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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
United States Department of Defense
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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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 . . . . . *Code of Federal Regulations*
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

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 . . . . . *Federal Acquisition Regulations*, CFR Title 48
FBO . . . . . *Federal Business Opportunities* [formerly *Commerce Business Daily*]
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 . . . . . *Guide to the Expression of Uncertainty in Measurement* (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

k . . . . . 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

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 . . . *Multi-Agency Radiation Survey and Site Investigation Manual*
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) . nitrilotriacetate
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'-biphenylyl) 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

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 . . . . . transfomed spectral index of the external standard
TTA . . . . . thenoyltrifluoroacetone

U . . . . . 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) Minutes (min) Hours (h)	3.16×10^{7} 5.26×10^{5} 8.77×10^{3}	s min h	y	3.17×10^{-8} 1.90×10^{-6} 1.14×10^{-4}
Disintegrations per second (dps)	Becquerels (Bq)	1.0	Bq	dps	1.0
Bq Bq/kg Bq/m^3 Bq/m^3	Picocuries (pCi) pCi/g pCi/L Bq/L	27.03 2.7×10^{-2} 2.7×10^{-2} 10^{3}	pCi pCi/g pCi/L Bq/L	Bq Bq/kg Bq/m^3 Bq/m^3	3.7×10^{-2} 37 37 10^{-3}
Microcuries per milliliter (µCi/mL)	pCi/L	10^{9}	pCi/L	µCi/mL	10^{-9}
Disintegrations per minute (dpm)	µCi pCi	4.5×10^{-7} 4.5×10^{-1}	pCi	dpm	2.22
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}