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Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)

Part I: Chapters 1 – 9
Appendices A – E
(Volume I)

United States Environmental Protection Agency
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United States Department of Energy
United States Department of Homeland Security
United States Nuclear Regulatory Commission
United States Food and Drug Administration
United States Geological Survey
National Institute of Standards and Technology

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ABSTRACT

The Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) manual provides guidance for the planning, implementation, and assessment of projects that require the laboratory analysis of radionuclides. MARLAP's basic goal is to provide guidance for project planners, managers, and laboratory personnel to ensure that radioanalytical laboratory data will meet a project's or program's data requirements. To attain this goal, the manual offers a framework for national consistency in the form of a performance-based approach for meeting data requirements that is scientifically rigorous and flexible enough to be applied to a diversity of projects and programs. The guidance in MARLAP is designed to help ensure the generation of radioanalytical data of known quality, appropriate for its intended use. Examples of data collection activities that MARLAP supports include site characterization, site cleanup and compliance demonstration, decommissioning of nuclear facilities, emergency response, remedial and removal actions, effluent monitoring of licensed facilities, environmental site monitoring, background studies, and waste management activities.

MARLAP is organized into two parts. Part I, intended primarily for project planners and managers, provides the basic framework of the directed planning process as it applies to projects requiring radioanalytical data for decision making. The nine chapters in Part I offer recommendations and guidance on project planning, key issues to be considered during the development of analytical protocol specifications, developing measurement quality objectives, project planning documents and their significance, obtaining laboratory services, selecting and applying analytical methods, evaluating methods and laboratories, verifying and validating radiochemical data, and assessing data quality. Part II is intended primarily for laboratory personnel. Its eleven chapters provide detailed guidance on field sampling issues that affect laboratory measurements, sample receipt and tracking, sample preparation in the laboratory, sample dissolution, chemical separation techniques, instrumentation for measuring radionuclides, data acquisition, reduction, and reporting, waste management, laboratory quality control, measurement uncertainty, and detection and quantification capability. Seven appendices provide complementary information and additional details on specific topics.

MARLAP was developed by a workgroup that included representatives from the U.S. Environmental Protection Agency (EPA), Department of Energy (DOE), Department of Defense (DOD), Department of Homeland Security (DHS), Nuclear Regulatory Commission (NRC), National Institute of Standards and Technology (NIST), U.S. Geological Survey (USGS), and Food and Drug Administration (FDA), and from the Commonwealth of Kentucky and the State of California.

FOREWORD

MARLAP is organized into two parts. Part I, consisting of Chapters 1 through 9, is intended primarily for project planners and managers. Part I introduces the directed planning process central to MARLAP and provides guidance on project planning with emphasis on radioanalytical planning issues and radioanalytical data requirements. Part II, consisting of Chapters 10 through 20, is intended primarily for laboratory personnel and provides guidance in the relevant areas of radioanalytical laboratory work. In addition, MARLAP contains seven appendices—labeled A through G—that provide complementary information, detail background information, or concepts pertinent to more than one chapter. Six chapters and one appendix are immediately followed by one or more attachments that the authors believe will provide additional or more detailed explanations of concepts discussed within the chapter. Attachments to chapters have letter designators (e.g., Attachment “6A” or “3B”), while attachments to appendices are numbered (e.g., “B1”). Thus, “Section B.1.1” refers to section 1.1 of appendix B, while “Section B1.1” refers to section 1 of attachment 1 to appendix B. Cross-references within the text are explicit in order to avoid confusion.

Because of its length, the printed version of MARLAP is bound in three volumes. Volume I (Chapters 1 through 9 and Appendices A through E) contains Part I. Because of its length, Part II is split between Volumes II and III. Volume II (Chapters 10 through 17 and Appendix F) covers most of the activities performed at radioanalytical laboratories, from field and sampling issues that affect laboratory measurements through waste management. Volume III (Chapters 18 through 20 and Appendix G) covers laboratory quality control, measurement uncertainty and detection and quantification capability. Each volume includes a table of contents, list of acronyms and abbreviations, and a complete glossary of terms.

MARLAP and its periodic revisions are available online at www.epa.gov/radiation/marlap and www.nrc.gov/reading-rm/doc-collections/nuregs/staff/sr1576/. The online version is updated periodically and may differ from the last printed version. Although references to material found on a web site bear the date the material was accessed, the material available on the date cited may subsequently be removed from the site. Printed and CD-ROM versions of MARLAP are available through the National Technical Information Service (NTIS). NTIS may be accessed online at www.ntis.gov. The NTIS Sales Desk can be reached between 8:30 a.m. and 6:00 p.m. Eastern Time, Monday through Friday at 1-800-553-6847; TDD (hearing impaired only) at 703-487-4639 between 8:30 a.m. and 5:00 p.m. Eastern Time, Monday through Friday; or fax at 703-605-6900.

MARLAP is a living document, and future editions are already under consideration. Users are urged to provide feedback on how MARLAP can be improved. While suggestions may not always be acknowledged or adopted, commentors may be assured that they will be considered carefully. Comments may be submitted electronically through a link on EPA’s MARLAP web site (www.epa.gov/radiation/marlap).

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John Griggs*, EPA, Chair

EPA: H. Benjamin Hull
Marianne Lynch*
Keith McCroan*
Eric Reynolds
Jon Richards

DOD: Andrew Scott (Army)
Ronald Swatski* (Army)
Jan Dunker (Army Corps of Engineers)
William J. Adams (Navy)
Troy Blanton (Navy)
David Farrand (Navy)
Dale Thomas (Air Force)

FDA: Edmond Baratta

DOE: Emile Boulos*
Stan Morton*
Stephanie Woolf*

DHS[†]: Carl Gogolak*
Pamela Greenlaw*
Catherine Klusek*
Colin Sanderson*

NRC: Rateb (Boby) Abu Eid
Tin Mo
George Powers

NIST: Kenneth G.W. Inn*

USGS: Ann Mullin*

* These workgroup members also served as chapter chairs.

[†] All with the Environmental Measurements Laboratory, which was part of DOE prior to the establishment of DHS on March 1, 2003.

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EPA: N. Jay Bassin (Environmental Management Support, Inc.)
U. Hans Behling (S. Cohen & Associates, Inc.)
Richard Blanchard (S. Cohen & Associates, Inc.)
Leca Buchan (Environmental Management Support, Inc.)
Jessica Burns (Environmental Management Support, Inc.)
Harry Chmelynski
Diane Dopkin (Environmental Management Support, Inc.)
Scott Hay (S. Cohen & Associates, Inc.)
Patrick Kelly (S. Cohen & Associates, Inc.)
Robert Litman
David McCurdy
Charles (Chick) Phillips (S. Cohen & Associates, Inc.)
William Richardson III (S. Cohen & Associates, Inc.)
Steven Schaffer (S. Cohen & Associates, Inc.)
Michael Schultz
Robert Shannon (Time Solutions Corp.)

DOE: Stan Blacker (MACTEC, Inc.)
Pat Harrington (MACTEC, Inc.)
David McCurdy
John Maney (Environmental Measurements Assessments)
Mike Miller (MACTEC, Inc.)
Lisa Smith (Argonne National Laboratory)

NRC: Eric W. Abelquist (ORISE)
Dale Condra (ORISE)

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John Arnold (USGS)	David Friedman (EPA)	Jim Mitchell (EPA)
David Bottrell (DOE)	Lino Fragoso (Navy)	Colleen Petullo (EPA)
Lloyd Currie (NIST)	Richard Graham (EPA)	Steve Pia (EPA)
Mike Carter (EPA)	Patricia Gowland (EPA)	Phil Reed (NRC)
Mary Clark (EPA)	Larry Jensen (EPA)	Cheryl Trottier (NRC)
Ron Colle (NIST)	K. Jack Kooyoomjian (EPA)	Mary C. Verwolf (DOE)
Mark Doehnert (EPA)	Jim Kottan (NRC)	John Warren (EPA)
Steve Domotor (DOE)	Ed Messer (EPA)	Mary L. Winston (EPA)
Joan Fisk (EPA)	Kevin Miller (DOE)	Tony Wolbarst (EPA)

EPA's Science Advisory Board (SAB) Radiation Advisory Committee's Review Subcommittee that conducted an extensive peer review of the MARLAP includes:

Chair

Dr. Janet A. Johnson, Shepherd Miller, Inc.

SAB Members

Dr. Lynn R. Anspaugh, University of Utah
Dr. Bruce B. Boecker, (Scientist Emeritus), Lovelace Respiratory Research Institute
Dr. Gilles Y. Bussod, Science Network International
Dr. Thomas F. Gesell, Idaho State University
Dr. Helen Ann Grogan, Cascade Scientific, Inc.
Dr. Richard W. Hornung, University of Cincinnati
Dr. Jill Lipoti, New Jersey Department of Environmental Protection
Dr. Genevieve S. Roessler, Radiation Consultant

SAB Consultants

Dr. Vicki M. Bier, University of Wisconsin
Dr. Stephen L. Brown, R2C2 (Risks of Radiation and Chemical Compounds)
Dr. Michael E. Ginevan, M.E. Ginevan & Associates
Dr. Shawki Ibrahim, Colorado State University
Dr. Bernd Kahn, Georgia Institute of Technology
Dr. June Fabryka-Martin, Los Alamos National Laboratory
Dr. Bobby R. Scott, Lovelace Respiratory Research Institute

Science Advisory Board Staff

Dr. K. Jack Kooyoomjian, Designated Federal Officer, EPA
Ms. Mary L. Winston, Management Assistant, EPA

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ACRONYMS AND ABBREVIATIONS

AC	alternating current
ADC	analog to digital convertor
AEA	Atomic Energy Act
AL	action level
AMS	accelerator mass spectrometry
ANSI	American National Standards Institute
AOAC	Association of Official Analytical Chemists
APHA	American Public Health Association
APS	analytical protocol specification
ARAR	applicable or relevant and appropriate requirement (CERCLA/Superfund)
ASL	analytical support laboratory
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
ATD	alpha track detector
BGO	bismuth germanate [detector]
BNL	Brookhaven National Laboratory (DOE)
BOA	basic ordering agreement
CAA	Clean Air Act
CC	charcoal canisters
CEDE	committed effective dose equivalent
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (“Superfund”)
c.f.	carrier free [tracer]
cfm	cubic feet per minute
CFR	<i>Code of Federal Regulations</i>
CL	central line (of a control chart)
CMPO	[octyl(phenyl)]-N,N-diisobutylcarbonylmethylphosphine oxide
CMST	Characterization, Monitoring, and Sensor Technology Program (DOE)
CO	contracting officer
COC	chain of custody
COR	contracting officer’s representative
cpm	counts per minute
cps	counts per second
CRM	(1) continuous radon monitor; (2) certified reference material
CSU	combined standard uncertainty
CV	coefficient of variation
CWA	Clean Water Act
CWLM	continuous working level monitor

Acronyms and Abbreviations

d	day[s]
D	homogeneous distribution coefficient
DAAP	diamylamylphosphonate
DC	direct current
DCGL	derived concentration guideline level
DHS	U.S. Department of Homeland Security
DIN	di-isopropylnaphthalene
DL	discrimination limit
DoD	U.S. Department of Defense
DOE	U.S. Department of Energy
DOELAP	DOE Laboratory Accreditation Program
DOT	U.S. Department of Transportation
DOP	dispersed oil particulate
dpm	disintegrations per minute
DPPP	dipentylpentylphosphonate
DQA	data quality assessment
DQI	data quality indicator
DQO	data quality objective
DTPA	diethylene triamine pentaacetic acid
DVB	divinylbenzene
E_e	emission probability per decay event
$E_{\beta\max}$	maximum beta-particle energy
EDD	electronic data deliverable
EDTA	ethylene diamine tetraacetic acid
EGTA	ethyleneglycol bis(2-aminoethylether)-tetraacetate
EMEDD	environmental management electronic data deliverable (DOE)
EPA	U.S. Environmental Protection Agency
ERPRIMS	Environmental Resources Program Management System (U.S. Air Force)
ESC	expedited site characterization; expedited site conversion
eV	electron volts
FAR	<i>Federal Acquisition Regulations</i> , CFR Title 48
FBO	<i>Federal Business Opportunities</i> [formerly <i>Commerce Business Daily</i>]
FDA	U.S. Food and Drug Administration
FEP	full energy peak
fg	femtogram
FOM	figure of merit
FWHM	full width of a peak at half maximum
FWTM	full width of a peak at tenth maximum

GC	gas chromatography
GLPC	gas-liquid phase chromatography
GM	Geiger-Mueller [detector]
GP	gas proportional [counter]
GUM	<i>Guide to the Expression of Uncertainty in Measurement</i> (ISO)
Gy	gray[s]
h	hour[s]
H ₀	null hypothesis
H _A , H ₁	alternative hypothesis
HDBP	dibutylphosphoric acid
HDEHP	bis(2-ethylhexyl) phosphoric acid
HDPE	high-density polyethylene
HLW	high-level [radioactive] waste
HPGe	high-purity germanium
HPLC	high-pressure liquid chromatography; high-performance liquid chromatography
HTRW	hazardous, toxic, and radioactive waste
IAEA	International Atomic Energy Agency
ICRU	International Commission on Radiation Units and Measurements
ICP-MS	inductively coupled plasma-mass spectroscopy
IPPD	integrated product and process development
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
<i>k</i>	coverage factor
keV	kilo electron volts
KPA	kinetic phosphorimeter analysis
LAN	local area network
LANL	Los Alamos National Laboratory (DOE)
LBGR	lower bound of the gray region
LCL	lower control limit
LCS	laboratory control samples
LDPE	low-density polyethylene
LEGe	low-energy germanium
LIMS	laboratory information management system
LLD	lower limit of detection
LLNL	Lawrence Livermore National Laboratory (DOE)
LLRW	low-level radioactive waste
LLRWPA	Low Level Radioactive Waste Policy Act

Acronyms and Abbreviations

LOMI	low oxidation-state transition-metal ion
LPC	liquid-partition chromatography; liquid-phase chromatography
LS	liquid scintillation
LSC	liquid scintillation counter
LWL	lower warning limit
MAPEP	Mixed Analyte Performance Evaluation Program (DOE)
MARSSIM	<i>Multi-Agency Radiation Survey and Site Investigation Manual</i>
MCA	multichannel analyzer
MCL	maximum contaminant limit
MDA	minimum detectable amount; minimum detectable activity
MDC	minimum detectable concentration
MDL	method detection limit
MeV	mega electron volts
MIBK	methyl isobutyl ketone
min	minute[s]
MPa	megapascals
MQC	minimum quantifiable concentration
MQO	measurement quality objective
MS	matrix spike; mass spectrometer
MSD	matrix spike duplicate
MVRM	method validation reference material
NAA	neutron activation analysis
NaI(Tl)	thallium-activated sodium iodide [detector]
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
NCRP	National Council on Radiation Protection and Measurement
NELAC	National Environmental Laboratory Accreditation Conference
NESHAP	National Emission Standards for Hazardous Air Pollutants (EPA)
NIM	nuclear instrumentation module
NIST	National Institute of Standards and Technology
NPL	National Physics Laboratory (United Kingdom); National Priorities List (United States)
NRC	U.S. Nuclear Regulatory Commission
NRIP	NIST Radiochemistry Intercomparison Program
NTA (NTTA)	nitrotriacetate
NTU	nephelometric turbidity units
NVLAP	National Voluntary Laboratory Accreditation Program (NIST)
OA	observational approach
OFHC	oxygen-free high-conductivity

OFPP	Office of Federal Procurement Policy
Φ_{MR}	required relative method uncertainty
Pa	pascals
PARCC	precision, accuracy, representativeness, completeness, and comparability
PBBO	2-(4'-biphenyl) 6-phenylbenzoxazole
PCB	polychlorinated biphenyl
pCi	picocurie
pdf	probability density function
PE	performance evaluation
PERALS	Photon Electron Rejecting Alpha Liquid Scintillation [®]
PFA	perfluoroalcoholoxil [™]
PIC	pressurized ionization chamber
PIPS	planar implanted passivated silicon [detector]
PM	project manager
PMT	photomultiplier tube
PT	performance testing
PTB	Physikalisch-Technische bundesanstalt (Germany)
PTFE	polytetrafluoroethylene
PUREX	plutonium uranium reduction extraction
PVC	polyvinyl chloride
QA	quality assurance
QAP	Quality Assessment Program (DOE)
QAPP	quality assurance project plan
QC	quality control
rad	radiation absorbed dose
RCRA	Resource Conservation and Recovery Act
REE	rare earth elements
REGe	reverse-electrode germanium
rem	roentgen equivalent: man
RFP	request for proposals
RFQ	request for quotations
RI/FS	remedial investigation/feasibility study
RMDC	required minimum detectable concentration
ROI	region of interest
RPD	relative percent difference
RPM	remedial project manager
RSD	relative standard deviation
RSO	radiation safety officer

Acronyms and Abbreviations

s	second[s]
SA	spike activity
S _c	critical value
SAFER	Streamlined Approach for Environmental Restoration Program (DOE)
SAM	site assessment manager
SAP	sampling and analysis plan
SEDD	staged electronic data deliverable
SI	international system of units
SMO	sample management office[r]
SOP	standard operating procedure
SOW	statement of work
SQC	statistical quality control
SPE	solid-phase extraction
SR	unspiked sample result
SRM	standard reference material
SSB	silicon surface barrier [alpha detector]
SSR	spiked sample result
Sv	sievert[s]
t _½	half-life
TAT	turnaround time
TBP	tributylphosphate
TC	to contain
TCLP	toxicity characteristic leaching procedure
TD	to deliver
TEC	technical evaluation committee
TEDE	total effective dose equivalent
TEC	technical evaluation committee (USGS)
TES	technical evaluation sheet (USGS)
TFM	tetrafluorometoxil™
TIMS	thermal ionization mass spectrometry
TIOA	triisooctylamine
TLD	thermoluminescent dosimeter
TnOA	tri-n-octylamine
TOPO	trioctylphosphinic oxide
TPO	technical project officer
TPP	technical project planning
TPU	total propagated uncertainty
TQM	Total Quality Management
TRUEX	trans-uranium extraction
TSCA	Toxic Substances Control Act

TSDf	treatment, storage, or disposal facility
tSIE	transformed spectral index of the external standard
TTA	thenoyltrifluoroacetone
<i>U</i>	expanded uncertainty
u_{MR}	required absolute method uncertainty
$u_c(y)$	combined standard uncertainty
UBGR	upper bound of the gray region
UCL	upper control limit
USACE	United States Army Corps of Engineers
USGS	United States Geological Survey
UV	ultraviolet
UWL	upper warning limit
V	volt[s]
WCP	waste certification plan
XML	extensible mark-up language
XtGe [®]	extended-range germanium
y	year[s]
Y	response variable
ZnS(Ag)	silver-activated zinc sulfide [detector]

UNIT CONVERSION FACTORS

To Convert	To	Multiply by	To Convert	To	Multiply by
Years (y)	Seconds (s)	3.16×10^7	s	y	3.17×10^{-8}
	Minutes (min)	5.26×10^5	min		1.90×10^{-6}
	Hours (h)	8.77×10^3	h		1.14×10^{-4}
Disintegrations per second (dps)	Becquerels (Bq)	1.0	Bq	dps	1.0
Bq	Picocuries (pCi)	27.03	pCi	Bq	3.7×10^{-2}
Bq/kg	pCi/g	2.7×10^{-2}	pCi/g	Bq/kg	37
Bq/m ³	pCi/L	2.7×10^{-2}	pCi/L	Bq/m ³	37
Bq/m ³	Bq/L	10^3	Bq/L	Bq/m ³	10^{-3}
Microcuries per milliliter (μCi/mL)	pCi/L	10^9	pCi/L	μCi/mL	10^{-9}
Disintegrations per minute (dpm)	μCi	4.5×10^{-7}	pCi	dpm	2.22
	pCi	4.5×10^{-1}			
Gallons (gal)	Liters (L)	3.78	Liters	Gallons	0.265
Gray (Gy)	rad	100	rad	Gy	10^{-2}
Roentgen Equivalent Man (rem)	Sievert (Sv)	10^{-2}	Sv	rem	10^2