

FEDERAL REPUBLIC OF NIGERIA

**STANDARD OPERATING PROCEDURE MANUAL
FOR LABORATORIES INVOLVED IN
CHEMICALS AND WASTE ANALYSIS IN NIGERIA**

FIRST EDITION

2026

ISSUED BY

**NATIONAL ENVIRONMENTAL STANDARDS AND
REGULATIONS ENFORCEMENT AGENCY ABUJA, NIGERIA**

FOREWORD	7
PREAMBLE AND AUTHORITY	8
ABBREVIATIONS AND ACRONYMS	9
GENERAL DEFINITIONS	11
1.1 Purpose	14
1.2 Scope	14
1.4 Responsibilities	14
1.4.1 Laboratory Director	14
1.4.2 Quality Officer	14
1.4.3 Responsible Analyst	14
1.5 Document Control Procedure	15
1.5.1 SOP Structure	15
1.5.2 Numbering Convention	15
1.5.3 Issue and Distribution	15
1.5.4 SOP Review and Amendment	15
1.6 Training and Competency Management	16
1.6.1 Induction Training	16
1.6.2 Competency Assessment	16
1.7 Management Review	16
1.8 Internal Audit	16
1.9 Records Retention	16
1.10 Data Integrity and Electronic Records Management	17
2.1 Purpose	18
2.2 Scope	18
2.3 Materials Required	18
2.5 Sample Reception Procedure	18
2.5.1 Pre-Receipt Verification	18
2.5.2 Sample Condition Assessment	19
2.5.3 Sample Registration and Labelling	19
2.5.4 Chain of Custody: Laboratory Continuation	19
2.5.5 Sample Splitting and Subsampling	20
2.5.6 Sample Assignment to Analyst	20
2.6 Rejection and Return of Samples	20
2.7 Documentation and Records	20
3.1 Purpose	21
3.2 Scope	21
3.3 General Storage Principles	21
3.4 Temperature Monitoring Requirements	21

3.5 Matrix-Specific and Analyte-Specific Storage Requirements.....	22
3.5A Contingency Preservation and Power Failure Management.....	23
3.6 Sample Disposal Following Analysis.....	23
3.7 Documentation and Records.....	23
4.1 Purpose.....	25
4.2 Scope.....	25
4.3.1 Principle and Applicable Instruments.....	25
4.3.2 Principal Reference Methods.....	25
4.3.3 Sample Preparation.....	25
4.3.4 Calibration and Measurement.....	26
4.3.5 QA/QC Requirements for Metals Analysis.....	26
4.3.6 Reporting Limits and Regulatory Reference Values.....	26
4.4 Section B: Organochlorine Pesticides and PCBs.....	27
4.4.1 Principle and Applicable Instruments.....	27
4.4.2 Principal Reference Methods.....	27
4.4.3 Sample Extraction and Cleanup.....	27
4.4.4 GC-ECD Analysis.....	27
4.4.5 QA/QC Requirements for OCP/PCB Analysis.....	27
4.5 Section C: Organophosphate and Carbamate Pesticides.....	28
4.5.1 Principal Reference Methods.....	28
4.6 Section D: Polycyclic Aromatic Hydrocarbons (PAHs).....	28
4.7 Section E: Volatile Organic Compounds (VOCs).....	28
4.8 Section F: Mercury (Total and Speciation).....	28
4.9 Section G: Petroleum Hydrocarbons.....	29
4.10 Section H: Physicochemical Parameters (Water and Waste).....	29
4.11 Section I: Electronic Waste (WEEE) Analysis.....	30
4.11.1 Analytes of Concern in WEEE.....	30
4.11.2 Sample Preparation.....	30
4.11.3 Brominated Flame Retardants (PBBs and PBDEs).....	30
4.12 Section J: Plastic Waste Analysis.....	30
4.12.1 Polymer Identification by ATR-FTIR.....	30
4.12.2 Heavy Metal Additives in Plastic Waste.....	31
4.12.3 Microplastic Analysis.....	31
4.13 Section K: Battery Waste Analysis.....	31
4.13.1 Regulatory and Standards Basis.....	31
4.13.2 Battery Types and Principal Hazardous Constituents.....	31
4.13.2 Lead-Acid Battery Waste Analysis.....	32
4.13.3 Nickel-Cadmium Battery Waste Analysis.....	32
4.13.4 Lithium-Ion Battery Waste Analysis.....	32

4.14 Referral Protocol for Analyses Beyond Domestic Capability.....	32
4.15 Method Validation Requirements.....	33
4.16 Emerging Contaminants.....	33
5.1 Purpose.....	34
5.2 Scope.....	34
5.3 Equipment Records.....	34
5.3.1 Equipment Inventory.....	34
5.3.2 Equipment Files.....	34
5.4 Calibration Requirements.....	34
5.4.1 General Calibration Principles.....	34
5.4.2 System Suitability Testing (SST).....	35
5.5 Preventive Maintenance.....	36
5.6 Equipment Failure, Repair, and Return to Service.....	36
5.7 Service Contracts and Spare Parts.....	36
5.8 Documentation.....	36
5.9 Instrument Qualification.....	37
6.1 Purpose.....	38
6.2 Scope.....	38
6.3 Required QC Samples per Analytical Batch.....	38
6.4 Continuing Calibration Verification (CCV).....	39
6.5 Calculation of Performance Metrics.....	39
6.5.1 Method Detection Limit (MDL).....	39
6.5.2 Relative Percent Difference (RPD).....	40
6.5.3 Percent Recovery (%R).....	40
6.6 QC Batch Record.....	40
6.7 QC Control Charts.....	40
6.8 Proficiency Testing (PT) and Inter-Laboratory Comparisons.....	40
6.9 Reference Material Procurement and Storage.....	40
6.10 Non-Conforming Work.....	41
6.11 Measurement Uncertainty.....	41
6.12 Corrective and Preventive Action.....	41
6.13 Proficiency Testing Frequency.....	42
7.1 Purpose.....	43
7.2 Scope.....	43
7.3 Laboratory Chemical Hygiene Plan.....	43
7.4 Chemical Inventory and Safety Data Sheets.....	43
7.5 PPE Requirements by Procedure Category.....	44
7.6 Fume Hood Requirements.....	45
7.7 Eyewash and Safety Shower Requirements.....	45

7.8 Chemical Storage Requirements.....	45
7.9 Electrical Safety.....	46
7.10 First Aid Requirements.....	46
7.11 Safety Training Requirements.....	46
7.12 Occupational Exposure Monitoring.....	46
7.13 Biosafety Provisions.....	47
7.14 GHS Labelling and Chemical Hazard Communication.....	47
8.1 Purpose.....	48
8.2 Scope.....	48
8.3 Legal Obligations.....	48
8.4 Waste Classification and Segregation.....	48
8.5 Waste Storage Requirements.....	50
8.6 On-Site Treatment Options.....	51
8.6.1 Acid and Alkali Neutralisation.....	51
8.6.2 Cyanide Oxidation.....	51
8.6.3 Treatment Limitations.....	51
8.7 Licensed Hazardous Waste Disposal.....	51
8.8 Documentation.....	51
9.1 Purpose.....	52
9.2 Scope.....	52
9.3 Raw Data Recording Requirements.....	52
9.4 Result Calculation and Verification.....	52
9.5 Analytical Result Report: Standard Format (Form LF-07).....	53
9.6 Escalation Pathway for Significant Findings.....	54
9.7 Amendment and Withdrawal of Reports.....	54
9.8 Record Retention.....	54
10.1 Purpose.....	55
10.2 Scope.....	55
10.3 Emergency Contact Information.....	55
10.4 General Incident Response Principles.....	56
10.5 Chemical Spill Response Procedures.....	56
10.5.1 Minor Spill (less than 1 litre; low hazard; confined to bench or inside fume hood). 56	
10.5.2 Major Spill (more than 1 litre; or any quantity of high-hazard substance: HF, HCN, concentrated mercury compounds, PCBs, or Li-ion battery electrolyte).....	57
10.6 Fire Response.....	57
10.7 Personal Chemical Exposure Response.....	58
10.7.1 Skin Contact.....	58
10.7.2 Eye Contact.....	58
10.7.3 Inhalation of Toxic Vapour.....	58

10.8 Incident Notification and Reporting.....	58
11.1 Purpose.....	59
11.2 Scope.....	59
11.3 The National Analytical Laboratory Network.....	59
11.3.1 Tier 1: National Reference Laboratories.....	59
11.3.2 Tier 2: Regional Specialist Laboratories.....	59
11.3.3 Tier 3: Routine Analytical Laboratories.....	60
11.3.4 International Accredited Reference Facilities.....	60
11.4 Referral Procedure.....	60
11.4.1 Triggering Events for Referral.....	60
11.4.2 Referral Steps.....	60
11.4.3 International Referral: Additional Requirements.....	61
11.5 Network Quality Assurance Activities.....	61
11.5.1 National Proficiency Testing Scheme.....	61
11.5.2 Method Harmonisation Workshops.....	61
11.5.3 On-Site Technical Assistance.....	61
11.6 Memoranda of Understanding.....	61
11.7 Documentation.....	62
REFERENCES.....	63
ANNEX A Standard SOP Template.....	68
ANNEX B Laboratory Sample Receipt Register (Form LF-01).....	69
ANNEX C Chain-of-Custody Record (Form LF-02).....	70
ANNEX D Sample Storage Log (Form LF-03).....	71
ANNEX E Equipment Calibration and Maintenance Log (Form LF-04).....	73
ANNEX F Analytical QC Batch Record (Form LF-05).....	74
ANNEX G Laboratory Waste Disposal Register (Form LF-06).....	75
ANNEX H Analytical Result Report (Form LF-07).....	76
ANNEX I Laboratory Incident Report (Form LF-08).....	78
ANNEX J Inter-Laboratory Referral Form (Form LF-09).....	79
ANNEX K Approved Analytical Methods Reference Index.....	81
ANNEX L Chemical Compatibility and Segregation Chart.....	83

FOREWORD

The integrity of Nigeria's environmental regulatory system rests, at its foundation, on the reliability of scientific evidence. Every compliance decision, every finding of environmental harm or clearance of a regulated facility, ultimately depends on the quality of the analytical data generated in laboratories. It is therefore both a regulatory imperative and an institutional obligation that the laboratories serving this system operate to the highest standards of scientific rigour, consistency, and integrity.

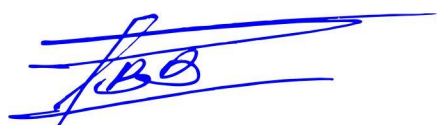
This Standard Operating Procedure Manual for Laboratories Involved in Chemicals and Waste Analysis in Nigeria is NESREA's response to a documented, evidence-based gap in the national laboratory system. The Research and Gap Analysis conducted under Activity 2.1.3 of this programme confirmed what practitioners in the field have long known: that Nigeria's analytical laboratories operate with strong individual commitment and considerable technical capacity, but without the standardized procedural framework that transforms individual competence into institutional reliability. That study identified thirteen specific gaps across seven operational domains, all of which this Manual is designed to address.

This Manual is structured around eleven core modules, each constituting a standard operating procedure for a distinct domain of laboratory operation: from the governance and documentation framework through sample reception, storage, analysis, equipment management, quality assurance, safety, waste management, data reporting, emergency response, and inter-laboratory network protocols. The Manual has been developed in deliberate alignment with the Standard Operating Procedures for Environmental Sample Collection and Management in Nigeria issued by the Federal Ministry of Environment, so that the full chain from field sample collection to laboratory analysis to result reporting is governed by compatible, cross-referenced procedural documentation. It is also aligned with the National Guidelines on Management and Disposal of Expired Chemicals (NESREA, 2026) in respect of laboratory chemical inventory management and waste disposal obligations.

The scope of this Manual has been extended beyond conventional chemicals and waste analysis to encompass the analysis of emerging issues including electronic waste, plastic waste, and battery waste, reflecting the growing analytical demands of Nigeria's regulatory programmes in these sectors.

This edition of the Manual has been produced following technical review by NESREA's subject-matter experts, partner agency representatives, and a stakeholder validation workshop held in Lagos in May 2026. The workshop brought together laboratory directors, quality officers, senior analysts, representatives of regulatory agencies, professional bodies, and international technical partners. All comments and contributions received during the review and validation exercise have been considered and integrated into this publication. The Manual is now presented for institutional adoption and national implementation.

Nigeria possesses the foundations of an excellent analytical laboratory system. What has been missing is the unifying procedural architecture. This Manual provides that architecture. Its implementation, sustained by training, adequate resourcing, and leadership commitment, will transform Nigeria's laboratory network from a collection of capable individual facilities into a coherent, accreditation-ready system that generates data worthy of the regulatory decisions it supports.



Director-General / Chief Executive Officer

National Environmental Standards and Regulations Enforcement Agency

Abuja, Nigeria | 2026

PREAMBLE AND AUTHORITY

1. This Manual is issued by the National Environmental Standards and Regulations Enforcement Agency (NESREA) under the authority of the NESREA Act, 2007 Amendment 2018 (Act No. 25 of 2007), specifically Sections 7(g), 7(f), 8(k)(vii), 8(k)(xiv), 8(m), and 34(c).
2. Section 7(g) of the NESREA Act charges the Agency with enforcing compliance with regulations on the importation, exportation, production, distribution, storage, sale, use, handling, and disposal of hazardous chemicals and waste. Reliable analytical laboratory data is indispensable to the discharge of this function.
3. Section 8(k) empowers NESREA to submit for ministerial approval proposals for guidelines, regulations, and standards on environmental matters, including waste management and hazardous substance control. This Manual constitutes such a guideline.
4. Section 30(4) of the NESREA Act provides that a sample seized by a NESREA officer may be submitted to an analyst for analysis, and that the analyst shall issue a certificate or report in prescribed form. This Manual prescribes the analytical procedures and report format for such certificates and reports.
5. This Manual is further issued in fulfilment of Nigeria's commitment to controlling the transboundary movement of hazardous wastes and their environmentally sound disposal, eliminating persistent organic pollutants from the Nigerian environment, ensuring prior informed consent procedures in the international trade of hazardous chemicals, phasing down the use and release of mercury, and advancing the objectives of the Global Framework on Chemicals 2023 to 2050.
6. This Manual shall be read in conjunction with: the Standard Operating Procedures for Environmental Sample Collection and Management in Nigeria (Federal Ministry of Environment); the National Guidelines on Management and Disposal of Expired Chemicals in Nigeria; Nigerian Industrial Standard on Environmentally Sound Management of Chemicals; and the applicable National Environmental Regulations.
7. This Manual applies to all analytical laboratories performing chemicals and waste analytical functions in support of regulatory enforcement, environmental monitoring, or compliance verification, including laboratories within relevant Ministries, Departments and Agencies, research institutes, academic institutions, and private analytical facilities.
8. This Manual shall be cited as: Standard Operating Procedure Manual for Laboratories Involved in Chemicals and Waste Analysis in Nigeria, 2026 (hereinafter "the Manual" or "the SOP Manual").

ABBREVIATIONS AND ACRONYMS

Acronym	Full Meaning
AAS	Atomic Absorption Spectrometry
ABU	Ahmadu Bello University, Zaria
ATR-FTIR	Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy
COC	Chain of Custody
CRM	Certified Reference Material
CV-AAS	Cold Vapour Atomic Absorption Spectrometry
CV-AFS	Cold Vapour Atomic Fluorescence Spectrometry
DG/CEO	Director-General / Chief Executive Officer
ECD	Electron Capture Detector
EPR	Extended Producer Responsibility
FCT	Federal Capital Territory
FID	Flame Ionization Detector
FME_{nv}	Federal Ministry of Environment
FTIR	Fourier Transform Infrared Spectroscopy
GC	Gas Chromatography
GC-MS	Gas Chromatography Mass Spectrometry
GFC	Global Framework on Chemicals 2023 to 2050
GFAAS	Graphite Furnace Atomic Absorption Spectrometry
GHS	Globally Harmonised System for Classification and Labelling of Chemicals
GLP	Good Laboratory Practice
GMP	Global Monitoring Plan
HPLC	High Performance Liquid Chromatography
HRMS	High Resolution Mass Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
IHR	International Health Regulations, 2005
IPAN	Institute of Public Analysts of Nigeria
ISO/IEC	International Organization for Standardization / International Electrotechnical Commission
LC-MS/MS	Liquid Chromatography Tandem Mass Spectrometry
LIMS	Laboratory Information Management System

LOD	Limit of Detection
LOQ	Limit of Quantification
NAFDAC	National Agency for Food and Drugs Administration and Control
NARICT	National Research Institute for Chemical Technology
NCDC	Nigeria Centre for Disease Control and Prevention
NESREA	National Environmental Standards and Regulations Enforcement Agency
NIMR	Nigerian Institute of Medical Research
NIPRD	National Institute for Pharmaceutical Research and Development
NISLT	Nigerian Institute of Science Laboratory Technology
NPD	Nitrogen-Phosphorus Detector
OHS	Occupational Health and Safety
OPCW	Organisation for the Prohibition of Chemical Weapons
PAHs	Polycyclic Aromatic Hydrocarbons
PBB	Polybrominated Biphenyls
PBDE	Polybrominated Diphenyl Ethers
PCBs	Polychlorinated Biphenyls
POPs	Persistent Organic Pollutants
PPE	Personal Protective Equipment
PT	Proficiency Testing
QA/QC	Quality Assurance / Quality Control
QMS	Quality Management System
QuEChERS	Quick, Easy, Cheap, Effective, Rugged, Safe (extraction method)
RPD	Relative Percent Difference
SDS	Safety Data Sheet
SON	Standards Organisation of Nigeria
SOP	Standard Operating Procedure
SPE	Solid Phase Extraction
UKHSA	United Kingdom Health Security Agency
UNEP	United Nations Environment Programme
UPS	Uninterruptible Power Supply
USEPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound
WEEE	Waste Electrical and Electronic Equipment

WHO	World Health Organization
XRF	X-ray Fluorescence Spectrometry

GENERAL DEFINITIONS

In this Manual, unless the context otherwise requires, the following terms bear the meanings ascribed to them below.

Term	Definition
Accreditation	Formal recognition by an authorised body that a laboratory is competent to carry out specific tests or calibrations in accordance with a stated standard, typically ISO/IEC 17025.
Analyst	A qualified person employed by a laboratory to perform chemical analyses, possessing at minimum a Bachelor of Science degree, HND, or equivalent recognized qualification in chemistry, biochemistry, environmental science, or a related discipline.
Analytical Batch	A defined set of samples analysed together under identical conditions in a single analytical sequence, including the associated quality control samples.
Battery Waste	Any spent, discarded, or end-of-life battery or accumulator arising from domestic, commercial, or industrial use, including lead-acid, nickel-cadmium, lithium-ion, and nickel-metal hydride batteries.
Blank	A sample consisting of the analytical solvent or matrix without the target analyte, processed through the entire analytical sequence to assess background contamination.
Calibration Standard	A certified solution of known concentration used to establish the relationship between instrument response and analyte concentration.
Certified Reference Material (CRM)	A reference material accompanied by documentation from an accredited body certifying the values of specified properties with associated measurement uncertainties, traceable to national or international measurement standards.
Chain of Custody (COC)	The chronological documentation of the possession, control, transfer, analysis, and final disposition of a sample, providing a verifiable and unbroken record from collection to disposal.
Chemical Waste	Waste chemicals resulting from laboratory operations, including spent solvents, acid and alkali solutions, heavy metal-containing digestion residues, extraction wastes, and expired reagents.
Compliance Sample	A sample collected by or on behalf of NESREA or a partner regulatory agency for the purpose of verifying compliance with an applicable environmental standard or limit.
Corrective Action	Action taken to eliminate the root cause of a detected non-conformity or deficiency to prevent recurrence.
Detection Limit (LOD)	The lowest concentration of an analyte that can be reliably distinguished from background noise, typically estimated using statistically validated procedures.

Duplicate	A second aliquot of the same sample, independently prepared and analysed under identical conditions, used to assess analytical precision.
Electronic Waste (WEEE)	Waste electrical and electronic equipment, comprising any electrical or electronic equipment that has been discarded, including computing devices, telecommunications equipment, consumer electronics, lighting equipment, electrical tools, and household appliances.
Holding Time	The maximum elapsed time from sample collection to analysis within which sample integrity is assured for a specified analyte in a specified matrix, under prescribed preservation and storage conditions.
Laboratory	For the purposes of this Manual, any facility performing chemical analysis of environmental, waste, food, or biological samples in support of regulatory enforcement, environmental monitoring, compliance verification, or research.
Laboratory Director	The person with overall responsibility for the management, quality, and safety of a laboratory's operations and for the technical validity of all analytical results issued by the laboratory.
Limit of Quantification (LOQ)	The lowest concentration of an analyte that can be reliably measured with a stated precision and accuracy, typically estimated using statistically validated procedures..
Matrix Spike	A known quantity of target analyte added to a portion of the sample matrix, processed through the complete analytical sequence, to assess analyte recovery in the specific sample matrix.
Method Blank	A sample consisting of the extraction or digestion solvent without sample matrix, processed through the complete analytical sequence to detect procedural contamination.
Module	A self-contained standard operating procedure within this Manual, constituting the authoritative procedural document for a specific domain of laboratory operation.
Plastic Waste	Any discarded or end-of-life material composed primarily of synthetic or semi-synthetic polymers, including plastic packaging, containers, films, fibres, and moulded articles, arising from domestic, commercial, agricultural, or industrial sources.
Preventive Action	Action taken to eliminate the cause of a potential non-conformity before it occurs.
Priority Chemical	A chemical substance subject to specific analytical monitoring requirements under Nigerian environmental legislation or under applicable international chemical and waste management obligations.
Quality Manual	The overarching document, prepared by each laboratory, describing its quality management system, its commitment to quality and impartiality, and referencing or incorporating all applicable SOPs.

Quality Officer	A designated person in the laboratory responsible for maintaining the quality management system, monitoring QA/QC performance, managing calibration and maintenance records, and coordinating proficiency testing participation.
Reference Laboratory	A laboratory formally designated by NESREA or a partner agency as a node laboratory for a specified analytical capability within the national laboratory network, to which other laboratories may refer samples for analysis.
Regulatory Analytical Certificate	A formal document issued by a designated qualified analyst under Section 30(4) of the NESREA Act, certifying the results of analysis of a sample seized by or submitted to NESREA for regulatory purposes, in the form prescribed by Module 9 of this Manual.
Responsible Analyst	The analyst who performed and is accountable for the analytical result reported for a specific sample.
Safety Data Sheet (SDS)	A document providing information on the physical, chemical, health, and environmental hazards of a chemical substance or mixture, and instructions for safe handling, use, storage, and disposal, prepared in accordance with GHS requirements.
SOP Master Register	A controlled list of all current, approved SOPs in a laboratory, showing SOP number, title, version number, date of issue, date of next review, and custodian.
Standard Addition	An analytical technique in which known quantities of a standard solution are added to the sample solution to determine analyte concentration while compensating for matrix effects.
System Suitability Test	A set of analytical performance criteria verified before or during an analytical run to confirm that the analytical system is performing adequately for its intended purpose.

MODULE 1

Laboratory Governance, Documentation Control, and Quality Management System

Reference:
NESREA-SOP-MO
D01
Revision: Rev. 1.0

Date: 2026
Status: First Edition

1.1 Purpose

This Module establishes the governance framework, documentation control system, and Quality Management System (QMS) requirements for laboratories operating under this Manual. It defines the roles and responsibilities of laboratory personnel, the structure and control of all SOP documents, training and competency requirements, and the management review process that ensures the QMS continuously improves. Compliance with this Module is the prerequisite for the implementation of all other Modules.

1.2 Scope

This Module applies to all laboratories falling within the scope of this Manual, encompassing analytical facilities in relevant Ministries, Departments and Agencies, research institutes, academia, and private entities. It applies to the Laboratory Director, Quality Officer, analysts, technical officers, support staff, and any external personnel working within the laboratory.

1.4 Responsibilities

1.4.1 Laboratory Director

- Overall accountability for the QMS, including its establishment, maintenance, and continuous improvement
- Appointment of the Quality Officer
- Approval and signing of the Laboratory Quality Manual
- Conducting or directing the annual Management Review
- Ensuring adequate resources (personnel, equipment, consumables, training) for QMS implementation
- Signing the Regulatory Analytical Certificate (Module 9)

1.4.2 Quality Officer

- Maintaining the SOP Master Register and version control for all laboratory documents
- Issuing, distributing, and retrieving controlled copies of SOPs
- Monitoring QA/QC performance and preparing the monthly QC summary (Module 6)
- Coordinating proficiency testing participation (Module 6)
- Managing calibration and maintenance records (Module 5)
- Initiating corrective and preventive action processes
- Preparing the QMS annual performance report

1.4.3 Responsible Analyst

- Reading and signing acknowledgement of receipt of all SOPs relevant to assigned analytical functions
- Working in strict accordance with current approved SOPs
- Recording all analytical activities contemporaneously in approved laboratory records

- Immediately notifying the Quality Officer or Laboratory Director of any departure from an SOP
- Maintaining competency through participation in training and proficiency testing

1.5 Document Control Procedure

1.5.1 SOP Structure

Every SOP in this Manual, and every laboratory-specific SOP developed to supplement it, shall be structured in accordance with Annex A (Standard SOP Template). The minimum required sections are: Module number and title; reference number; version and date; status; purpose; scope; regulatory basis; definitions; responsibilities; materials and equipment required; procedure (numbered steps); QA/QC requirements; documentation and records; cross-references; and review and amendment history.

1.5.2 Numbering Convention

SOPs issued under this Manual shall carry reference numbers in the format NESREA-SOP-MOD[NN] where NN is the two-digit module number (01 to 11). Laboratory-specific supplementary SOPs shall carry the prefix [AGENCY-ABBREV]-SOP-[FUNCTIONAL AREA CODE]-[SEQUENTIAL NUMBER], for example NAFDAC-SOP-MET-001 for the first NAFDAC SOP on metals analysis.

1.5.3 Issue and Distribution

9. The Quality Officer shall maintain an up-to-date SOP Master Register, updated within 48 hours of any SOP issuance, revision, or withdrawal.
10. Controlled hard copies shall be stamped in red ink "CONTROLLED COPY: [Copy Number]" and distributed against a signed distribution list. Uncontrolled copies shall be stamped "UNCONTROLLED: FOR REFERENCE ONLY".
11. Superseded versions shall be immediately withdrawn from circulation. The Quality Officer shall stamp the superseded version "SUPERSEDED: [Date]" and retain one copy in the SOP archive. All other superseded copies shall be destroyed.
12. Where the laboratory operates an electronic document management system, the system shall maintain a version history log and shall prevent editing of approved SOPs without an authorised change request.

1.5.4 SOP Review and Amendment

13. Each SOP shall be reviewed not less than once every three years, or immediately upon: change in applicable legislation or regulation; change in applicable international treaty requirements; introduction of new equipment requiring amended operating procedures; identification of a systematic analytical error traceable to a procedural deficiency; recommendations from a management review, internal audit, or proficiency testing exercise; or instruction from NESREA.
14. Proposed amendments shall be submitted to the Quality Officer on a Change Request Form specifying the section to be amended, the proposed change, and the technical justification.
15. The Laboratory Director shall approve all amendments. For Modules 1 through 11 of this Manual, amendments shall additionally be approved by the Chemicals and Pharmaceutical Division, Inspection and Enforcement Department, NESREA.

1.6 Training and Competency Management

1.6.1 Induction Training

16. Every new analyst or technical officer shall, before performing unsupervised analytical work, complete an induction programme covering: laboratory layout, safety procedures, and emergency protocols (Modules 7 and 10); the QMS framework and documentation requirements (this Module); the chain-of-custody and sample reception procedure (Module 2); equipment safety and operation training relevant to their assigned instruments (Module 5); and all analytical method SOPs relevant to their assigned functions (Module 4).
17. Induction training shall be documented on a Training Record Form and signed by both the trainee and the supervising analyst.

1.6.2 Competency Assessment

18. Competency for each assigned analytical function shall be assessed before the analyst performs that function unsupervised, comprising: witnessed performance of the complete analytical procedure on a known sample (CRM or spiked blank); review of the analyst's raw data records; and comparison of the analyst's result with the certified or expected value.
19. Acceptance criterion: the analyst's result shall fall within two standard deviations of the expected value, with relative standard deviation of duplicate analyses not exceeding 15 percent.
20. If the acceptance criterion is not met, additional supervised training shall be provided and the competency assessment repeated.
21. Competency shall be reassessed after any period of absence of more than six months, after any significant equipment change, or upon implementation of a revised analytical method.

1.7 Management Review

The Laboratory Director shall conduct a formal Management Review of the QMS at least once per year. The review shall assess: QA/QC performance data (Module 6); results of proficiency testing; calibration and maintenance status (Module 5); corrective and preventive action status; results of any audits; adequacy of resources; and recommendations for improvement. The minutes of the Management Review shall be recorded and retained.

1.8 Internal Audit

Each laboratory shall conduct an internal audit of its QMS implementation at least once per year. The audit shall assess compliance with each Module of this Manual as implemented at that laboratory. Audit findings shall be recorded, significant non-conformities shall generate corrective action requests, and the audit report shall be presented to the Management Review. NESREA may conduct supervisory audits of any laboratory operating under this Manual.

1.9 Records Retention

All laboratory records required by this Manual shall be retained for a minimum of five years from the date of sample analysis or, in the case of records relating to legal proceedings, until the conclusion of all proceedings to which the record is relevant, whichever is the longer. Records shall be stored in a manner that prevents deterioration, loss, or unauthorised access.

1.10 Data Integrity and Electronic Records Management

Laboratories utilizing electronic data systems, including Laboratory Information Management Systems, instrument software, or other electronic records platforms, shall establish documented controls to ensure data integrity, traceability, and protection against unauthorized alteration throughout the analytical lifecycle.

Such controls shall include, at minimum: password-protected user access with individual user accounts; user-specific activity logs and audit trails that record all data entries, modifications, and deletions; restricted editing permissions preventing unauthorized alteration of finalized records; routine electronic data backup to a secure off-system location; protection against malware and unauthorized external access; and secure retention of electronic analytical records for the minimum retention period specified in this Manual.

Electronic records shall be considered equivalent to paper records only where integrity and traceability can be demonstrated to the satisfaction of the Laboratory Director and, where applicable, NESREA. All data integrity procedures shall be consistent with the ALCOA principles, requiring that analytical records be Attributable, Legible, Contemporaneous, Original, and Accurate. The laboratory shall document its data integrity policy and include data integrity compliance as a standing item in internal audits and Management Review.

CROSS-REFERENCE: *This Module should be read in conjunction with Module 6 (QA/QC), Module 5 (Equipment), and Annex A (Standard SOP Template). For enforcement-related analytical records, refer to Section 30(4) of the NESREA Act and Module 9 (Data Recording and Reporting).*

MODULE 2**Sample collection, Reception, Registration, and Chain of Custody**

Reference:
NESREA-SOP-MO
D02
Revision: Rev. 1.0

Date: 2026
Status: First Edition

2.1 Purpose

This Module establishes standardised procedures for the collection, reception, registration, condition assessment, and chain-of-custody management of samples arriving at the laboratory. It ensures that all samples are accurately identified, their physical condition is documented at receipt, custody is formally transferred and recorded, and every sample is traceable from receipt through analysis to final disposal.

2.2 Scope

This Module applies to all samples received by the laboratory, including compliance samples collected under Section 30(1) of the NESREA Act; monitoring samples submitted under regulatory programmes; research and surveillance samples; samples of electronic waste, plastic waste, and battery waste submitted for compositional analysis; inter-laboratory referral samples (Module 11); and samples submitted by regulated entities for self-compliance verification.

CROSS-REFERENCE: *This Module is the downstream continuation of the FME_{Env} SOPs for Environmental Sample Collection and Management. The field Chain-of-Custody form remains the primary custody document up to the point of laboratory receipt, from which point this Module governs. Together, the two instruments constitute a complete and unbroken custody record.*

2.3 Materials Required

- Laboratory Sample Receipt Register (Form LF-01, Annex B)
- Chain-of-Custody Record (Form LF-02, Annex C) for laboratory continuation
- Sample Analysis Request Form
- Pre-printed sample labels with barcode or sequential reference number
- Permanent marker and tamper-evident sample custody seals
- Inspection gloves and appropriate PPE for sample type
- Refrigerator/freezer at designated reception staging area (where required)

2.5 Sample Reception Procedure

2.5.1 Pre-Receipt Verification

22. The Laboratory Reception Officer shall verify, before accepting any sample, that: a completed Sample Analysis Request Form (or equivalent written request on official letterhead) accompanies the sample; the submitting officer's name, designation, and contact details are recorded; the sample type, collection date, collection location, and chain-of-custody number are stated; and the requested analyses are within the laboratory's current analytical capability.
23. If the above documentation is incomplete, the Reception Officer shall notify the submitting officer and request the missing information before formal acceptance. The reason for any delay shall be noted in the Receipt Register.

24. Every sample received for analysis shall be accompanied by complete documentation covering: the identity and classification of the sample; the analysis required; the applicable analytical method reference; the name and contact details of the submitting officer; and a declaration of any known or suspected hazards associated with handling, storage, or analysis of that sample. Where hazardous characteristics are undeclared or unknown, the Reception Officer shall treat the sample as potentially hazardous and apply appropriate safety precautions until the nature of the sample is established.

2.5.2 Sample Condition Assessment

25. On receipt, the Reception Officer shall examine each sample container and record the following in Form LF-01: container type and material; container condition (intact, cracked, leaking, damaged lid); headspace; label condition; and any visible unusual characteristics such as temperature, colour, odour, precipitate, phase separation, or turbidity.
26. Where a sample is received with a broken seal, cracked container, or visible contamination inconsistent with normal sample collection, this shall be recorded as a sample integrity concern in Form LF-01 and the laboratory COC continuation sheet. The submitting officer shall be notified immediately. Analysis may proceed at the Laboratory Director's discretion, but the integrity concern shall be noted on the analytical result report (Module 9).
27. No sample shall be accepted for regulatory compliance analysis if it arrives without intact custody seals, unless written authorisation is provided by the NESREA officer-in-charge.

2.5.3 Sample Registration and Labelling

28. Each accepted sample shall be assigned a unique Laboratory Reference Number (LRN) in the format [YEAR]/[LAB CODE]/[SEQUENTIAL NUMBER], for example 2026/NSR/0347. The LRN shall be assigned immediately upon acceptance and shall be the sample's sole identifier within the laboratory.
29. A secondary laboratory label bearing the LRN and sample type shall be applied to each container without removing or obscuring the original field label.
30. All entries in Form LF-01 shall be made contemporaneously in permanent ink, legibly, without correction fluid. Errors shall be crossed through with a single line, corrected, and initialled.
31. If the laboratory operates a LIMS, all Form LF-01 data shall be entered into the LIMS within two hours of sample acceptance.

2.5.4 Chain of Custody: Laboratory Continuation

32. The Laboratory COC Continuation Sheet (Form LF-02) shall document every internal transfer of custody within the laboratory: reception to sample storage; storage to analytical bench; analytical bench to co-worker if applicable; analyst to disposal. Each transfer shall be recorded with the time, name, and signature of both the person transferring and the person receiving custody.
33. Form LF-02 shall be kept with the sample at all times within the laboratory until analysis is complete.
34. The completed COC Continuation Sheet shall be retained with the analytical file as a permanent record (see Module 9 and Section 1.9).

2.5.5 Sample Splitting and Subsampling

35. Where a single submitted container is to be analysed for multiple analyte groups, the Reception Officer shall prepare sub-aliquots from the parent sample into clean, appropriately labelled containers. Sub-aliquot labels shall carry the LRN plus a sequential sub-aliquot suffix, for example 2026/NSR/0347-A and 2026/NSR/0347-B.
36. All subsampling shall be performed in an area free from contamination sources, using pre-cleaned laboratory ware. The subsampling procedure, container types, and aliquot volumes shall be recorded in Form LF-01.
37. Where a sample is insufficient in volume for all requested analyses, the Laboratory Director or designee shall decide the analytical priority and inform the submitting officer.

2.5.6 Sample Assignment to Analyst

38. The Reception Officer shall notify the relevant Responsible Analyst of sample receipt and forward the Sample Analysis Request Form, Form LF-01, and Form LF-02 to the analyst.
39. The analyst shall sign Form LF-02 to confirm acceptance of custody and shall proceed with analysis in accordance with the holding time requirements in Module 3.

2.6 Rejection and Return of Samples

Samples shall be rejected and returned to the submitting officer with a written statement of reasons if: the sample is received after the applicable holding time has elapsed; the container is damaged to the extent that sample integrity is compromised; the required information is absent and the submitting officer cannot provide it within 24 hours; or the requested analysis is outside the laboratory's capability and no referral arrangement exists.

Where a sample is found on receipt to have been incorrectly preserved, the Reception Officer shall record the nature of the preservation failure in Form LF-01, notify the submitting officer in writing, and refer the matter to the Laboratory Director for a decision on whether to proceed with analysis. Incorrect preservation includes the use of a wrong preservative type or concentration, the use of a contaminated or expired preservative, and delivery in containers incompatible with the required preservation method. Any analytical result obtained from an incorrectly preserved sample shall carry a qualification statement in the Analytical Result Report disclosing the preservation concern and the resulting limitation on the validity and interpretation of the results.

2.7 Documentation and Records

Form LF-01 (Laboratory Sample Receipt Register) and Form LF-02 (COC Continuation Sheet) are mandatory records for every sample. They shall be retained for five years minimum. The completed forms are provided in Annexes B and C respectively.

CROSS-REFERENCE: *For sample storage requirements following reception, proceed to Module 3. For samples requiring urgent regulatory compliance analysis, refer to the NESREA Act (Section 30(4)) and Module 9 for the Regulatory Analytical Certificate.*

MODULE 3**Sample Storage, Preservation, and Holding Times**

Reference:
NESREA-SOP-MO
D03
Revision: Rev. 1.0

Date: 2026
Status: First Edition

3.1 Purpose

This Module prescribes the storage conditions, preservation requirements, and maximum holding times for samples in the laboratory from receipt through to analysis and final disposal. It is designed to prevent analyte degradation, contamination, or transformation during the pre-analysis period and to ensure that results reported reflect the actual composition of the sample at the time of collection.

3.2 Scope

This Module applies to all categories of samples received by the laboratory, including water, wastewater, soil, sediment, sludge, biological tissue, food, waste material, electronic waste components, plastic waste, battery waste, air filter media, and swab extracts and other relevant samples. It governs storage from the point of laboratory receipt (Module 2) until commencement of extraction, digestion, or direct analysis.

CROSS-REFERENCE: *The preservation requirements specified in the FMEnv SOPs for Environmental Sample Collection and Management govern sample preservation from the point of collection in the field. Where a discrepancy exists between the field SOP and this Module, the more conservative requirement shall prevail.*

3.3 General Storage Principles

40. All samples shall be stored in a designated, secure sample storage area to which access is restricted to only authorised laboratory personnel. The storage area shall be clearly labelled and separated from analytical bench areas and chemical storage areas.
41. Samples shall be stored in the containers in which they were received (if suitable) or transferred into appropriate laboratory containers as specified in this Module. Any container change shall be documented in Form LF-01.
42. Samples shall be organised by LRN. A sample location log shall be maintained so that any sample can be located within five minutes.
43. Samples known or suspected to contain volatile compounds, odorous substances, or highly toxic materials such as cyanide or mercury compounds shall be stored in a separately ventilated area or in a fume cupboard with exhaust ventilation.
44. Incompatible samples shall be segregated in storage. Refer to Annex L (Chemical Compatibility and Segregation Chart).

3.4 Temperature Monitoring Requirements

45. Refrigerators used for sample storage shall maintain a temperature between 2 and 8 degrees Celsius. Freezers shall maintain temperatures at or below negative 20 degrees Celsius.
46. Temperature shall be recorded at least twice daily using calibrated thermometers or data loggers. Temperature logs shall be maintained in Form LF-03 (Sample Storage Log, Annex D).

47. Where automatic data loggers are available, alarms shall be set to activate when temperature deviates beyond plus or minus 3 degrees Celsius from the target temperature.
48. Where power supply interruptions occur, the analyst shall immediately check storage temperatures and record the interruption time and temperature deviation. If temperature has exceeded the maximum limit by more than 2 degrees Celsius for more than 30 minutes, the affected samples shall be flagged and the Laboratory Director notified.

3.5 Matrix-Specific and Analyte-Specific Storage Requirements

The table below specifies storage conditions and holding times for the priority sample and analyte combinations most frequently encountered in chemicals and waste analysis laboratories. For analyte-matrix combinations not listed, the analyst shall consult the relevant USEPA SW-846 method chapter or ISO 5667 series, and shall document the reference used.

Matrix / Analyte Group	Container	Storage Condition	Holding Time
Water: Total metals (except Hg)	HDPE or glass, 500 mL	2 to 8 °C, acidified to pH <2 with HNO ₃	6 months
Water: Total Mercury (THg)	Glass, 500 mL (no plastic)	2 to 8 °C, acidified with BrCl or HNO ₃ /K ₂ Cr ₂ O ₇	28 days
Water: OCPs / PCBs	Amber glass, 1 L, PTFE-lined cap	2 to 8 °C, no headspace	7 days to extraction; 40 days extracted
Water: PAHs	Amber glass, 1 L	2 to 8 °C, acidified to pH <2 with HCl	7 days to extraction; 40 days extracted
Water: VOCs	Glass vials, PTFE septum, no headspace	2 to 8 °C, HCl to pH <2	14 days
Soil / Sediment: Metals	HDPE or glass jar	2 to 8 °C or air-dried	6 months (digested extract)
Soil / Sediment: OCPs / PCBs	Glass jar with PTFE liner	2 to 8 °C, avoid UV light	14 days to extraction; 40 days extracted
Soil / Sediment: Mercury	Glass jar	2 to 8 °C	28 days
Waste (liquid): Metals	HDPE or glass	2 to 8 °C, acidified to pH <2 with HNO ₃	6 months
Biological tissue: Metals	Glass or HDPE, sealed	-20 °C; -80 °C for long-term	6 months at -20 °C; 2 years at -80 °C
Food: Pesticide residues	Polypropylene or glass	-20 °C	3 months
Air filter (PM/metals)	Zip-lock bag or glass vial	Room temperature, desiccated	6 months

WEEE components (metals/polymers)	Sealed plastic container or glass jar	Room temperature, desiccated	12 months
Plastic waste (polymer ID)	Sealed plastic bag	Room temperature, away from UV light	6 months
Battery waste: lead-acid electrolyte	HDPE bottle, acid-washed	2 to 8 °C, pH <2	28 days
Battery waste: electrode material	Sealed HDPE or glass jar	Room temperature, desiccated	6 months

NOTE: Where holding times specified above differ from holding times stated on the Sample Analysis Request Form or in the FMEnv field SOP, the shorter holding time shall be applied. If a sample is received with insufficient time remaining within the holding time to complete analysis, the Laboratory Director shall immediately notify the submitting officer. Analysis of out-of-holding-time samples shall only proceed with written authorisation from the Laboratory Director, and the fact shall be disclosed in the Analytical Result Report (Module 9).

3.5A Contingency Preservation and Power Failure Management

Each laboratory shall maintain a documented contingency preservation plan to protect sample integrity during periods of power interruption, equipment failure, or other conditions affecting cold storage capacity. The contingency plan shall form part of the laboratory quality management documentation and shall be reviewed annually.

The contingency plan shall address, at minimum: the availability of backup cooling capacity such as standby refrigeration units, dry ice, or ice baths sufficient to maintain samples within acceptable temperature ranges; the designation of a responsible officer for sample welfare during emergencies; a decision tree for determining whether samples can be salvaged or must be flagged and re-collected; and documented thresholds for temperature excursions beyond which samples are considered compromised and analytical results are qualified or voided.

Where a sample cannot be analyzed immediately but requires analysis within a short timeframe, it shall be stored at between 1 and 5 degrees Celsius, away from direct sunlight, and shall be submitted for analysis within two working days of receipt unless a shorter holding time applies. This short-term cold storage requirement applies regardless of whether the laboratory has suffered a power interruption or whether samples are awaiting instrument availability.

3.6 Sample Disposal Following Analysis

49. After completion of analysis and result authorisation (Module 9), samples shall be retained for a minimum of 30 calendar days. For regulatory enforcement samples, the retention period shall be 90 calendar days or until written release is received from the requesting NESREA officer, whichever is the later.
50. Sample disposal shall be documented in Form LF-03. Hazardous sample waste shall be disposed of in accordance with Module 8 (Laboratory Waste Management).

3.7 Documentation and Records

Form LF-03 (Sample Storage Log, Annex D) shall be maintained for all samples in refrigerated, frozen, or special storage. Temperature logs shall be retained as specified in Section 1.9.

CROSS-REFERENCE: *For holding time requirements specified in the field, see the FMEnv SOPs for Environmental Sample Collection and Management. For sample disposal, see Module 8. For temperature data as QC evidence in analytical reporting, see Module 9.*

MODULE 4

Analytical Method Protocols for Priority Chemical Categories

Reference:
NESREA-SOP-MO
D04
Revision: Rev. 1.0

Date: 2026
Status: First Edition

4.1 Purpose

This Module specifies the approved standard analytical procedures for priority chemical categories encountered in chemicals and waste analysis conducted in support of NESREA's regulatory mandate and Nigeria's international environmental obligations. Detailed instrument-specific operating procedures shall be developed as laboratory-specific supplementary SOPs using the format in Annex A.

4.2 Scope

This Module covers analytical procedures for: heavy and trace metals; organochlorine pesticides and polychlorinated biphenyls (PCBs); organophosphate and carbamate pesticides; polycyclic aromatic hydrocarbons (PAHs); volatile organic compounds (VOCs); mercury (total and speciation); petroleum hydrocarbons; physicochemical parameters in water and waste; electronic waste (WEEE) components; plastic waste; battery waste.

NOTE ON METHOD VALIDATION: Methods used for regulatory compliance and enforcement purposes must be validated or verified for standard methods before first use on compliance samples. Minimum validation parameters are: linearity ($R^2 \geq 0.999$ for calibration curve); method detection limit (MDL); limit of quantification (LOQ); precision (RSD $\leq 10\%$ for three replicate analyses of CRM); accuracy (recovery 80 to 120% for matrix spike or CRM). Validation data shall be documented and retained as permanent laboratory records. 4.3 Section A: Heavy and Trace Metals Analysis

4.3.1 Principle and Applicable Instruments

Heavy metals in environmental, food, biological, and waste matrices are determined by Atomic Absorption Spectrometry (AAS) using flame (FAAS) or graphite furnace (GFAAS) modes; Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES); or Inductively Coupled Plasma Mass Spectrometry (ICP-MS). FAAS is appropriate for elements at concentrations above approximately 0.05 mg/L in solution. GFAAS is required for elements at trace concentrations, particularly cadmium, lead, and arsenic in biological matrices. ICP-OES enables simultaneous multi-element analysis and is preferred where available. ICP-MS provides the lowest detection limits and is required for speciation analysis.

4.3.2 Principal Reference Methods

- USEPA Method 200.7 (ICP-OES, water and wastes)
- USEPA Method 7000B (AAS general guidance)
- USEPA Method 7470A (mercury in liquid waste, CV-AAS)
- USEPA Method 3050B (acid digestion of sediment, sludge, soil)
- USEPA Method 3051A (microwave-assisted acid digestion)
- ISO 11885:2007 (ICP-OES, water quality)
- ISO 15586:2003 (GFAAS, water quality)

4.3.3 Sample Preparation

51. Water samples: For dissolved metals, filter through 0.45 μm membrane, then dilute if required. For total recoverable metals, digest 100 mL of sample with 3 mL

concentrated HNO₃ at 90 to 95 °C for 30 minutes, cool, filter, and make to volume (USEPA Method 200.2).

52. Solid matrices (soil, sediment, sludge, waste): Weigh 0.5 g of homogenised, air-dried sample into digestion vessel. For USEPA 3050B: add 10 mL HNO₃, heat to 95 °C for 10 to 15 min, add 5 mL concentrated HNO₃ and 3 mL 30% H₂O₂, reflux, cool, add 10 mL HCl and heat for 15 min, filter and make to volume. For microwave digestion (3051A): transfer to microwave vessel, add HNO₃/HCl (1:3 aqua regia), digest per validated microwave programme, cool and filter.
53. Biological tissue: Weigh 0.1 to 0.5 g dry weight. Digest with concentrated HNO₃ plus H₂O₂ in a microwave digestion system (or on hotplate reflux). Ensure complete digestion evidenced by a clear, colourless digest.
54. For mercury analysis of any matrix: use a separate digestion procedure (USEPA 7470A for aqueous, 7471B for solids) with BrCl or HNO₃/K₂Cr₂O₇. Do not use standard acid digestion vessels for mercury without dedicated equipment to prevent cross-contamination.

4.3.4 Calibration and Measurement

55. Prepare calibration standards from certified stock solutions by serial dilution in the blank matrix (2% HNO₃ for most metals). Prepare minimum five calibration points spanning the expected sample concentration range plus a zero standard.
56. Calibration curve linearity: coefficient of determination $R^2 \geq 0.999$. If $R^2 < 0.999$, investigate and recalibrate before proceeding.
57. For AAS, use standard addition method where matrix effects are suspected. For ICP, use internal standards (yttrium, rhodium) to compensate for matrix suppression or enhancement.
58. For each analytical sequence, bracket sample groups with continuing calibration verification (CCV) standards at every ten samples. CCV acceptance criterion: result within $\pm 10\%$ of the expected value.

4.3.5 QA/QC Requirements for Metals Analysis

Every analytical batch shall include: one method blank; one calibration verification standard (CCV) at 10% of calibration range; one certified reference material appropriate to the matrix and analytes; one matrix spike (MS) with target recovery 80 to 120%; and one duplicate (laboratory replicate). RPD between duplicates shall not exceed 20%.

4.3.6 Reporting Limits and Regulatory Reference Values

Metal	Matrix	Indicative LOQ	Regulatory Reference
Lead (Pb)	Water	0.5 µg/L	0.01 mg/L (NESREA Surface/Groundwater Regulations)
Cadmium (Cd)	Water	0.1 µg/L	0.003 mg/L
Mercury (THg)	Water	0.05 µg/L (CV-AAS)	0.001 mg/L
Arsenic (As)	Water	1 µg/L (GFAAS/ICP-MS)	0.01 mg/L
Lead (Pb)	Soil	1 mg/kg	USEPA RSL 400 mg/kg for reference

Mercury (THg)	Soil	0.05 mg/kg	Global Monitoring Plan background reference: 0.05 mg/kg
---------------	------	------------	---

4.4 Section B: Organochlorine Pesticides and PCBs

4.4.1 Principle and Applicable Instruments

Organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs), which are among the priority persistent organic pollutants subject to global elimination commitments, are determined by Gas Chromatography with Electron Capture Detection (GC-ECD), providing high selectivity for halogenated compounds, or by GC-MS for confirmation. GC-MS (or GC-MS/MS) confirmation is required for all compliance and enforcement samples and for any result above the applicable regulatory limit.

4.4.2 Principal Reference Methods

- USEPA Method 8081A: Organochlorine pesticides by GC-ECD
- USEPA Method 8082A: PCBs as Aroclors by GC-ECD
- USEPA Method 8270D: Semi-volatile organic compounds by GC-MS (confirmation)
- Global Monitoring Plan for persistent organic pollutants: Analytical methods (Chapters 3 and 4)
- USEPA Method 3540C / 3545A: Soxhlet / pressurised fluid extraction (solid matrices)

4.4.3 Sample Extraction and Cleanup

59. Water samples: Liquid-liquid extraction (LLE) with 3 x 60 mL dichloromethane from 1 L sample, pH adjusted to neutral. Combine extracts, dry over anhydrous Na_2SO_4 , concentrate to approximately 1 mL by rotary evaporation or gentle nitrogen blow-down. Alternatively, use SPE with C18 cartridge preconditioned with 5 mL MeOH then 5 mL water.
60. Solid matrices: Soxhlet extraction with 300 mL (1:1) acetone/hexane for 16 to 24 hours, or pressurised fluid extraction (USEPA 3545A) at 100 °C, 1500 psi with dichloromethane.
61. Cleanup: Florisil or alumina column cleanup is required for all soil, sediment, and tissue extracts. Elute Fraction 1 (non-polar OCPs and PCBs) with 100 mL petroleum ether; elute Fraction 2 (more polar OCPs) with 100 mL 15% diethyl ether in petroleum ether.
62. Concentrate final extract to 1.0 mL in isooctane under gentle nitrogen stream. Do not allow to go to dryness as volatile OCPs will be lost.

4.4.4 GC-ECD Analysis

63. Column: capillary column, 30 m x 0.25 mm x 0.25 µm film thickness, DB-5 or equivalent non-polar phase. Injector temperature: 250 °C. Detector temperature: 320 °C. Carrier gas: nitrogen or helium at 1 mL/min. Injection volume: 1 µL splitless.
64. Temperature programme (indicative): 60 °C (2 min hold) to 150 °C at 20 °C/min to 200 °C at 5 °C/min to 280 °C at 10 °C/min (10 min hold). Adjust for analyte resolution as needed.
65. Identify analytes by retention time comparison with certified reference standards. Retention time windows: ±0.1 minutes for a known compound. For any result ≥ LOQ, confirm identity by GC-MS before final reporting on a compliance sample.

4.4.5 QA/QC Requirements for OCP/PCB Analysis

Every analytical batch shall include: one solvent blank; one method blank; one matrix spike at 50 to 100 µg/kg (soil) or 0.1 to 1 µg/L (water) with target recovery 70 to 130%; one laboratory duplicate (RPD ≤ 30%); and one OCP CRM appropriate to the matrix analysed. Internal standards shall be added to every sample before extraction to monitor recovery.

NOTE ON DIOXIN REFERRAL: High-resolution analysis of dioxins (PCDD/Fs) and non-ortho PCBs requires High Resolution GC-MS (HRMS) on a magnetic sector or Orbitrap instrument. This capability is not currently available at any assessed laboratory in Nigeria. Where HRMS POPs analysis is required, samples shall be referred to a designated international accredited laboratory under the inter-laboratory referral protocol in Module 11.

4.5 Section C: Organophosphate and Carbamate Pesticides

Organophosphate pesticides (OPPs) and carbamate pesticides are analysed by GC with nitrogen-phosphorus detection (NPD) for routine multi-residue screening, or by LC-MS/MS for confirmation and for carbamates (which are heat-labile and unsuitable for GC without derivatisation). LC-MS/MS with electrospray ionisation (ESI) in positive mode provides the highest sensitivity and selectivity for multi-residue pesticide analysis across both OPPs and carbamates.

4.5.1 Principal Reference Methods

- USEPA Method 8141B: Organophosphorus compounds by GC-NPD/FPD
- Codex Alimentarius MRL methods: multi-residue screening by LC-MS/MS
- EN 15662 (QuEChERS, widely adopted for food matrices)

4.6 Section D: Polycyclic Aromatic Hydrocarbons (PAHs)

The EPA priority PAH list of 16 compounds is determined by HPLC with fluorescence detection (HPLC-FLD, preferred for sensitivity) or by GC-MS. HPLC-FLD achieves significantly lower detection limits than GC-MS for the higher-molecular-weight PAHs, which are the more carcinogenic compounds. Principal reference methods: USEPA Method 8310 (PAHs by HPLC); USEPA Method 8270D (GC-MS); USEPA Method 3540C (Soxhlet extraction for solid matrices).

4.7 Section E: Volatile Organic Compounds (VOCs)

VOCs in water are determined by purge-and-trap GC-MS (USEPA Method 8260C). VOCs in soil/waste are determined by static headspace GC-MS or direct headspace injection. VOC samples are among the most susceptible to loss during sample preparation: samples shall remain sealed until the moment of analysis; samples shall not be allowed to warm above 4 °C prior to purge-and-trap loading; sample transfers shall be performed using a syringe directly from the collection vial; soil samples shall be weighed without homogenisation and added directly to the purge vessel in methanol preservation.

4.8 Section F: Mercury (Total and Speciation)

Total mercury (THg) in environmental and biological matrices is determined by Cold Vapour Atomic Absorption Spectrometry (CV-AAS) or Cold Vapour Atomic Fluorescence Spectrometry (CV-AFS). CV-AAS is the most widely available technique in Nigeria. Methylmercury speciation, required for biological monitoring under the global monitoring programme for mercury, requires GC-ICP-MS or HPLC-ICP-MS. All equipment used for mercury analysis shall be dedicated to mercury and not used for any other metal analysis. Mercury vapour concentration in the laboratory air shall not exceed 0.05 mg/m³ (TWA). Principal reference methods: USEPA Methods 7470A and 7471B; global monitoring programme technical annex (UNEP, 2017); ISO 17852:2006.

4.9 Section G: Petroleum Hydrocarbons

Total petroleum hydrocarbons (TPH) in soil and water is determined by GC-FID following hexane or DCM extraction. USEPA Method 8015D is the standard approach. Gravimetric oil and grease measurement may be used for gross contamination screening. Additional reference methods: USEPA Method 1664A/B (oil and grease gravimetric); Standard Methods 5520B/C (hexane-extractable material in aqueous samples).

4.10 Section H: Physicochemical Parameters (Water and Waste)

Physicochemical parameters required for waste characterisation under the NESREA Sanitation and Wastes Control Regulations 2009 and for water quality compliance monitoring shall be determined using calibrated meters and titrimetric or spectrophotometric methods as follows:

Parameter	Method	Calibration Frequency	Reference Method
pH	Calibrated pH meter, 2-point calibration	Daily or before each use	ISO 10523 / APHA 4500-H
Conductivity (EC)	Conductivity meter with calibrated cell	Daily	ISO 7888
Dissolved Oxygen	Membrane electrode (Winkler titration for compliance)	Daily or before each use	ISO 5814 / APHA 4500-O
Turbidity	Nephelometer (NTU), formazin calibration	Before each batch	ISO 7027
TDS	Gravimetric (evaporation at 180 °C) or EC calculation	Per batch	APHA 2540C
COD	Closed reflux dichromate method	Calibration curve per batch	ISO 15705 / APHA 5220
BOD ₅	5-day incubation at 20 ± 1 °C, manometric or Winkler	Reference dilution water per batch	ISO 5815 / APHA 5210
TSS	Filtration through 0.45 µm GF/C filter, gravimetric	Blank filter weight per batch	ISO 11923 / APHA 2540D

4.11 Section I: Electronic Waste (WEEE) Analysis

4.11.1 Analytes of Concern in WEEE

Priority analytes in WEEE analysis include: lead (from solder, cathode ray tube glass, and cable sheaths); cadmium (from semiconductors and contacts); mercury (from fluorescent lamp backlights and relays); hexavalent chromium (from metal plating); arsenic and beryllium (from semiconductors); polybrominated biphenyls (PBBs) and polybrominated diphenyl ethers (PBDEs) from flame-retarded plastic casings and circuit boards; and phthalates from flexible cable insulation.

4.11.2 Sample Preparation

66. Physical fractionation: WEEE components shall be physically separated into material fractions before analysis. Metal-rich fractions (circuit boards, batteries, connectors) are separated from plastic-dominant fractions (casings, cables) and glass fractions (screens, CRTs). Each fraction shall be labelled and stored separately.
67. Metal-rich solid fractions: Crush or mill representative fraction to less than 1 mm particle size. Weigh 0.5 g, digest per USEPA 3050B or 3051A with aqua regia. Analyse digests by ICP-OES for Pb, Cd, Hg, Cr, As, Be, Ba, Cu, Ni, and Zn.
68. Plastic-dominant fractions: XRF screening per IEC 62321-4 provides a rapid first-tier result for Pb, Cd, Cr, Hg, and Br. For quantitative confirmation, microwave digest 0.3 g with concentrated HNO₃ and H₂O₂, then analyse by ICP-OES or GFAAS.
69. Fluorescent lamp and backlight components: Digest in HNO₃/HCl, analyse for mercury by CV-AAS per USEPA 7470A. Handle with full mercury PPE per Section 7.6 and dedicated equipment per Section 4.8.

4.11.3 Brominated Flame Retardants (PBBs and PBDEs)

70. Extract plastic fraction with toluene or DCM using ultrasonic extraction (USEPA 3550C). Clean up extract with multilayer silica/alumina column.
71. Analyse by GC-MS (USEPA 8270D) in selected ion monitoring mode, monitoring characteristic fragment ions for each PBDE congener group.
72. Express results as mg/kg per congener and as sum of PBDE congeners (total PBDE). Compare with applicable threshold values; EU RoHS limits (1000 mg/kg per substance) are the primary international reference in the absence of specific Nigerian limits.

4.12 Section J: Plastic Waste Analysis

4.12.1 Polymer Identification by ATR-FTIR

73. ATR-FTIR spectroscopy provides characteristic absorption spectra enabling identification of the dominant polymer type. Clean the sample surface with isopropanol. Place the sample on the ATR crystal. Collect spectrum across 4000 to 400 cm⁻¹ with 32 scans at 4 cm⁻¹ resolution. Match the resulting spectrum against a certified polymer spectral library.
74. Identification criteria: A library match score of 80% or above constitutes a positive identification. Report the polymer type (PE, PP, PVC, PET, PS, ABS, PC, PU, or other). For black or dark-coloured plastics, which may absorb excessively in ATR mode, use pyrolysis-GC-MS as an alternative identification technique.

4.12.2 Heavy Metal Additives in Plastic Waste

75. Sample preparation: Mill plastic to fine powder less than 0.5 mm. Weigh 0.5 g into digestion vessel. Microwave digest with concentrated HNO_3 and H_2O_2 per USEPA 3051A. Dilute and analyse by ICP-OES or GFAAS for Pb, Cd, Cr, Ba, Sb.
76. Screening option: XRF analysis per IEC 62321 provides rapid qualitative and semi-quantitative screening before destructive digestion. A positive XRF screen for Pb above 500 mg/kg or Cd above 50 mg/kg shall trigger confirmation by digestion and ICP-OES.
77. Regulatory reference values: In the absence of specific Nigerian limits, the laboratory shall apply EU RoHS limits as international benchmarks and shall note this in the analytical report.

4.12.3 Microplastic Analysis

78. Scope: Visual identification and enumeration of microplastic particles in water, sediment, or biological tissue samples.
79. Sample preparation for water: Filter sample through 0.3 mm stainless steel sieve, transfer residue to filter paper. Digest organic matter with 30% H_2O_2 if necessary. For sediment: mix with 30% H_2O_2 to digest organic matter, filter through 0.3 mm sieve.
80. Visual identification: Stereomicroscope at 40x magnification. Count and categorise particles by shape (fragment, fibre, film, bead), size class (less than 1 mm or 1 to 5 mm), and apparent colour.
81. Confirmation: ATR-FTIR or micro-Raman spectroscopy on a representative subset (minimum 10% of counted particles or 20 particles per sample, whichever is lower) for polymer type confirmation.
82. Report particle count per litre for water or per kilogram dry weight for sediment. Report morphology categories and confirmed polymer types.

4.13 Section K: Battery Waste Analysis

4.13.1 Regulatory and Standards Basis

- National Environmental (Sanitation and Wastes Control) Regulations, 2009 (S.I. No. 28 of 2009).
- NESREA Act, 2007, Section 7(g): Handling and disposal of hazardous chemicals and waste
- EPR framework for battery waste established under NESREA's Extended Producer Responsibility Policy
- International technical guidelines on environmentally sound management of waste lead-acid batteries
- SON NIS standards on battery specifications and testing where applicable

4.13.2 Battery Types and Principal Hazardous Constituents

Battery Type	Principal Hazardous Constituents	Key Analytical Parameters
Lead-acid	Lead (60 to 70% by weight), sulfuric acid electrolyte	Pb, Sb, Sn, Ca (metals); H_2SO_4 concentration, sulfate (electrolyte)

Nickel-cadmium (Ni-Cd)	Cadmium (15 to 20%), nickel, KOH electrolyte	Cd, Ni (metals); OH ⁻ concentration (electrolyte)
Lithium-ion (Li-ion)	Lithium, cobalt (LiCoO ₂ cathode), nickel, manganese, organic carbonate electrolyte with LiPF ₆	Co, Ni, Mn, Li (metals); F ⁻ (electrolyte)
Nickel-metal hydride (Ni-MH)	Rare earth metals, nickel, KOH electrolyte	Rare earth elements, Ni; OH ⁻ concentration

SAFETY NOTE: Battery waste analysis requires additional precautions. Spent batteries may contain residual electrical charge, flammable electrolytes, and reactive lithium. Batteries shall be fully discharged before disassembly. All disassembly shall be performed in a well-ventilated fume hood. Lithium-ion batteries shall not be cut or punctured until fully discharged. The two-person rule (Module 7) applies to all battery disassembly procedures.

4.13.2 Lead-Acid Battery Waste Analysis

83. Electrolyte analysis: Collect electrolyte using a syringe or pipette. Dilute with distilled water. Determine pH, specific gravity, sulfate content by barium sulfate gravimetric method or ion chromatography, and total lead by ICP-OES or FAAS after appropriate dilution.
84. Active material (plates): Crush and homogenise plate material. Weigh 0.5 g, digest with 10 mL HNO₃ plus 3 mL HCl at 95 °C. Dilute and analyse by ICP-OES for Pb, Sb, Sn, Ca, and Se.

4.13.3 Nickel-Cadmium Battery Waste Analysis

Separate and dry electrode material. Digest 0.1 g in 5 mL HNO₃ plus 1 mL HCl by microwave digestion. Dilute and analyse by GFAAS (cadmium at 228.8 nm) and FAAS or ICP-OES (nickel at 232.0 nm). Cadmium is the critical parameter. Report Cd in mg/kg with comparison to NESREA Hazardous Wastes Regulations 2014 classification thresholds.

4.13.4 Lithium-Ion Battery Waste Analysis

85. Cathode material analysis: Microwave digest 0.2 g in aqua regia. Analyse by ICP-OES for Co, Ni, Mn, Li, Al, and Cu.
86. Electrolyte analysis: Where accessible, dissolve electrolyte in acetonitrile or DMF. Analyse by ion chromatography for F⁻, PF₆⁻, and SO₄²⁻. Fluoride determination is critical for lithium-ion electrolytes. LiPF₆ hydrolyses on contact with moisture to generate hydrogen fluoride. Ion chromatography analysis for fluoride shall be conducted with appropriate safety precautions per Section 7.6.

4.14 Referral Protocol for Analyses Beyond Domestic Capability

The following analytical requirements cannot currently be met by any assessed laboratory in Nigeria and shall be referred to a designated international accredited laboratory under Module 11:

- Dioxins and furans (PCDD/Fs) and non-ortho PCBs: High-resolution GC-MS (HRMS) required under the Global Monitoring Plan for persistent organic pollutants.
- Methylmercury speciation at sub-ppb levels in biological matrices: GC-ICP-MS required under the global monitoring programme for mercury.
- Chemical weapons agents and precursors: OPCW-approved multi-technique characterisation (limited to one designated national facility).
- Ultra-trace multi-element analysis in precipitation or remote area monitoring requiring ICP-MS at ng/L levels.
- Advanced polymer characterisation requiring pyrolysis-GC-MS not available at domestic laboratories.
- Emerging contaminants including PFAs

4.15 Method Validation Requirements

Before implementation for regulatory or compliance purposes, all analytical methods shall undergo validation sufficient to demonstrate fitness for the intended use. This requirement applies to new methods, modified standard methods, and methods transferred from another laboratory. Validation data shall be documented, reviewed by the Laboratory Director or Quality Officer, and retained as permanent quality records.

Validation shall address, where applicable to the method: accuracy; precision expressed as repeatability and reproducibility; selectivity and specificity; linearity and calibration range; limit of detection; limit of quantification; recovery efficiency across the applicable concentration range and matrix types; robustness against deliberate variation in critical parameters; and matrix interference evaluation. For methods routinely applied to complex environmental matrices, the validation shall include at least two representative matrix types.

Internationally recognized standard methods from USEPA SW-846, ISO, ASTM, or equivalent reference bodies, when applied without modification, shall be considered validated for the scope stated in the reference method. Any modification to a standard method shall be treated as a new method and shall require full or partial re-validation as determined by the Laboratory Director based on the nature and extent of the modification.

4.16 Emerging Contaminants

Laboratories are encouraged to develop or acquire capability for the analysis of emerging contaminants of environmental significance, including per- and polyfluoroalkyl substances (PFAS), microplastics, pharmaceutical and personal care product residues, and other contaminants of emerging concern identified in UNEP guidance or Nigerian regulatory frameworks. Where such capability does not currently exist, laboratories shall establish referral arrangements under Module 11 for samples where these contaminants are suspected or specified by the submitting authority.

NESREA shall periodically review and update the list of analytes covered by this Manual to reflect emerging regulatory priorities and advances in analytical capability across the national laboratory network.

All referral samples shall be processed under the inter-laboratory referral procedure in Module 11. The Laboratory Director shall authorise all referrals in writing. Results received from international laboratories shall be incorporated into the Analytical Result Report (Module 9) with attribution.

CROSS-REFERENCE: For QA/QC requirements applying to all analyses in this Module, see Module 6. For equipment-specific operating procedures, see Module 5. For sample preparation and holding time constraints, see Module 3. For reporting of results, see Module 9. For referral, see Module 11.

MODULE 5 Equipment Operation, Calibration, and Maintenance

Reference:
NESREA-SOP-MO
D05
Revision: Rev. 1.0

Date: 2026
Status: First Edition

5.1 Purpose

This Module establishes the requirements for the operation, calibration, performance verification, and preventive maintenance of analytical instruments and supporting equipment in chemicals and waste analysis laboratories. It ensures that all instruments used in the generation of analytical data are fit for purpose, operating within specified performance parameters, and supported by documentation that demonstrates their performance status at the time any given sample was analysed.

5.2 Scope

This Module applies to all major analytical instruments, including: AAS, ICP-OES, GC-MS, GC-ECD, GC-FID, GC-NPD, HPLC, LC-MS, FTIR, ATR-FTIR, XRF, UV-Vis spectrophotometers, pH meters, conductivity meters, dissolved oxygen meters, balances, pipettes, and sample preparation equipment. It also applies to sample storage equipment (refrigerators and freezers) and environmental monitoring equipment (temperature loggers and fume hood airflow monitors).

5.3 Equipment Records

5.3.1 Equipment Inventory

The Quality Officer shall maintain an Equipment Inventory Register listing every instrument and item of significant equipment in the laboratory. The Register shall record for each item: unique equipment identification number; equipment description, manufacturer, model, and serial number; location within the laboratory; date of acquisition; date of last calibration and calibration due date; date of last preventive maintenance and next due date; current operational status; and service contract details where applicable.

5.3.2 Equipment Files

An individual Equipment File shall be maintained for each major analytical instrument. The file shall contain: a copy of the equipment identification label; manufacturer's operation manual; calibration and maintenance records (Form LF-04); records of any faults, repairs, and modifications; any validation data applicable to the instrument; and service engineer visit reports. Equipment Files shall be retained for the life of the instrument plus five years.

5.4 Calibration Requirements

5.4.1 General Calibration Principles

87. All calibrations shall be traceable to national or international measurement standards through an unbroken chain of comparisons, each with stated uncertainty. Calibration standards shall be obtained from National Metrology Institute (NMI), SANAS-accredited or internationally accredited suppliers. Certificates of analysis with certified values and uncertainties shall accompany all calibration standards and CRMs.
88. Calibration of measurement equipment shall be performed by a qualified metrological service or internally against traceable reference standards at the frequencies shown

in the table below or as contained in the equipment manual. All calibration activities shall be recorded in Form LF-04.

Equipment Type	Calibration Frequency	Performance Check	Reference Standard
Analytical balance (4 d.p.)	Annually (external)	Daily (calibration weight)	OIML-traceable weights
Top-loading balance (2 d.p.)	Annually (external)	Weekly	OIML-traceable weights
Micropipettes (all sizes)	6-monthly (gravimetric)	Monthly (check weight)	Calibrated balance + distilled water
pH meter and electrode	Electrode replacement per manufacturer	Daily (2-point, pH 4 and 7 or 7 and 10)	NIST-traceable buffer solutions
Conductivity meter	Annually	Before each batch	Certified conductivity standard
Thermometers / data loggers	Annually	Monthly	Calibrated reference thermometer
Fume hood air velocity	Annually by engineer	Monthly (visual indicator)	Calibrated anemometer
AAS (wavelength/response)	Per manufacturer	Daily system suitability test (SST)	CVM / hollow cathode lamp check
GC (retention time, response)	After any column change	Before each analytical run (SST)	Certified mixed standard solution
HPLC (flow, pressure, UV)	After column change / pump service	Before each analytical run (SST)	Certified standard, caffeine or similar
FTIR/ATR-FTIR	Per manufacturer schedule	Before each use (polystyrene reference)	NIST polystyrene film SRM 1921b

5.4.2 System Suitability Testing (SST)

Before commencing any analytical run that will produce results for compliance or enforcement purposes, the analyst shall perform and record a System Suitability Test. If any SST criterion is not met, the run shall not proceed. The failure shall be recorded in Form LF-04, the cause investigated, and the SST repeated after corrective action. Minimum SST requirements are:

- GC-MS: injection of a certified mixed standard at 50% of calibration range; check peak area response for defined analytes within $\pm 15\%$ of historical average; resolution between specified adjacent peaks ≥ 1.5 ; signal-to-noise ratio ≥ 10 for target analyte.
- HPLC: retention time reproducibility $\leq 0.5\%$ RSD (five injections of standard); theoretical plates ≥ 5000 for main peak; tailing factor ≤ 2.0 .
- AAS: hollow cathode lamp stability after 30 min warm-up; absorbance check against certified standard within $\pm 5\%$ of historical value; background corrector operational.
- FTIR/ATR-FTIR: polystyrene film reference spectrum recorded and compared with reference file; peak positions within $\pm 2 \text{ cm}^{-1}$ for standard peaks.

5.5 Preventive Maintenance

89. Preventive maintenance (PM) shall be performed at intervals specified by the instrument manufacturer and documented in supplementary instrument-specific SOPs. PM activities shall be recorded in Form LF-04, signed by the person performing the maintenance.
90. The Quality Officer shall maintain a PM schedule for every instrument and shall issue a reminder to the relevant analyst at least 14 days before each scheduled PM.

5.6 Equipment Failure, Repair, and Return to Service

91. When an instrument fails or is taken out of service, the analyst shall: immediately notify the Laboratory Director and Quality Officer; attach a clear "OUT OF SERVICE: DO NOT USE" label to the instrument; record the failure in Form LF-04 with the date, description of the fault, and any analytical runs that may have been affected.
92. Analytical results generated on an instrument during any period in which its performance may have been sub-specification shall be flagged for review. If the instrument failure is likely to have compromised analytical results, the affected samples shall be re-analysed on a functional instrument or referred (Module 11).
93. Before returning an instrument to routine analytical service after repair or major maintenance, a return-to-service check shall be performed, comprising: an SST; a CRM analysis; and comparison of the CRM result with the certified value (acceptance criterion: result within $\pm 10\%$ of certified value). Return to service shall be authorised in writing by the Laboratory Director and recorded in Form LF-04.

5.7 Service Contracts and Spare Parts

Every laboratory operating under this Manual is strongly encouraged to enter into service contracts with the manufacturer or an authorised service provider for each major analytical instrument. Where service contracts cannot be secured due to funding constraints, the laboratory shall: maintain a documented record of local and international service contacts for each instrument; identify and maintain a minimum stock of commonly failing components; and pursue collaborative service contract arrangements with other laboratories in the same geopolitical zone.

5.8 Documentation

Form LF-04 (Equipment Calibration and Maintenance Log, Annex E) shall be completed for all calibration and maintenance activities. Entries shall be made contemporaneously and signed by the analyst performing the activity.

5.9 Instrument Qualification

Major analytical instruments used for regulatory compliance analysis, including GC-MS, LC-MS/MS, ICP-MS, ICP-OES, and HPLC systems, shall undergo documented qualification before being placed into routine service and following any major repair, relocation, or significant modification.

Qualification shall proceed in three documented stages. Installation Qualification establishes that the instrument has been delivered, installed, and configured in accordance with the manufacturer's specifications and the laboratory's requirements. Operational Qualification verifies that the instrument operates within defined performance parameters under representative laboratory conditions. Performance Qualification confirms that the instrument consistently performs within acceptance criteria for the intended analytical methods and analyte-matrix combinations.

Qualification records shall be maintained throughout the operational life of the instrument and shall be reviewed during internal audits and accreditation assessments. Where an instrument has not been formally qualified, any analytical results produced on that instrument for regulatory purposes shall be clearly identified as unqualified and shall carry an appropriate qualification statement in the Analytical Result Report.

CROSS-REFERENCE: *For analytical run performance criteria, see Module 6 (QA/QC). For instrument-specific operating procedures, develop supplementary SOPs using the template in Annex A. For safety precautions specific to instrument operation, see Module 7.*

MODULE 6
Quality Assurance and Quality Control
Reference:
 NESREA-SOP-MO
 D06
Revision: Rev. 1.0

Date: 2026
Status: First Edition

6.1 Purpose

This Module establishes the minimum quality assurance (QA) and quality control (QC) programme for laboratories conducting chemicals and waste analysis under this Manual. It specifies the QC samples required in every analytical batch, acceptance criteria for each QC measure, corrective action requirements when acceptance criteria are not met, and the documentation and reporting of QC data. Implementation of this Module transforms ad hoc laboratory practice into a systematic quality management programme aligned with ISO/IEC 17025:2017 and WHO GLP requirements.

6.2 Scope

This Module applies to all quantitative analytical procedures performed by the laboratory, encompassing all chemical categories covered in Module 4. The QA programme at the system level is established through Module 1 and Module 5. This Module addresses the QC programme at the batch and run level.

6.3 Required QC Samples per Analytical Batch

An analytical batch is a set of samples extracted, digested, or prepared together under the same conditions in a single sequence. Every analytical batch shall include the following QC samples:

QC Sample Type	Preparation	Acceptance Criterion	Corrective Action if Failed
Method Blank (MB)	Reagent water or solvent through complete extraction and analysis sequence	Analyte not detected above LOD; if detected, MB concentration $\leq 10\%$ of sample concentration or 5x LOD	Investigate source of contamination; prepare new reagents and repeat batch if MB exceeds criterion
Calibration Verification Standard (CVS)	Certified standard at 50% of calibration range, from a different stock than calibration standards	Measured value within $\pm 10\%$ of expected value	Recalibrate; check standard preparation; do not report data from affected run
Certified Reference	Appropriate CRM for analyte and	Measured value within certified	Investigate method; re-validate extraction/digestion; do not report

Material (CRM)	matrix; process through complete extraction sequence	uncertainty range or within $\pm 15\%$ of certified value	compliance data until CRM performance restored
Matrix Spike (MS)	Fortify one sample in the batch with known amount of target analyte (typically 1 to 5x sample concentration)	Recovery 70 to 130% for most analytes; 50 to 150% for complex matrices	Indicate potential matrix interference in report; consider standard addition method; report result with qualification
Matrix Spike Duplicate (MSD)	Second fortified portion of the same sample as MS; processed independently	RPD between MS and MSD $\leq 20\%$ ($\leq 35\%$ for complex or heterogeneous matrices)	Investigate extraction reproducibility; check homogenisation
Laboratory Duplicate (LD)	Second independent aliquot of one field sample processed identically	RPD $\leq 20\%$ ($\leq 30\%$ for heterogeneous solid matrices)	Investigate sample homogeneity and analytical precision; flag result
Internal Standard (where applicable)	Added to every sample at constant concentration before extraction (GC-MS, LC-MS analyses)	Recovery 60 to 140% of expected response	Investigate instrument performance; check extraction; potential sample interferences

6.4 Continuing Calibration Verification (CCV)

In addition to the initial calibration and the CVS at the beginning of each batch, a CCV standard shall be injected or measured every 10 samples throughout the analytical run. The CCV acceptance criterion is $\pm 10\%$ of the expected value. If the CCV fails, all samples measured since the last acceptable CCV shall be re-analysed after recalibration. Data from the failed period shall not be reported without qualification.

6.5 Calculation of Performance Metrics

6.5.1 Method Detection Limit (MDL)

MDL shall be determined at method establishment and annually thereafter, following the procedure in 40 CFR Part 136, Appendix B (USEPA). Prepare seven replicate analyses of a sample fortified at 2 to 5x the estimated LOD. Calculate $MDL = t(n-1, 0.99) \times s$, where t is the one-tailed t -distribution value at 99% confidence for $(n-1)$ degrees of freedom and s is the standard deviation of the replicates.

6.5.2 Relative Percent Difference (RPD)

$RPD = |Result_1 \text{ minus } Result_2| / [(Result_1 + Result_2)/2] \times 100\%$. Used to assess precision between duplicates (LD) and between matrix spike and matrix spike duplicate (MS/MSD).

6.5.3 Percent Recovery (%R)

$\%R = (\text{Measured Concentration minus Background Concentration}) / \text{Added Concentration} \times 100\%$. Applied to matrix spike and CRM analyses.

6.6 QC Batch Record

All QC data for each analytical batch shall be recorded in Form LF-05 (Analytical QC Batch Record, Annex F). The QC Batch Record shall document: batch identification; analytical method; instrument used; analyst name; date; all QC sample identifications; all QC results; acceptance criteria; pass/fail status; and any corrective actions taken. The QC Batch Record shall be signed by the analyst and reviewed by the Quality Officer before results are reported.

6.7 QC Control Charts

Laboratories processing ten or more batches per month for a given analyte-matrix combination shall maintain statistical process control (SPC) charts for that combination. Minimum chart types required are an X-bar chart and an s-chart or R-chart for the CRM and MS results. Control limits shall be established from 20 or more historical data points: warning limits at $\pm 2\sigma$ and action limits at $\pm 3\sigma$. A data point falling outside action limits, or two successive points between warning and action limits, shall trigger a corrective action investigation.

6.8 Proficiency Testing (PT) and Inter-Laboratory Comparisons

94. Every laboratory covered by this Manual shall participate in at least one proficiency testing scheme per year for each major analyte category routinely analysed. Available PT schemes include: the IPAN PT Programme; NAFDAC-coordinated national PT scheme; WHO-affiliated laboratory quality assurance schemes for specific matrix types; and inter-laboratory comparison exercises for POPs and mercury facilitated through NESREA.
95. PT results shall be evaluated using z-scores. $|z| \leq 2.0$: satisfactory. $2.0 < |z| < 3.0$: questionable (requires investigation and corrective action). $|z| \geq 3.0$: unsatisfactory (requires root cause analysis, corrective action, and Quality Officer notification to Laboratory Director).
96. All PT results and corrective actions shall be documented and presented at the annual Management Review (Section 1.7). Laboratories with consistently poor PT performance ($|z| \geq 3.0$ in three successive rounds for the same analyte) shall suspend the reporting of compliance results for that analyte until satisfactory performance is demonstrated, and shall notify NESREA.

6.9 Reference Material Procurement and Storage

The procurement of certified reference materials is acknowledged as a significant practical constraint for many Nigerian laboratories due to foreign currency requirements and import

lead times. Recommended strategies include: pool procurement of CRMs with other laboratories through NESREA coordination; participation in a NESREA-coordinated national CRM repository if established; preparation of in-house secondary reference standards from certified primary standards, validated against CRMs every six months; and exploration of Nigerian-matrix CRMs through collaborative agreement with NARICT. In no case shall a laboratory report compliance results without having validated the method against at least one CRM within the current calendar year.

6.10 Non-Conforming Work

When any QC acceptance criterion is not met, the analyst shall: stop the analytical run; record the failure in Form LF-05; notify the Quality Officer; investigate the probable cause; apply the corrective action specified in Section 6.3; document the corrective action and its outcome; and, where the non-conformance may have affected previously reported results, notify the Laboratory Director who shall determine whether to issue a revised result report.

6.11 Measurement Uncertainty

Each laboratory shall establish and maintain procedures for estimating measurement uncertainty for quantitative analytical methods applied for regulatory compliance determination, enforcement action, or scientific interpretation. Measurement uncertainty shall be estimated using a documented procedure reviewed and approved by the Laboratory Director.

Measurement uncertainty estimation shall consider, where applicable and relevant to the analytical method: calibration uncertainty derived from reference standards and calibration curve fitting; instrument precision including short-term and long-term repeatability; sample preparation variability including subsampling, digestion, extraction, and dilution steps; analyst-to-analyst and shift-to-shift variability; matrix effects on instrument response; recovery efficiency and its variability; and relevant environmental conditions affecting analysis such as temperature variation. The methodology used shall be consistent with internationally accepted guidance, such as that of EURACHEM/CITAC or JCGM 100, and shall be documented in a method-specific uncertainty budget.

Where required by regulation, accreditation standard, or the request of the submitting authority, reported analytical results shall include an expanded uncertainty statement expressed at the 95 per cent confidence level. Measurement uncertainty estimates shall be reviewed whenever significant changes are made to method parameters, instrumentation, or laboratory conditions.

6.12 Corrective and Preventive Action

Where analytical non-conformities, QC failures, audit findings, equipment malfunctions, or procedural deviations are identified, the laboratory shall implement documented corrective action procedures. Corrective action shall include: identification and documentation of the non-conformity in Form LF-05; root cause analysis to identify the underlying cause of the failure; implementation of corrective measures to address the root cause; verification that the corrective action has been effective; and measures to prevent recurrence. All corrective action records shall be maintained by the Quality Officer and reviewed during Management Review meetings.

In addition to corrective action following identified failures, laboratories shall implement a preventive action system based on risk-based analysis of trends in QC data, audit observations, and near-miss events. Preventive action shall be documented and tracked through the same Form LF-05 system used for corrective action. Where a recurring pattern of non-conformity is identified, the Laboratory Director shall initiate a formal root cause review and update procedures accordingly.

6.13 Proficiency Testing Frequency

Laboratories conducting regulatory analytical functions shall participate in relevant proficiency testing or inter-laboratory comparison programmes at least once annually for each major analytical category performed. Laboratories performing high-priority or high-risk analyses, including heavy metals, persistent organic pollutants, pesticides, and mercury speciation, should participate in proficiency testing at least twice annually where suitable programmes are available. Proficiency testing participation records and performance evaluations shall be retained and reviewed at Management Review meetings. Unsatisfactory proficiency testing results shall generate corrective action under Section 6.12.

CROSS-REFERENCE: *QA/QC requirements for specific analytical methods are stated in Module 4. Calibration and system suitability data are generated under Module 5. QC results are incorporated in the Analytical Result Report per Module 9. Management Review of QC performance occurs under Section 1.7.*

MODULE 7**Laboratory Safety, Chemical Hygiene, and Personal Protective Equipment**

Reference:
NESREA-SOP-MO
D07
Revision: Rev. 1.0

Date: 2026
Status: First Edition

7.1 Purpose

This Module establishes the minimum safety requirements, chemical hygiene standards, personal protective equipment (PPE) specifications, and emergency preparedness requirements for chemicals and waste analysis laboratories. It is designed to protect laboratory personnel from chemical hazards inherent in analytical operations, to minimise environmental release of hazardous substances from the laboratory, and to establish consistent safety practices across all laboratories operating under this Manual.

7.2 Scope

This Module applies to all persons entering and working within the laboratory, including analysts, technical officers, support staff, visiting officers, and contractors, and to all laboratory chemical operations including sample receipt, sample preparation, instrumental analysis, waste handling, and equipment cleaning and occupational exposure monitoring. It applies to the analysis of all sample types covered in Module 4, including electronic waste, plastic waste, and battery waste.

7.3 Laboratory Chemical Hygiene Plan

Every laboratory covered by this Manual shall maintain a written Laboratory Chemical Hygiene Plan. It is the central safety management document for the laboratory, specific to that laboratory's chemical inventory and analytical operations. It shall be reviewed and updated annually or whenever a new class of hazardous chemical is introduced. At minimum, it shall contain: a laboratory chemical inventory and SDS library; a chemical hazard assessment by procedure type; PPE requirements by chemical category and procedure; standard operating procedures for high-hazard operations (Sections 7.6 to 7.11 of this Module); a designated Chemical Hygiene Officer and responsibilities; a training schedule; emergency response procedures (Module 10); waste disposal procedures (Module 8); and an annual review record. Responsibility for the Chemical Hygiene Plan rests with the Laboratory Director, who shall assign day-to-day coordination to a designated Chemical Hygiene Officer.

7.4 Chemical Inventory and Safety Data Sheets

97. The laboratory shall maintain a complete, current inventory of all chemicals in use and in storage, updated whenever chemicals are received or stocks are exhausted. The inventory shall record for each chemical: name (IUPAC and common name); CAS number; quantity on hand; storage location; GHS hazard classification; and SDS availability.
98. An SDS shall be available for every chemical in the laboratory inventory. SDS documents shall be current (not more than five years old unless the manufacturer certifies no change) and shall be accessible to all laboratory personnel during working hours without requiring access to a locked room. A printed SDS binder shall be maintained in both the chemical storage area and the analyst workstation area.
99. For expired laboratory chemicals, the National Guidelines on Management and Disposal of Expired Chemicals apply in full. The obligations under Sections 5 through

10 of those Guidelines, covering annual inventory reviews, notification to NESREA within 14 days of identifying expired stock, and licensing requirements for generators, apply to laboratories as they do to industrial and institutional generators.

CROSS-REFERENCE: *Expired laboratory chemicals are subject to the National Guidelines on Management and Disposal of Expired Chemicals. This Module governs hazards from chemicals in active use. The two instruments are complementary.*

7.5 PPE Requirements by Procedure Category

Procedure Category	Minimum PPE	Additional PPE	Notes
General laboratory entry	Lab coat, closed-toe shoes	Safety spectacles	Lab coat must be cotton or Fire Resistant (FR) material for solvent work
Sample receipt and handling (all matrices)	Gloves (nitrile), lab coat, safety spectacles	Face shield for waste samples	Double-glove for unknown waste matrices
Acid/alkali digestion	Nitrile gloves, lab coat, safety spectacles, face shield	Chemical-resistant apron	Perform in fume hood only; verify airflow before starting
Hydrofluoric acid (HF) digestion	Neoprene or butyl rubber gloves, face shield, acid-resistant apron and boots	Calcium gluconate gel (antidote) on person	RESTRICTED: requires Laboratory Director approval; two-person rule; two complete sets of protective gear required
Aqua regia preparation	Nitrile gloves, face shield, acid-resistant apron, lab coat	Chemical-resistant gloves (double)	Prepare in fume hood; never seal in closed container; cool before adding samples
Organic solvent handling	Nitrile gloves, lab coat, safety spectacles	Organic vapour respirator if ventilation is marginal	No naked flames; use non-sparking equipment; store in flammable cabinet
Mercury CV-AAS analysis	Nitrile gloves (double), lab coat, safety spectacles	Mercury vapour monitor or detector	Dedicated equipment; never share bench with general metals; mercury spill kit within 10 m; ventilation ≥ 10 air changes/h
Cyanide-containing samples	Nitrile gloves, lab coat, face shield	Monitor pH: must remain alkaline	Strictly segregate from acids; antidote cyanide kit available; two-person minimum rule

PCB/OCP extraction with chlorinated solvents	Nitrile or neoprene gloves, lab coat, safety spectacles	Organic vapour respirator	Use amber glassware; avoid skin contact with DCM
WEEE component disassembly	Nitrile gloves, lab coat, safety spectacles, cut-resistant gloves	Face shield for CRT/screen work	Discharge batteries fully; use non-sparking tools; ventilate for dust and lead aerosol
Battery waste disassembly	Nitrile gloves (double), lab coat, safety spectacles, face shield	Chemical-resistant apron for electrolyte handling	Two-person rule; Li-ion batteries must be fully discharged before cutting; electrolyte may be flammable
Biological samples (unscreened human specimens)	Double gloves, lab coat, safety spectacles, face shield	Class II Biological Safety Cabinet required	If Class II BSC unavailable, do not process; refer per Module 11

7.6 Fume Hood Requirements

100. Minimum face velocity for fume hoods used for acid digestion, volatile solvent handling, or toxic gas generation procedures: 0.4 to 0.5 m/s. Face velocity shall be verified before each use using the hood's built-in flow indicator and formally measured by calibrated anemometer not less than annually.
101. The following operations shall only be performed inside a functioning fume hood with verified adequate airflow: all acid digestions; all organic solvent evaporations exceeding 100 mL; mercury cold vapour analysis; preparation of cyanide solutions; and any procedure generating pungent or irritating vapours.
102. If the fume hood fan is non-functional or airflow is inadequate, the hood shall be taken out of service, labelled "OUT OF SERVICE", and all procedures requiring the hood shall be suspended until the fault is rectified.

7.7 Eyewash and Safety Shower Requirements

103. A functioning eyewash station shall be available within 10 seconds of any area where corrosive chemicals are used. The eyewash shall be activated and flushed for at least 30 seconds weekly, and the flush recorded in the laboratory safety log.
104. A safety shower shall be available within the laboratory building for any laboratory regularly using quantities of corrosive, oxidising, or flammable chemicals exceeding 1 litre. Laboratories lacking fixed safety showers shall maintain a portable emergency eyewash station and shall prioritise installation of a fixed safety shower in their infrastructure upgrade plans.

7.8 Chemical Storage Requirements

All chemicals shall be stored in accordance with GHS hazard class compatibility as set out in Annex L (Chemical Compatibility and Segregation Chart). The following segregations are mandatory and non-negotiable:

- Acids shall not be stored with bases, oxidisers, or flammable solvents.
- Oxidisers (KMnO_4 , H_2O_2 , HNO_3) shall be stored separately from organic solvents.
- Cyanide compounds shall be stored separately from all acids and oxidisers.
- Mercury and mercury compounds shall be stored in a sealed secondary container, separately from all reagents.
- Flammable solvents shall be stored in an approved flammable material safety cabinet.
- Quantities of flammable solvents on the open bench shall not exceed one working day's requirement; all remaining stock shall be in the flammable cabinet.
- Chemicals shall not be stored above eye level, on the floor, in hallways, or in areas accessible to unauthorised persons.
- Chemical storage areas shall be locked when unattended.

7.9 Electrical Safety

105. All analytical instruments shall be connected to a properly grounded electrical supply. Extension cords shall not be used as a permanent wiring solution. Instruments with high-voltage components shall have their isolation switches clearly labelled and accessible.
106. Uninterruptible Power Supplies shall be installed on instruments with data systems and on temperature-sensitive storage equipment wherever possible, given the frequency of power supply interruptions in Nigeria.
107. Generators used as backup power shall be located outside the laboratory building with adequate ventilation to prevent CO accumulation. Generator exhaust shall not be directed toward laboratory air intakes.

7.10 First Aid Requirements

Every laboratory shall maintain a first aid kit readily accessible to all personnel, stocked at minimum with: 500 mL sterile eye irrigation saline; disposable gloves; adhesive dressings; sterile gauze dressings; burn dressing or gel; bandages; first aid scissors and forceps. Laboratories handling HF shall additionally stock calcium gluconate gel 2.5% (at least two tubes). Laboratories handling cyanide shall additionally stock a hydroxocobalamin injection kit. All personnel shall be trained in basic first aid and in the location and use of antidotes for chemicals they regularly handle.

7.11 Safety Training Requirements

108. All laboratory personnel shall receive safety training on the Laboratory Chemical Hygiene Plan before commencing laboratory work and shall demonstrate competency in: correct use of all relevant PPE; response to chemical spill (Module 10); evacuation procedure; eyewash and safety shower operation; fire extinguisher operation; and emergency contact numbers.
109. Safety training records shall be maintained by the Chemical Hygiene Officer and reviewed at each annual Management Review (Section 1.7 of Module 1).

7.12 Occupational Exposure Monitoring

Laboratories routinely handling chemicals with recognized occupational exposure limits, including mercury, lead, chromium compounds, persistent organic pollutants, and volatile

organic solvents, shall implement a programme of occupational exposure monitoring to verify that analyst exposures remain within acceptable limits. The monitoring programme shall be documented as part of the Laboratory Chemical Hygiene Plan and reviewed annually.

Where air monitoring or biological monitoring indicates exceedance of occupational exposure limits, the Laboratory Director shall immediately implement interim engineering or administrative controls, notify affected personnel, and arrange for medical evaluation by a qualified occupational health practitioner. Monitoring results and any consequent actions shall be documented and retained.

7.13 Biosafety Provisions

Samples reasonably suspected to contain infectious biological material, including wastewater, sewage sludge, biological waste streams, and samples from disease-surveillance programmes, shall be handled in accordance with applicable biosafety procedures in addition to the chemical safety requirements of this Module.

Laboratories handling mixed chemical-biological samples shall implement: biological hazard identification procedures at the sample reception stage; appropriate containment measures consistent with the assessed biosafety risk level; decontamination protocols for surfaces, equipment, and waste streams; biological waste segregation and disposal procedures compliant with applicable health and environmental regulations; and biosafety training for all personnel handling such samples. Where a laboratory does not have biosafety infrastructure commensurate with the assessed risk of a sample, the sample shall be referred to a suitably equipped facility under Module 11 rather than being analysed in-house.

7.14 GHS Labelling and Chemical Hazard Communication

All chemicals within the laboratory shall be labelled in accordance with the Globally Harmonized System of Classification and Labelling of Chemicals. Labels shall include the chemical name, GHS hazard pictograms, signal word, hazard statements, precautionary statements, and the name and contact details of the supplier. Where chemicals are transferred from original containers into working stock containers or secondary containers, the secondary containers shall carry labels meeting the same GHS requirements. Unlabelled containers shall not be used. A current Safety Data Sheet shall be immediately accessible for every chemical in the laboratory.

CROSS-REFERENCE: *For emergency response procedures in the event of a laboratory chemical incident, see Module 10. For laboratory waste disposal, see Module 8. For handling of expired laboratory chemicals, see the National Guidelines on Management and Disposal of Expired Chemicals, Sections 7 through 10.*

MODULE 8
Laboratory Waste Management
Reference:
 NESREA-SOP-MO
 D08
Revision: Rev. 1.0

Date: 2026
Status: First Edition

8.1 Purpose

This Module establishes the minimum requirements for the management, classification, segregation, treatment, and disposal of laboratory chemical waste generated in chemicals and waste analysis laboratories. It ensures that laboratory waste is handled in a manner that protects the health and safety of laboratory personnel and the public, prevents environmental contamination, and complies with applicable Nigerian environmental legislation. It is a fundamental principle of this Module that laboratories whose analytical work supports the enforcement of environmental law shall themselves be exemplary in their compliance with those same laws.

8.2 Scope

This Module applies to all waste streams generated by the laboratory, including: spent solvents and solvent residues; acid and alkali waste solutions; heavy metal-containing digestion residues and extract solutions; aqueous waste containing trace organic compounds; mercury-containing waste; cyanide-containing waste; sample residues and disposable extraction equipment; WEEE component wastes generated during WEEE analysis; battery electrolyte and component wastes generated during battery waste analysis; and empty chemical containers. Municipal solid waste not contaminated with chemicals is governed by the laboratory's institutional general waste procedures.

8.3 Legal Obligations

WARNING: Discharge of laboratory chemical waste to drains, soakaways, or the environment without treatment is a violation of Section 27 of the NESREA Act and the National Environmental (Sanitation and Wastes Control) Regulations, 2009 (S.I. No. 28 of 2009). It may additionally constitute an offence under the Harmful Waste (Special Criminal Provisions, Etc.) Act. Compliance with this Module is mandatory for every laboratory operating under this Manual.

CROSS-REFERENCE: *For expired laboratory chemicals (reagents, standards, solvents past their expiry date), the National Guidelines on Management and Disposal of Expired Chemicals apply in full. This Module covers waste generated during active analytical operations. The two instruments are complementary and shall be applied concurrently.*

8.4 Waste Classification and Segregation

All laboratory chemical waste shall be classified according to the GHS hazard classification of its primary hazardous component and segregated into the following categories before storage or disposal:

Waste Category	Primary Contents	Container Type	Colour Code / Label
Halogenated solvent waste	Chlorinated solvents (DCM, CHCl ₃ , CCl ₄)	Amber glass carboy, PTFE-lined cap, ≤20 L	Orange: HALOGENATED SOLVENT WASTE

Non-halogenated solvent waste	Hexane, ethyl acetate, acetone, methanol, acetonitrile	Amber glass or HDPE carboy, ≤20 L	Yellow: NON-HALOGENATED SOLVENT WASTE
Acid waste	HNO ₃ , HCl, H ₂ SO ₄ solutions; acid digestion residues	HDPE carboy, ≤20 L	Red: ACID WASTE: pH below 6
Alkali waste	NaOH, KOH solutions; alkali digestion waste	HDPE carboy, ≤20 L	Blue: ALKALI WASTE: pH above 8
Heavy metals aqueous waste	Solutions containing Pb, Cd, Hg, As, Cr(VI)	HDPE carboy, acid-washed, ≤20 L	Purple: HEAVY METALS WASTE
Mercury-containing waste	Any waste containing elemental Hg or Hg compounds; CV-AAS waste	Dedicated glass vessel, double-sealed	Black: MERCURY WASTE: HIGHLY HAZARDOUS
Cyanide waste	Solutions containing CN ⁻ at any concentration	Sealed HDPE, alkaline pH maintained	White: CYANIDE WASTE: KEEP ALKALINE. DO NOT MIX WITH ACIDS
Aqueous trace organic waste	Aqueous solutions with less than 1% organic solvent	HDPE carboy	Green: AQUEOUS ORGANIC WASTE
Solid chemical waste	Used cartridges, contaminated filter paper, contaminated disposable labware	Sealed heavy-duty plastic bag in outer rigid container	Red: SOLID CHEMICAL WASTE
WEEE component waste	Metal-rich fractions, circuit boards, CRT components after analysis	Heavy-duty sealed container, inner plastic liner	Purple/orange: WEEE COMPONENT WASTE: HAZARDOUS
Battery electrolyte waste	Lead-acid sulfuric acid; alkaline KOH	Segregated HDPE containers	Red (acid) or blue (alkali); note electrolyte type on label

	electrolyte; Li-ion fluoride-contain ing electrolyte	per electrolyte type, ≤10 L	
Battery component solid waste	Spent cathode/anode materials, battery casings after disassembly	Sealed HDPE container or heavy-duty bag	Purple: BATTERY WASTE: HAZARDOUS. Note battery type on label

INCOMPATIBILITY RULE: Halogenated and non-halogenated solvent waste shall never be mixed: they require different disposal routes. Acid waste and cyanide waste shall never be mixed: hydrogen cyanide gas is generated. Mercury waste shall remain in a completely separate sealed container. Battery acid waste (lead-acid electrolyte) shall never be mixed with cyanide waste or alkaline battery electrolyte before hazard identification and treatment planning.

8.5 Waste Storage Requirements

110. The laboratory shall designate a specific, ventilated chemical waste storage area separated from analytical bench areas and chemical reagent storage. The waste storage area shall be equipped with secondary containment capable of holding the volume of the largest container plus 10%.
111. Maximum storage periods before transfer to a licensed disposal facility are as follows: all solvent wastes, 90 days; acid and alkali waste, 90 days; heavy metals aqueous waste, 90 days; mercury-containing waste, 90 days (notify NESREA if accumulation exceeds 1 kg); cyanide waste, 30 days; solid chemical waste, 90 days; WEEE component waste, 90 days; battery electrolyte and component waste, 90 days.
112. These periods align with the interim storage periods in Section 8.1 of the National Guidelines on Management and Disposal of Expired Chemicals.

Each laboratory shall conduct an internal waste audit at least once annually. The waste audit shall assess: the types and volumes of waste generated across all analytical operations; the adequacy of current waste segregation, storage, and disposal practices; compliance with the holding periods and disposal routes specified in this Module; the accuracy and completeness of waste records in Form LF-06; and any deviations from this Manual or applicable legal requirements. Waste audit findings shall be documented, reported to the Laboratory Director, and reviewed at the annual Management Review meeting. Where deficiencies are identified, corrective action shall be initiated under Module 6, Section 6.12.

All waste storage areas shall be equipped with secondary containment capable of holding at least 110 per cent of the volume of the largest single container in the storage area. Secondary containment shall be constructed of materials resistant to the chemicals stored. Drains within secondary containment areas shall be sealed or controlled to prevent uncontrolled release. Spill response materials, including absorbents appropriate for chemical classes in use, shall be stored in accessible locations adjacent to all waste storage and handling areas. A documented spill response procedure shall form part of the Laboratory Chemical Hygiene Plan and shall be reviewed in conjunction with emergency response training under Module 10.

8.6 On-Site Treatment Options

8.6.1 Acid and Alkali Neutralisation

Acid and alkali waste streams not containing heavy metals, organics, or cyanide may be neutralised on-site before discharge to drain: slowly add acid waste to a vessel containing excess alkali waste with stirring; monitor pH continuously; when pH is between 6.0 and 9.0 and the waste contains no other hazardous components, discharge to drain with a minimum 50:1 dilution of water. The neutralisation procedure and discharge shall be recorded in Form LF-06. This option is not available for waste containing heavy metals, organic solvents, or cyanide. Lead-acid battery electrolyte shall not be neutralised by this method until tested for dissolved lead; if lead exceeds 0.5 mg/L the waste must go to a licensed contractor.

8.6.2 Cyanide Oxidation

Dilute cyanide waste (CN^- below 100 mg/L) may be treated by alkaline chlorination on-site: adjust pH above 10 with NaOH; slowly add dilute NaOCl with stirring, maintaining alkaline pH throughout; monitor with cyanide indicator paper for complete destruction; confirm residual chlorine removal by sodium metabisulphite addition; discharge treated effluent to drain. All cyanide oxidation shall be performed in a fume hood under supervision of the Laboratory Director or designee.

8.6.3 Treatment Limitations

The following waste streams cannot be treated on-site and must be transferred to a licensed hazardous waste disposal contractor: halogenated solvents; mercury-containing waste; waste containing PCBs or persistent organic pollutants; concentrated heavy metals waste (above 100 mg/L of any regulated metal); biological waste with potential infectious content; and lithium-ion battery electrolyte.

8.7 Licensed Hazardous Waste Disposal

113. The laboratory shall engage a contractor licensed under the National Environmental (Sanitation and Wastes Control) Regulations, 2009 (S.I. No. 28 of 2009). Before engaging any contractor, the laboratory shall: request and review the contractor's current NESREA licence; verify the licence is current; and confirm the contractor can accept each waste category for which disposal is required.
114. Waste transfer shall be documented using the four-copy manifest system in Annex IV of the National Guidelines on Management and Disposal of Expired Chemicals. The laboratory's copy shall be retained for five years.
115. The Disposal Completion Certificate from the contractor shall be retained with the waste transfer manifest as evidence of lawful disposal.

8.8 Documentation

Form LF-06 (Laboratory Waste Disposal Register, Annex G) shall be maintained for all laboratory waste streams, recording: waste category; date container opened; date container closed; volume or weight; disposal route; contractor name and licence number; manifest number; date of disposal; and disposal completion confirmation. Form LF-06 shall be retained for five years.

CROSS-REFERENCE: *For expired laboratory chemicals, see the National Guidelines on Management and Disposal of Expired Chemicals. For chemical spill response, see Module 10. For PPE requirements during waste handling, see Module 7.*

MODULE 9 Data Recording, Reporting, and Result Communication	Reference: NESREA-SOP-MO D09 Revision: Rev. 1.0	Date: 2026 Status: First Edition
---	---	---

9.1 Purpose

This Module establishes the requirements for the recording of raw analytical data, calculation and verification of results, preparation and authorisation of Analytical Result Reports, and communication of results to requesting parties. It specifies the format and minimum content of the Regulatory Analytical Certificate issued under Section 30(4) of the NESREA Act, and establishes the escalation pathway for significant findings, that is, analytical results indicating that a chemical substance is present at a concentration that exceeds or may exceed applicable regulatory thresholds.

9.2 Scope

This Module applies from the point at which raw analytical data is generated by an instrument or by a titrimetric or gravimetric procedure, through to the final authorised report and its submission to the requesting officer or agency. It applies to all sample types and all analytical categories covered by Module 4 of this Manual.

CROSS-REFERENCE: *This Module is the downstream continuation of the result communication process initiated at field collection level under the FMEnv SOPs for Environmental Sample Collection and Management. The Analytical Result Report produced under this Module shall be compatible with the reporting templates in that document.*

9.3 Raw Data Recording Requirements

116. All raw analytical data shall be recorded contemporaneously, that is, at the time the data is generated or read. Raw data shall be recorded in permanent ink in a bound laboratory notebook or on official laboratory worksheets, never in pencil. Errors shall be corrected by crossing through with a single line, writing the correct value, and initialling with the date. Correction fluid shall never be used.
117. Raw data records shall include at minimum: date and time of analysis; instrument identification and calibration status; analyst name; sample LRN; all instrument readings; all blank and calibration standard values; calculations with intermediate values shown; and any observations relevant to sample condition or analytical behaviour.
118. Instrument output in electronic form (chromatograms, spectrograms) shall be retained as raw data, labelled with the LRN, date, and analyst name, and securely filed with the analytical record. Electronic data files shall be backed up to a secondary storage location at the end of each analytical day.
119. Where the laboratory operates a validated LIMS with a complete, uneditable audit trail, electronic data capture constitutes raw data recording and paper backup is not required, provided records are backed up daily.

9.4 Result Calculation and Verification

120. Results shall be calculated from raw data using the equations and conversion factors specified in the relevant Module 4 method section and in supplementary

instrument-specific SOPs. All intermediate calculation steps shall be shown in the laboratory record.

121. Every result shall be independently verified by a second person before reporting. Verification means checking: calculation arithmetic; correct use of the calibration equation; dilution factor application; units; and consistency with expected concentration range for the sample type.
122. For results that will appear on a Regulatory Analytical Certificate under Section 30(4) of the NESREA Act, the verification step is mandatory and shall be documented by the verifier's signature on the calculation sheet.
123. Results below the Limit of Detection shall be reported as "Not Detected: LOD = [value] [unit]". Results between LOD and LOQ shall be reported as "Detected below LOQ: estimated value [X] [unit]". Only results at or above the LOQ shall be reported as quantitative results.

9.5 Analytical Result Report: Standard Format (Form LF-07)

Every analytical result issued by the laboratory shall take the form of a completed Analytical Result Report (Form LF-07, Annex H). The report shall contain the following minimum elements:

- Laboratory name, address, and accreditation status
- Report reference number and date of issue
- Client or requesting officer name, designation, and agency
- Sample description: LRN, field sample identification, matrix, collection date, collection location, collection officer name
- Date of receipt at laboratory; sample condition on receipt
- Chain-of-custody reference numbers
- Date(s) of analysis
- Analytical method(s) applied, with full method reference
- Results table: analyte name; result value with unit; reporting limit (LOQ); applicable regulatory standard or threshold where available; compliance status
- QC summary: CRM result and certified value; matrix spike recovery; duplicate RPD; any QC failures and corrective actions taken
- Sample holding time compliance: confirmation that analysis was completed within the holding time, or disclosure of any exceedance
- Measurement uncertainty where determined, or a statement that uncertainty has not been calculated
- Qualifications and limitations (matrix effects, interferences, analytical deviations)
- Name, signature, and designation of Responsible Analyst
- Name, signature, and designation of verifier
- Name, signature, and designation of Laboratory Director authorising release
- Statement: "This report relates only to the items tested and represents the results obtained at the time of analysis."

REGULATORY ANALYTICAL CERTIFICATE: For samples collected under Section 30(1) of the NESREA Act for regulatory purposes, the Analytical Result Report shall additionally carry the statement: "This certificate is issued pursuant to Section 30(4) of the National Environmental Standards and Regulations Enforcement Agency (Establishment) Act, 2007 (Amendment, 2018). The analysis was conducted at [Laboratory Name] in accordance with the NESREA Standard Operating Procedure Manual for Laboratories." The certificate shall be signed by the Laboratory Director.

9.6 Escalation Pathway for Significant Findings

A significant finding is an analytical result indicating that the concentration of a hazardous substance exceeds or is likely to exceed the applicable regulatory threshold. The following escalation pathway shall be activated for all such findings:

124. The Responsible Analyst shall immediately notify the Quality Officer and the Laboratory Director upon generating a preliminary result that may constitute a significant finding, before the result is finalised.
125. The Laboratory Director shall review the raw data and QC records and confirm that the result is analytically valid and not attributable to contamination, calibration error, or procedural deviation.
126. Where the significant finding is confirmed, the Laboratory Director shall release the Analytical Result Report and simultaneously notify the requesting NESREA officer in writing (electronic notification is acceptable as initial contact, to be followed by the signed report within 24 hours).
127. Where the finding may pose an immediate threat to human health, including but not limited to heavy metals in drinking water above WHO guideline values, cyanide or mercury at acutely toxic concentrations in a community environment, or evidence of a chemical release incident, the Laboratory Director shall additionally notify the NESREA Environmental Emergency Hotline within two hours of confirmation, and, where human health is implicated, the Nigeria Centre for Disease Control and Prevention consistent with IHR reporting obligations.
128. All notifications under this escalation pathway shall be documented in the laboratory record and cross-referenced in the Analytical Result Report.

9.7 Amendment and Withdrawal of Reports

Where an error is discovered in a released report, or where additional analysis has materially changed a reported result, the Laboratory Director shall issue an Amended Report. The Amended Report shall: carry the same reference number with the suffix "/A[number]"; state clearly at the top "AMENDED REPORT: supersedes Report [reference] of [date]"; specify the nature of the amendment; and be signed by the Laboratory Director. The original report shall be retained with a notation referencing the amendment. The recipient of the original report shall be notified immediately and provided with the Amended Report.

9.8 Record Retention

All raw data records, calculation sheets, QC batch records, instrument output, and Analytical Result Reports shall be retained in accordance with Section 1.9 of Module 1 (minimum five years, or until conclusion of legal proceedings where the records are relevant). Records shall be protected from loss, damage, and unauthorised access.

CROSS-REFERENCE: *For chain-of-custody records accompanying results, see Module 2. For QC data incorporated in reports, see Module 6. For escalation of chemical incident findings, see Module 10 and Section 27 of the NESREA Act. For inter-laboratory reports, see Module 11.*

MODULE 10
Emergency Response for Laboratory Incidents
Reference:
 NESREA-SOP-MO
 D10
Revision: Rev. 1.0

Date: 2026
Status: First Edition

10.1 Purpose

This Module establishes the laboratory-specific emergency response procedures for chemical spill, fire, toxic vapour release, personal chemical exposure, and loss or theft of hazardous substances. It specifies the notification requirements for significant laboratory incidents, including the obligation to notify NESREA within two hours of any uncontrolled release of a hazardous substance from the laboratory to the environment. It is designed to minimise harm to laboratory personnel, the public, and the environment arising from laboratory incidents, and to ensure that such incidents are managed in a controlled, documented, and legally compliant manner.

10.2 Scope

This Module applies to all emergency situations arising within the laboratory or its immediate environment, including: chemical spills inside and outside fume hoods; fires involving flammable solvents or other flammable materials; release of toxic gases or vapours; personal injury from chemical exposure (skin contact, eye contact, inhalation, ingestion); equipment failure with potential safety consequence; battery thermal runaway events; and loss, theft, or unauthorised access to controlled or highly toxic substances.

CROSS-REFERENCE: *This Module is aligned with the emergency response provisions in the FMEnv SOPs for Environmental Sample Collection and Management and with the relevant provisions of the National Guidelines on Management and Disposal of Expired Chemicals. The NESREA Environmental Emergency Hotline referenced in this Module is the same notification channel referenced in both of those instruments.*

10.3 Emergency Contact Information

The following contact information shall be prominently posted in the laboratory, on all entry doors, and in the first aid area. Details shall be reviewed and updated at least once per year.

Contact	Number/Channel	When to Use	Available
NESREA Environmental Emergency Hotline	[+23491539931 91]	Any uncontrolled release of hazardous substance from laboratory to environment	24/7
Nigeria Emergency Management Agency (NEMA)	0800-225-5636 2	Large-scale incidents; evacuation required	24/7
Nigeria Police Force (Emergency)	199	Theft, loss, or unauthorised access to	24/7

		controlled substances	
Federal Fire Service	193	Laboratory fire beyond first-response control	24/7
NCDC Emergency Operations Centre	0800-970000-10	Chemical exposure with public health implication; human casualty	24/7
Laboratory Director (personal)	[Insert]	All incidents requiring management decision	Working hours; on-call
Chemical Hygiene Officer (personal)	[Insert]	All safety incidents	Working hours
Nearest hospital emergency unit	[Insert local number]	Personal injury requiring medical attention	24/7

10.4 General Incident Response Principles

- PEOPLE FIRST: the safety of laboratory personnel takes absolute priority over the protection of equipment, samples, or analytical data.
- ALERT BEFORE ACTING ALONE: immediately alert a colleague of any significant incident. Never deal with a serious spill, fire, or exposure alone.
- USE APPROPRIATE PPE: no person shall approach a chemical incident without appropriate PPE for the hazard presented. If adequate PPE is not available, evacuate and call for specialist response.
- REFER TO THIS MODULE: if unsure of the correct response, evacuate the immediate area and consult the Chemical Hygiene Officer or Laboratory Director.

10.5 Chemical Spill Response Procedures

10.5.1 Minor Spill (less than 1 litre; low hazard; confined to bench or inside fume hood)

129. Alert a colleague.
130. Don appropriate PPE (nitrile gloves, safety spectacles, lab coat; face shield if corrosive).
131. Confine the spill using absorbent material from the spill kit (absorbent pads, dry sand or vermiculite, scoop, plastic bag, gloves).

132. For acid spills: apply sodium bicarbonate then absorbent. For alkali spills: apply citric acid or boric acid then absorbent. For solvent spills: apply dry absorbent only (no water); eliminate ignition sources; ventilate area.
133. Collect contaminated absorbent material in a labelled plastic bag and dispose as chemical waste per Module 8.
134. Clean the area with appropriate rinse solvent; decontaminate with dilute detergent.
135. Document the incident in Form LF-08 (Laboratory Incident Report, Annex I).

10.5.2 Major Spill (more than 1 litre; or any quantity of high-hazard substance: HF, HCN, concentrated mercury compounds, PCBs, or Li-ion battery electrolyte)

136. Alert everyone in the laboratory immediately. Direct all non-essential personnel to evacuate the affected area.
137. If safe to do so, contain the source (close fume hood sash, close solvent container, close drain valves).
138. Evacuate the laboratory. Do not re-enter until the Chemical Hygiene Officer has assessed whether conditions are safe.
139. Notify the Laboratory Director and Chemical Hygiene Officer immediately.
140. For HF spills: immediately apply calcium gluconate gel 2.5% to any exposed skin, even if no pain is felt; call for medical assistance.
141. For mercury spills: do not use a vacuum cleaner (disperses mercury vapour). Use a mercury spill kit (zinc dust, sulphur powder, or commercial amalgamation material). Seal the area and ventilate. Measure ambient mercury vapour with detector before re-entry.
142. For Li-ion battery electrolyte spills: ventilate immediately; contain using dry absorbent; do not allow contact with water (HF generation from LiPF_6 hydrolysis); activate HF protocol if burning or irritation occurs.
143. Notify the NESREA Environmental Emergency Hotline within two hours if any hazardous substance has been released to the environment (drainage system, soil outside building, or air beyond the building).
144. Complete Form LF-08 within 48 hours of the incident.

10.6 Fire Response

145. Activate the fire alarm and direct all personnel to evacuate via the nearest safe exit.
146. For small fires (waste basket, paper): use CO_2 or dry powder extinguisher. Apply from a safe distance; if the fire is not extinguished within 30 seconds, evacuate.
147. For solvent fires: evacuate immediately; use CO_2 or dry chemical extinguisher only; never water on solvent fires. Close fume hood sashes and laboratory doors to limit oxygen supply.
148. For lithium battery fires: do not use water. Use Class D extinguisher or dry sand. Evacuate and call the fire service.
149. Call the Federal Fire Service (193) and the Laboratory Director. Do not re-enter the building until cleared by the fire service.
150. Notify the NESREA Environmental Emergency Hotline if the fire involved hazardous chemicals or if there is any environmental release.

10.7 Personal Chemical Exposure Response

10.7.1 Skin Contact

151. Remove contaminated clothing immediately.
152. Flush the affected area copiously with water for a minimum of 15 minutes at the safety shower or sink.
153. For HF skin contact: apply calcium gluconate gel 2.5% immediately and continue applying during water flush; seek immediate medical attention.
154. Seek medical attention for all acid, alkali, or toxic substance skin contacts.

10.7.2 Eye Contact

155. Immediately go to the nearest eyewash station.
156. Hold eyelids open and flush eyes with a continuous stream of water for a minimum of 15 minutes, rolling eyes in all directions.
157. Remove contact lenses only after initial flushing.
158. Seek immediate medical attention.

10.7.3 Inhalation of Toxic Vapour

159. Move the affected person immediately to fresh air. Do not re-enter a vapour-contaminated area without breathing protection.
160. If the person is unconscious or breathing is impaired, call emergency services immediately.
161. For cyanide inhalation: administer hydroxocobalamin injection if trained and kit is available; seek immediate emergency medical attention.

10.8 Incident Notification and Reporting

162. NESREA shall be notified within two hours of: any uncontrolled release of a hazardous substance to the environment; fire or explosion involving hazardous chemicals; human exposure resulting in medical treatment or hospitalisation; theft or unexplained loss of controlled or highly toxic substances; and any transportation incident during off-site waste transfer.
163. A written Incident Report (Form LF-08, Annex I) shall be completed within 48 hours of any incident, including minor ones. The report shall document: date, time, and location; description of incident; substances and quantities involved; personnel affected; immediate response actions; whether NESREA was notified; and corrective and preventive actions taken or planned.
164. All Incident Reports shall be retained for five years and reviewed at the annual Management Review (Module 1, Section 1.7 of Module 1) for systemic patterns requiring preventive action.

CROSS-REFERENCE: For waste generated as a result of a laboratory incident, see Module 8. For PPE requirements during incident response, see Module 7. For formal NESREA notification requirements, see also Section 13 of the National Guidelines on Management and Disposal of Expired Chemicals.

MODULE 11

Inter-Laboratory Referral and Network Protocols

Reference:
NESREA-SOP-MO
D11
Revision: Rev. 1.0

Date: 2026
Status: First Edition

11.1 Purpose

This Module establishes the formal protocols governing the referral of samples, analytical tasks, and quality control comparisons between laboratories within Nigeria's analytical laboratory network, and between domestic laboratories and designated international accredited reference facilities. It operationalises the recommendation of the national laboratory mapping exercise for a formal laboratory network, assigns roles to participating laboratories based on assessed capability, and specifies the documentation, chain-of-custody, and reporting requirements for inter-laboratory work.

11.2 Scope

This Module applies to: any transfer of a physical sample from one laboratory to another for analysis; any analytical task commissioned externally; inter-laboratory comparison exercises and proficiency testing organised through the network; and the incorporation of results received from external laboratories into reports issued under this Manual.

11.3 The National Analytical Laboratory Network

NESREA, in coordination with NAFDAC, SON, the Federal Ministry of Agriculture and Food Security, and research institute counterparts, shall establish a National Analytical Laboratory Network comprising all laboratories covered by this Manual. The Network shall be structured around three tiers:

11.3.1 Tier 1: National Reference Laboratories

Tier 1 laboratories provide the highest level of analytical capability, serve as technical references for other network laboratories, lead proficiency testing schemes, and are the first point of referral for complex samples. Based on the current national assessment, designated Tier 1 laboratories include: NESREA National Environmental Reference Laboratories (Kano and Port Harcourt); NAFDAC Central Laboratory (Lagos) covering metals, OCPs, PCBs, PAHs, pharmaceuticals, and food contaminants; the Dr. Olukoya Central Research and Reference Laboratory at the University of Lagos; the Nigerian Institute of Medical Research Toxicology and Drug Discovery Laboratory (Lagos) for the toxicological suite and biological matrices; the National Institute for Pharmaceutical Research and Development (FCT) for pharmaceutical chemicals and heavy metals in biologicals; and the Global Research Centre Laboratory, University of Ibadan (Oyo) for POPs in environmental matrices.

11.3.2 Tier 2: Regional Specialist Laboratories

Tier 2 laboratories provide full analytical service for their geopolitical zone and one or more analyte specialities, serve as the first point of referral for Tier 3 laboratories in their zone, and participate in the network PT scheme. Designated Tier 2 laboratories include: NARICT Analytical Services Laboratory (Kaduna); Federal University Wukari Central Laboratory (Taraba); Bayero University Kano Instrumentation and Analytical Laboratory (Kano); ABU Multi-User Science Research Laboratory (Kaduna); and University of Ibadan Multi-disciplinary Laboratory (Oyo).

11.3.3 Tier 3: Routine Analytical Laboratories

Tier 3 laboratories conduct routine analysis in their sector and location, refer complex samples to Tier 1 or Tier 2, and receive technical support from higher-tier laboratories in their zone. NESREA zonal laboratory facilities and sector-specific laboratories not covered by the above tiers constitute Tier 3.

11.3.4 International Accredited Reference Facilities

For analytical capabilities that cannot be met domestically (see Module 4, Section 4.14), NESREA shall maintain a current list of designated international accredited laboratories to which referral samples may be sent. These facilities shall hold ISO/IEC 17025 accreditation for the specific analyses to be performed and shall carry documented experience in hazardous waste monitoring and persistent organic pollutant analysis as required under Nigeria's international chemical and waste management obligations. The list shall be updated annually and made available to all Network laboratories.

11.4 Referral Procedure

11.4.1 Triggering Events for Referral

A laboratory shall initiate a referral when any of the following conditions apply:

- The required analytical capability does not exist at the laboratory (instrumentation or validated method absent).
- The required capability has been temporarily lost due to instrument failure and the sample holding time does not permit waiting for repair.
- A result requires confirmation by a second method or by a higher-capability instrument.
- The analysis requires an international accredited facility, such as HRMS for dioxins or biological matrix mercury speciation.
- Human biological samples with dual chemical and biological hazard require a Class II BSC environment not available at the originating laboratory.
- A request for independent analysis is received from a regulated entity or legal party in connection with proceedings.

11.4.2 Referral Steps

165. The Laboratory Director of the originating laboratory shall authorise the referral in writing, specifying: the sample LRN; the analysis required; the reason for referral; and the name of the receiving laboratory.
166. The originating laboratory shall complete Form LF-09 (Inter-Laboratory Referral Form, Annex J) in triplicate: one copy for the originating laboratory file; one copy accompanying the sample; one copy for the receiving laboratory file.
167. Sub-aliquots rather than the primary sample shall be transferred where sample volume permits. The sample shall be prepared for transfer in accordance with Module 3 storage requirements.
168. A Chain-of-Custody Continuation Sheet shall document the custody transfer from originating laboratory to transport agent to receiving laboratory. Both Laboratory Directors shall sign at the point of transfer.
169. The receiving laboratory shall confirm receipt in writing, noting arrival condition, on Form LF-09, within 24 hours of receipt.

170. Results shall be returned to the originating laboratory in the form of an Analytical Result Report conforming to Module 9 requirements, signed by the Laboratory Director of the receiving laboratory.
171. The originating laboratory shall incorporate referred results into its final Analytical Result Report with explicit attribution, for example: "[Analyte] was analysed at [Receiving Laboratory Name, accreditation number] under referral reference [Form LF-09 number]. Full results and method details are attached."

11.4.3 International Referral: Additional Requirements

172. All international referrals shall be authorised by the NESREA Director-General/CEO or a formally delegated representative, recognising the export control implications of sending samples containing regulated substances across international borders.
173. The originating laboratory shall verify that the proposed sample transfer complies with applicable requirements for transboundary movement of hazardous waste and any applicable restrictions under chemical safety or arms control instruments.
174. Samples transferred internationally shall be accompanied by complete SDS documentation, a signed certification of non-commercial research nature, and a copy of the NESREA export authorisation. The sending analyst shall comply with IATA/ICAO dangerous goods regulations for air transport of laboratory samples.

11.5 Network Quality Assurance Activities

11.5.1 National Proficiency Testing Scheme

NESREA, in coordination with NAFDAC and the Institute of Public Analysts of Nigeria (IPAN), shall organise or facilitate at least one national inter-laboratory PT exercise per year covering the priority analyte categories in Module 4. Results shall be evaluated by z-scores (Module 6, Section 6.9). PT performance shall inform tier assignments and shall be reviewed at the annual Management Review of each laboratory.

11.5.2 Method Harmonisation Workshops

NESREA shall convene at least one technical workshop per year for all Network laboratories to review: implementation experience with this Manual; QA/QC performance trends across the Network; new analytical requirements arising from international obligations; training needs identified from PT performance; and proposed revisions to Modules of this Manual.

11.5.3 On-Site Technical Assistance

Tier 1 and Tier 2 laboratories are encouraged to provide on-site technical assistance to Tier 3 laboratories in their geographic zone, on request, including method demonstration, instrument performance review, QMS audit support, and mentoring toward ISO/IEC 17025 accreditation. NESREA shall coordinate these visits and recognise assisting laboratories in annual performance reporting.

11.6 Memoranda of Understanding

Referral relationships between Network laboratories shall be formalised through Memoranda of Understanding signed by the heads of the relevant institutions. These shall specify: the analytes and matrices covered by the referral arrangement; agreed turnaround times; cost-sharing arrangements where applicable; data ownership and confidentiality provisions; and the reporting chain for regulatory samples. NESREA shall maintain a registry of all MoUs in force.

11.7 Documentation

Form LF-09 (Inter-Laboratory Referral Form, Annex J) shall be completed for all referrals. Originating laboratories shall retain copies of all referral forms and results as permanent analytical records subject to the retention requirements in Module 1, Section 1.9.

CROSS-REFERENCE: *For analytical capability gaps that drive referral decisions, see Module 4, Section 4.14. For chain-of-custody requirements during sample transfer, see Module 2. For sample storage requirements during transport, see Module 3. For incorporation of referral results into final reports, see Module 9, Section 9.6.*

REFERENCES

A. Nigerian Legislation and Regulations

- National Environmental Standards and Regulations Enforcement Agency (Establishment) Act, 2007 (Amendment, 2018) (Act No. 25 of 2007).
- Factories Act, CAP F1 Laws of the Federation of Nigeria 2004.
- Harmful Waste (Special Criminal Provisions, Etc.) Act, CAP H1 Laws of the Federation of Nigeria 2004.
- National Environmental (Chemicals, Pharmaceuticals, Soap and Detergent Manufacturing Industries) Regulations, S. I. No. 36, 2009.
- National Environmental (Chemicals and Pesticides) Regulations 2023 B4209 - 262
- National Environmental (Sanitation and Wastes Control) Regulations, 2009 (S.I. No. 28 of 2009).
- National Environmental (Electrical/Electronic Sector) Regulations, 2011 (S.I. No. 23 of 2011).
- National Environmental (Domestic and Industrial Plastic, Rubber and Foam Sector) Regulations, S.I. No. 17, 2011
- National Environmental (Plastic Waste Control) Regulations, 2026B145-215.
- NESREA Harmonised Guidelines and Standards for Water Quality Management in Nigeria, 2011.
- National Environmental (Mining and Processing of Coal, Ores and Industrial Minerals) Regulations, 2009.
- National Environmental (Battery Control) Regulations, 2024.
- National Environmental (Polychlorinated Biphenyls (PCBs) Control and Disposal) Regulations, 2020 B249-298.
- National Environmental (Healthcare Waste Control) Regulations, B2565-2592, 2021.
- Nigeria Centre for Disease Control and Prevention Act, 2018 (Act No. 13 of 2018).
- Food and Drugs Act, CAP F32 Laws of the Federation of Nigeria 2004.
- Standards Organisation of Nigeria Act, 2015 (Act No. 14 of 2015).

B. National Guidelines and Technical Documents

- Standard Operating Procedures for Environmental Sample Collection and Management in Nigeria. Federal Ministry of Environment (current edition).
- National Guidelines on Management and Disposal of Expired Chemicals in Nigeria. National Environmental Standards and Regulations Enforcement Agency, 2026.
- Research and Gap Analysis Report. NESREA/I&ED/002/2026. National Environmental Standards and Regulations Enforcement Agency, 2026.
- Nigerian Industrial Standard NIS:2026 on Environmentally Sound Management of Chemicals. Standards Organisation of Nigeria.
- Global Framework on Chemicals 2023 to 2050. United Nations Environment Programme, Geneva, 2023.
- NAFDAC Regulations on food contaminants, packaging materials, and pharmaceutical quality control (current editions). National Agency for Food and Drugs Administration and Control.
- EPR Policy Framework for Nigeria. National Environmental Standards and Regulations Enforcement Agency (working document), 2025.

- NARICT Analytical Services Reference Materials Catalogue. National Research Institute for Chemical Technology, Zaria.

C. International Standards: ISO/IEC and IEC

- ISO/IEC 17025:2017. General requirements for the competence of testing and calibration laboratories. International Organization for Standardization.
- ISO 5667-3:2018. Water quality ; Sampling: Guidance on the preservation and handling of water samples.
- ISO 5667-15:2009. Water quality ; Sampling: Guidance on the preservation and handling of sludge and sediment samples.
- ISO 5814:2012. Water quality ; Determination of dissolved oxygen.
- ISO 5815-1:2019. Water quality ; Determination of biochemical oxygen demand after n days (BOD_n).
- ISO 7027-1:2016. Water quality ; Determination of turbidity.
- ISO 7888:1985. Water quality ; Determination of electrical conductivity.
- ISO 8655 series. Piston-operated volumetric apparatus: calibration methods.
- ISO 10523:2008. Water quality ; Determination of pH.
- ISO 11885:2007. Water quality ; Determination of selected elements by inductively coupled plasma optical emission spectrometry.
- ISO 11923:1997. Water quality ; Determination of suspended solids.
- ISO 15586:2003. Water quality ; Determination of trace elements using atomic absorption spectrometry with graphite furnace.
- ISO 15705:2002. Water quality ; Determination of chemical oxygen demand index.
- ISO 17852:2006. Water quality ; Determination of mercury.
- IEC 62321 series. Electrotechnical products: determination of levels of six regulated substances. International Electrotechnical Commission.

D. USEPA Reference Methods

- United States Environmental Protection Agency (USEPA). SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Applicable methods include: 200.7, 200.2, 3050B, 3051A, 3540C, 3545A, 3550C, 5030C, 5035A, 7000B, 7470A, 7471B, 8015D, 8081A, 8082A, 8141B, 8260C, 8270D, 8310, 8321B, 1664A/B.
- USEPA. Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act. 40 CFR Part 136, Appendix B (method detection limit determination procedure).
- USEPA. Regional Screening Levels. (For reference values where Nigerian limits are not established.)

E. WHO and Codex Alimentarius Documents

- World Health Organization. Laboratory Quality Management System: Handbook. 2nd ed. Geneva: WHO, 2011.
- World Health Organization. Laboratory Biosafety Manual. 4th ed. Geneva: WHO, 2020.
- World Health Organization. Guidelines for Drinking-Water Quality. 4th ed. Geneva: WHO, 2017.

- Codex Alimentarius Commission. Maximum Residue Limits for pesticides in food. Current edition. FAO/WHO.
- European Committee for Standardization. EN 15662:2018. Foods of plant origin: determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction and dispersive SPE (QuEChERS).

F. International Technical Guidelines on Chemicals and Waste Management

- United Nations Environment Programme (UNEP). Guidance on the Global Monitoring Plan for Persistent Organic Pollutants: Analytical Methods for POPs in Environmental Matrices. Geneva: UNEP, 2015.
- UNEP. Technical guidelines for the environmentally sound management of waste lead-acid batteries. Geneva: UNEP.
- UNEP. Global Mercury Partnership: Guidance on Mercury in Products and Waste. Geneva: UNEP.
- International Air Transport Association (IATA). Dangerous Goods Regulations. Current edition. Montreal: IATA.
- Technical guidelines on the environmentally sound management of hazardous waste under international waste management conventions (current versions). Geneva: Secretariat.
- Organisation for the Prohibition of Chemical Weapons (OPCW). Approved methods for the analysis of chemicals. Technical Secretariat, The Hague: OPCW.
- NIST Standard Reference Materials Programme. Certificate of Analysis documents for applicable SRMs. National Institute of Standards and Technology, USA.

C. International Standards

175. 1. ISO/IEC 17025:2017. General Requirements for the Competence of Testing and Calibration Laboratories. International Organization for Standardization, Geneva.
176. 2. ISO 5667-3:2018. Water Quality Sampling: Guidance on Preservation and Handling of Samples. International Organization for Standardization, Geneva.
177. 3. ISO 5667-15:2009. Water Quality Sampling: Guidance on Preservation and Handling of Sludge. International Organization for Standardization, Geneva.
178. 4. ISO 11885:2007. Water Quality: Determination of Selected Elements by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). International Organization for Standardization, Geneva.
179. 5. ISO 15586:2003. Water Quality: Determination of Trace Elements Using Atomic Absorption Spectrometry with Graphite Furnace. International Organization for Standardization, Geneva.
180. 6. ISO 17852:2006. Water Quality: Determination of Mercury by Atomic Fluorescence Spectrometry. International Organization for Standardization, Geneva.
181. 7. ISO 10523. Water Quality: Determination of pH. International Organization for Standardization, Geneva.
182. 8. ISO 15705. Water Quality: Determination of Chemical Oxygen Demand by the Small-Scale Sealed-Tube Method. International Organization for Standardization, Geneva.
183. 9. ISO 5815-1. Water Quality: Determination of Biochemical Oxygen Demand after 5 Days. International Organization for Standardization, Geneva.

184. 10. IEC 62321 series. Electrotechnical Products: Determination of Levels of Restricted Substances. International Electrotechnical Commission, Geneva.
185. 11. WHO Laboratory Quality Management System Handbook (2nd edition). World Health Organization, Geneva, 2011.
186. 12. WHO Laboratory Biosafety Manual, 4th Edition. World Health Organization, Geneva, 2020.
187. 13. GHS: Globally Harmonised System of Classification and Labelling of Chemicals (9th revised edition). United Nations, New York and Geneva, 2021.

D. USEPA Reference Methods

188. 1. USEPA Method 200.7. Determination of Metals and Trace Elements in Water and Wastes by ICP-OES. United States Environmental Protection Agency, Cincinnati, Ohio.
189. 2. USEPA Method 7000B. Flame Atomic Absorption Spectrometry. United States Environmental Protection Agency.
190. 3. USEPA Method 7470A. Mercury in Liquid Waste (Manual Cold-Vapour Technique). United States Environmental Protection Agency.
191. 4. USEPA Method 7471B. Mercury in Solid or Semisolid Waste (Manual Cold-Vapour Technique). United States Environmental Protection Agency.
192. 5. USEPA Method 3050B. Acid Digestion of Sediments, Sludges, and Soils. United States Environmental Protection Agency.
193. 6. USEPA Method 3051A. Microwave Assisted Acid Digestion of Sediments, Sludges, and Oils. United States Environmental Protection Agency.
194. 7. USEPA Method 8081A. Organochlorine Pesticides by Gas Chromatography. United States Environmental Protection Agency.
195. 8. USEPA Method 8082A. Polychlorinated Biphenyls (PCBs) by Gas Chromatography. United States Environmental Protection Agency.
196. 9. USEPA Method 8270D. Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry. United States Environmental Protection Agency.
197. 10. USEPA Method 8141B. Organophosphorus Compounds by Gas Chromatography. United States Environmental Protection Agency.
198. 11. USEPA Method 8260C. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry. United States Environmental Protection Agency.
199. 12. USEPA Method 8015D. Nonhalogenated Organics Using GC/FID. United States Environmental Protection Agency.
200. 13. USEPA Method 8310. Polycyclic Aromatic Hydrocarbons. United States Environmental Protection Agency.
201. 14. USEPA Method 3540C. Soxhlet Extraction. United States Environmental Protection Agency.
202. 15. USEPA Method 3545A. Pressurized Fluid Extraction. United States Environmental Protection Agency.
203. 16. USEPA Method 3550C. Ultrasonic Extraction. United States Environmental Protection Agency.

204. 17. USEPA Method 1664A/B. N-Hexane Extractable Material and Silica Gel Treated N-Hexane Extractable Material by Extraction and Gravimetry. United States Environmental Protection Agency.
205. 18. USEPA Method 40 CFR Part 136, Appendix B. Definition and Procedure for the Determination of the Method Detection Limit. United States Environmental Protection Agency.

ANNEX A Standard SOP Template

Every Standard Operating Procedure developed under or supplementary to this Manual shall follow the structure below. Laboratories shall use this template when preparing laboratory-specific supplementary SOPs.

Section	Required Content
Header Block	SOP Title; Reference Number; Version; Date of Issue; Status; Next Review Date; Author; Approver signature
1. Purpose	One paragraph describing what the SOP governs and why
2. Scope	Define who it applies to, which samples/matrices/instruments
3. Regulatory/Standards Basis	List all applicable legislation, regulations, and international standards
4. Definitions	Define terms specific to this SOP not already defined in the Manual
5. Responsibilities	State who is responsible for each step
6. Materials and Equipment	List all reagents, standards, equipment, forms required
7. Procedure	Numbered, sequential steps with sufficient detail for a competent technician to follow without additional instruction
8. QA/QC Requirements	Specify QC samples, acceptance criteria, and corrective actions per Module 6
9. Safety Precautions	Specific hazards and PPE for this procedure per Module 7
10. Documentation and Records	Specify which forms to complete; where to file; retention period
11. Cross-References	List all Manual modules and other SOPs referenced by this SOP
12. Revision History	Table: Version Date Summary of changes Author Approver

ANNEX B Laboratory Sample Receipt Register (Form LF-01)

Field	Entry
Laboratory Name and Code	
Date of Receipt	
Time of Receipt	
Laboratory Reference Number (LRN)	
Field COC Reference Number	
Submitting Officer: Name, Designation, Agency	
Sample Type / Matrix	
Sample Collection Date (from field label)	
Sample Collection Location	
Number of Containers Received	
Container Type (glass / HDPE / other)	
Container Condition (intact / damaged / seal broken)	
Visible Characteristics (colour, odour, turbidity, phase)	
Requested Analyses	
Any Sample Integrity Concerns (Y/N): if Y, describe	
Action Taken on Integrity Concern	
Accepted / Rejected (with reason if rejected)	
Aliquots Prepared (LRN-A, LRN-B, etc.)	
Assigned Analyst	
Storage Location	

Reception Officer: Name, Signature, Date	
---	--

ANNEX C Chain-of-Custody Record (Form LF-02)

The Laboratory Chain-of-Custody Continuation Sheet documents internal custody transfers within the laboratory. It shall accompany the sample at all times from receipt to disposal.

Transfer No.	From (Name/Designation)	To (Name/Designation)	Date and Time	Signature

LRN: _____ Field COC No.: _____ Final
Disposition: _____

ANNEX D Sample Storage Log (Form LF-03)

Field	Entry
Laboratory Name	
Month / Year	
Storage Unit ID (Fridge/Freezer number)	
Target Temperature	

Temperature Record:

Date	AM Temp (°C)	PM Temp (°C)	In Spec? (Y/N)	Action Taken (if out of spec)	Initials

Sample Storage Index (LRN to Storage Location):

LRN	Matrix	Storage Location	Date Placed / Removed

ANNEX E Equipment Calibration and Maintenance Log (Form LF-04)

Field	Entry
Equipment Identification Number	
Equipment Description / Model / Serial No.	
Activity Type (Calibration / PM / SST / Repair / Return-to-Service)	
Date of Activity	
Description of Activity Performed	
Standards / Reagents Used (with lot numbers)	
Results / Observations	
Acceptance Criterion Met? (Y/N)	
Corrective Action (if criterion not met)	
Instrument Status after Activity (Operational / Out of Service)	
Next Calibration / PM Due Date	
Performed By: Name, Signature	
Reviewed By (Quality Officer): Name, Signature	

ANNEX F Analytical QC Batch Record (Form LF-05)

Field	Entry
Batch Identification Number	
Date of Analysis	
Instrument ID	
Analytical Method Reference	
Analyst Name	
Matrix	
Analyte(s)	

QC Results:

QC Type	Expected Value	Measured Value	Acceptance Criterion	Pass/Fail	Corrective Action
Method Blank	<LOD		Conc < 10% of lowest sample		
Calibration Verification (CVS)			±10% of expected		
Certified Reference Material			Within cert. range or ±15%		
Matrix Spike (MS)			70 to 130% recovery		
Matrix Spike Duplicate (MSD)			RPD ≤ 20%		
Laboratory Duplicate			RPD ≤ 20%		
Internal Standard Recovery			60 to 140%		

Overall Batch Status: PASS ☐ FAIL ☐ Analyst: _____ QO
 Review: _____ Date: _____

ANNEX G Laboratory Waste Disposal Register (Form LF-06)

To be maintained for all laboratory chemical waste streams. Retain for five years.

No.	Waste Category	Container Start Date	Volume/Wt.	Disposal Route	Contractor / Manifest No.	Disposal Date / Confirmation

ANNEX H Analytical Result Report (Form LF-07)

Field	Entry
Report Reference Number	
Date of Issue	
Laboratory Name, Address, Accreditation Status	
Client / Requesting Officer: Name, Designation, Agency, Contact	
Laboratory Reference Number (LRN)	
Field Sample ID	
Sample Matrix	
Collection Date	
Collection Location	
Date of Receipt at Laboratory	
Date(s) of Analysis	
Sample Condition on Receipt	
Field COC No.	
Laboratory COC No.	
Analytical Method(s) Applied (full reference)	
Holding Time Compliance (Y/N; if N, explain)	

Results Table:

Analyte	Result	Unit	Reporting Limit (LOQ)	Regulatory Std / Threshold	Compliance Status

QC Summary: CRM: _____ (Certified: _____); MS Recovery: _____%; Duplicate RPD: _____%

Qualifications _____ / Limitations: _____

Responsible Analyst: _____ | Signature: _____ |
 Date: _____

Verified by (QO/Senior Analyst): _____ | Signature: _____
 _____ | Date: _____

Authorised by (Laboratory Director): _____ | Signature: _____
 _____ | Date: _____

This report relates only to the items tested and represents the results obtained at the time of analysis. For regulatory enforcement samples: "This certificate is issued pursuant to Section 30(4) of the NESREA Act, 2007 (Act No. 25 of 2007), conducted in accordance with NESREA Guidelines on Management and Disposal of Expired and Obsolete Chemicals in Nigeria."

ANNEX I Laboratory Incident Report (Form LF-08)

Field	Entry
Incident Reference Number	
Date and Time of Incident	
Location within Laboratory	
Type of Incident (spill / fire / exposure / release / loss/theft / equipment failure / battery event)	
Substance(s) Involved	
Estimated Quantity Involved	
Description of Incident (what happened, sequence of events)	
Immediate Response Actions Taken	
Persons Affected (name, nature of exposure/injury, first aid / medical treatment)	
Environmental Release? (Y/N): if Y, describe pathway and estimated quantity	
NESREA Notified? (Y/N): if Y, time of notification and name of contact	
NCDC/State Health Authority Notified? (Y/N: if human health implication)	
Root Cause Analysis	
Corrective Actions Implemented	
Preventive Actions Planned	
Report Completed By: Name, Designation, Signature, Date	
Reviewed by Laboratory Director: Signature, Date	

ANNEX J Inter-Laboratory Referral Form (Form LF-09)

Field	Entry
Referral Reference Number	
Date of Referral	
Originating Laboratory: Name, Address, Contact, Laboratory Director	
Receiving Laboratory: Name, Address, Contact, Laboratory Director	
Sample LRN (Originating Laboratory)	
Sample Type / Matrix	
Analysis Requested (analyte, method, LOQ required)	
Reason for Referral	
NESREA Authorisation (name of authorising officer, date): required for international referral	
Sample Condition at Dispatch (temperature, container, seal status)	
Dispatch Date and Method (courier, in-person, etc.)	
Expected Turnaround Time	
— RECEIVING LABORATORY SECTION —	
Date and Time of Receipt at Receiving Laboratory	
Sample Condition on Receipt	

Discrepancies from Dispatch Condition	
Analysis Commenced Date	
Analysis Completed Date	
Results Report Reference Number	
Receiving Laboratory Director: Signature, Date	
— ORIGINATING LABORATORY ACKNOWLEDGEMENT —	
Results Received Date	
Incorporated in Report No.	
Originating Laboratory Director: Signature, Date	

ANNEX K Approved Analytical Methods Reference Index

The following table lists the primary reference methods approved for use under Module 4 of this Manual. Laboratories may adopt equivalent validated methods subject to method validation or verification per Module 6 and approval by the Laboratory Director.

Section	Analyte Category	Method Reference	Description
4.3	Metals (ICP-OES)	USEPA 200.7	Metals by ICP-OES, water and wastes
4.3	Metals (AAS general)	USEPA 7000B	AAS general guidance document
4.3	Acid digestion (solids)	USEPA 3050B	Acid digestion of sediments, sludge, soils
4.3	Microwave digestion	USEPA 3051A	Microwave-assisted acid digestion
4.3	Mercury (aqueous)	USEPA 7470A	Hg in liquid waste by CV-AAS
4.3	Mercury (solids)	USEPA 7471B	Hg in solid/semi-solid waste by CV-AAS
4.3	Mercury (water)	ISO 17852:2006	Mercury in water by CV-AFS
4.4	OCPs by GC-ECD	USEPA 8081A	Organochlorine pesticides by GC-ECD
4.4	PCBs by GC-ECD	USEPA 8082A	PCBs as Aroclors by GC-ECD
4.4	SVOCs confirmation	USEPA 8270D	Semi-volatile organics by GC-MS
4.4	Soxhlet extraction	USEPA 3540C	Soxhlet extraction for solid matrices
4.4	Pressurised fluid extr.	USEPA 3545A	Pressurised fluid extraction (solid matrices)
4.5	OPPs by GC-NPD	USEPA 8141B	Organophosphorus compounds by GC
4.6	PAHs by HPLC	USEPA 8310	PAHs by HPLC fluorescence/UV
4.6	PAHs by GC-MS	USEPA 8270D	Semi-volatile organics by GC-MS (confirmation)
4.7	VOCs (aqueous)	USEPA 8260C	VOCs by purge-and-trap GC-MS
4.7	VOCs (GC-FID)	USEPA 8015D	Non-halogenated organics by GC/FID

4.9	Petroleum hydrocarbons	USEPA 8015D	Non-halogenated and halogenated VOCs by GC-FID
4.10	pH	ISO 10523	Water quality: pH determination
4.10	COD	ISO 15705	COD: small sealed tube method
4.10	BOD ₅	ISO 5815-1	BOD ₅ determination
4.11	WEEE metals (XRF)	IEC 62321	Determination of restricted substances (XRF)
4.11	WEEE: PBBs/PBDEs	USEPA 8270D	Semi-volatile organics by GC-MS
4.12	Polymer identification	ATR-FTIR / Raman	Polymer type identification; library match ≥80%
4.12	Plastics: heavy metals	USEPA 3051A + ICP-OES	Microwave digestion, ICP-OES analysis
4.12	Phthalate plasticisers	USEPA 8270D	GC-MS, selected ion monitoring
4.12	Microplastics	Stereomicroscopy + FTIR	Enumeration and polymer confirmation
4.13	Lead-acid: Pb	USEPA 3050B + FAAS	Acid digestion, flame AAS
4.13	Ni-Cd: Cadmium	USEPA 3050B + GFAAS	Acid digestion, graphite furnace AAS
4.13	Li-ion: Co/Ni/Mn	USEPA 3051A + ICP-OES	Microwave digestion, ICP-OES
4.14 (REFERRAL)	Dioxins/Furans (PCDD/Fs)	USEPA 8290A	HRMS required: international referral only
4.14 (REFERRAL)	Methylmercury speciation	GC-ICP-MS	International referral only

ANNEX L Chemical Compatibility and Segregation Chart

Use this chart to determine whether two chemical categories may be stored together (✓) or must be segregated (✗). Cells marked ✗ indicate chemicals that may react violently, generate toxic gas, or create fire or explosion risk if mixed or stored in proximity.

Category	Acids	Bases	Oxidisers	Flam. Solvents	Halog. Solvents	Cyanides	Mercury Cpds	Heavy Metals	Battery Acid	Li-ion Electrolyte
Acids	✓	✗	✗	✗	✓	✗ HCN!	✗	✓	✗	✗
Bases	✗	✓	✗	✗	✓	✓	✗	✓	✗	✗
Oxidisers	✗	✗	✓	✗ FIRE!	✗	✗	✗	✓	✗	✗
Flam. Solvents	✗	✗	✗ FIRE!	✓	✗	✓	✓	✓	✓	✗
Halog. Solvents	✓	✓	✗	✗	✓	✓	✓	✓	✓	✗
Cyanides	✗ HCN!	✓	✗	✓	✓	✓	✓	✓	✗	✗
Mercury Cpds	✗	✗	✗	✓	✓	✓	✓	✓	✗	✗
Heavy Metals	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Battery Acid	✗	✗	✗	✓	✓	✗	✗	✓	✓	✗
Li-ion Electrolyte	✗	✗	✗	✗	✗	✗	✗	✓	✗	✓

WARNING: Cyanide compounds must never be stored with acids (HCN gas generation). Oxidisers must never be stored with flammable solvents (fire and explosion). Mercury compounds must never be stored with acids or strong oxidisers. Li-ion battery electrolyte must never be mixed with aqueous solutions without prior hazard assessment. When in doubt, segregate and consult the SDS.

First Edition | 2026

National Environmental Standards and Regulations Enforcement Agency
No. 56 Lome Crescent, Wuse Zone 7, FCT Abuja, Nigeria