Bq/kg Bq/m^3 Bq/m^3	3.70×10^{-2} 37.0 37.0 10^{3}
microcuries per milliliter (µCi/mL)	pCi/L	10^{9}	pCi/L	µCi/mL	10^{-9}
disintegrations per minute (dpm)	µCi pCi	4.50×10^{-7} 4.50×10^{-1}	pCi µCi	dpm	2.22 2.22×10^{6}
cubic feet (ft^3)	cubic meters (m^3)	2.83×10^{-2}	m^3	ft^3	35.3
gallons (gal)	liters (L)	3.78	L	gal	0.264
gray (Gy)	rad	10^{2}	rad	Gy	10^{-2}
roentgen equivalent man (rem)	sievert (Sv)	10^{-2}	Sv	rem	10^{2}

NOTE: Traditional units are used throughout this document instead of the International System of Units (SI). Conversion to SI units will be aided by the unit conversions in this table.

Acknowledgments

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Dedication

This publication is dedicated to the memory of our friend and colleague, David Garman. Dave administered nearly three dozen separate contracted radiochemistry projects for EPA dating back nearly 17 years, beginning with the *Multi-Agency Radiological Laboratory Analytical Protocols* (MARLAP) in 1994. Dave put up with countless changes of prime contractors, priorities, subcontractors, and budgets, all with good cheer, diligence, and all while keeping up with his "day job" as counting room lead for alpha-spectrometry analysis at NAREL.

Dave started with EPA's National Air and Radiation Environmental Laboratory in 1992. He left many friends throughout EPA and the radioanalytical community, and he will be greatly missed.