

CHAPTER 10 MC SAMPLING

10-1. Introduction.

a. This chapter has been prepared to address the planning and performing of MC investigations by USACE MM DCs, Removal Districts, and their contractors at MRAs under the MMRP. It is focused on FUDS, but could be applied to Base Realignment and Closure (BRAC) or Installation Restoration Program (IRP) sites with MC concerns. An overview of the environmental chemistry of military munitions and appropriate sampling and analyses at MRAs is provided. Table B-7 in Appendix B is a checklist for the PDT to follow when planning MC investigations.

10-2. Objective.

a. Project-specific sampling requirements should be determined by development of clear project objectives, definition of data needs, and establishing specific data quality objectives through the TPP process. An appropriate sampling design, including the type and number of samples, should be developed based on those project-specific objectives. A multi-disciplinary PDT is needed to adequately develop appropriate sampling designs.

b. MC investigations are typically performed at MRAs for one of two purposes:

(1) Determining Presence or Absence of MC Contamination. If MEC is present (or suspected) at a site and the presence of MC in environmental media is unknown, sampling is conducted to determine whether it exists. This type of investigation is typically biased to look at areas where contamination is suspected to be the worst case. Limited sampling to evaluate the presence or absence of MC contamination should be conducted during the SI phase of a munitions response project. Determination of presence of MC at a site is not sufficient to make a decision, its significance in terms of potential threat to human health and the environment should be determined through screening level risk assessment in the SI.

(2) Establishing Nature and Extent of MC Contamination. If MC contamination is determined to exist, further investigation may be required to determine the nature and extent of the contamination, as well as to define the risk to human health and the environment. This investigation would typically be conducted during the RI/FS phase of a munitions response project and should support preparation of a baseline risk assessment.

c. Risk assessments prepared for MC contamination should comply with applicable USACE and USEPA requirements for HTRW risk assessments as defined in, but not limited to, EM 200-1-4 and EP 200-1-15.

d. The requirements provided in this document focus on scoping and executing investigations to determine the presence or absence of MC contamination. The sampling requirements for all projects should be determined on a project-specific basis by the PDT through the TPP process (see EM 200-1-2) and development of a CSM (see EM 1110-1-1200).

e. Most of the requirements outlined in this document also apply to investigations to determine the nature and extent of MC contamination, but those investigations will also include additional requirements not described here. If evaluation of presence or absence of MC contamination is delayed until the RI/FS phase, it is recommended that sampling be conducted in a phased approach within the RI/FS (i.e., that initial samples be collected to determine whether contamination is present with additional samples being collected prior to the completion of the RI/FS to establish the nature and extent of contamination). For additional information on RI/FS requirements, see US Environmental Protection Agency's (EPA's) Guidance on Conducting Remedial Investigations and Feasibility Studies under CERCLA, EM 1110-1-502, Technical Guidelines for Hazardous and Toxic Waste Treatment and Cleanup Activities, and EP 1110-1-18.

f. Additionally, Long-Term Management (LTM) activities may be required for the MC portion of MMRP projects following the Remedial Action Operation (RA-O) phase. If sampling and analysis is required during the LTM phase, many of the requirements and recommendations provided in this document would also apply.

10-3. Initial MC Investigation Planning.

a. An MC investigation process that is capable of effectively identifying MC contamination must employ three fully integrated components, as follows:

(1) Experienced Personnel. Personnel involved with the MC investigation should be experienced with the theoretical and practical aspects of military munitions chemistry, field sampling, laboratory analyses, and risk assessment. The selection of laboratories and analytical methodology, determination of appropriate screening levels, and preparation of screening level or baseline risk assessment require qualified and experienced individuals. A qualified chemist and a qualified risk assessor should actively participate in the management of all MC investigations beginning with the initial planning and formulation of project objectives. A "qualified chemist" is a person with a minimum of a Bachelor's degree in chemistry or a closely related field and at least 5 years of directly related environmental chemistry experience, preferably involving military munitions. A "qualified risk assessor" is a person with a minimum of a Bachelor's degree in chemistry, biology, or toxicology [or a closely related field] and at least 5 years of directly related environmental risk assessment experience. Sampling personnel should be trained in appropriate sampling procedures and associated documentation requirements. If field analytical methods are used, personnel executing these methods should have documented training and experience performing the planned methodology.

(2) Experienced Laboratory. The laboratory used should have experience in handling military munitions samples. The analytical laboratory should be identified early in the project planning (preferably at the proposal stage). The laboratory must be identified in the Sampling and Analysis Plan (SAP) and hold applicable state certifications to perform the analytical methods required (if available). Laboratories must also meet the requirements of the Hazardous, Toxic, and Radioactive Waste (HTRW) Chemical Data Quality Management (CDQM) Policy for Environmental Laboratory Testing, to include National Environmental Laboratory Accreditation Program (NELAP) accreditation for all applicable and available fields of testing (FoT) and self declaration of compliance with the Department of Defense (DoD) Quality Systems Manual (QSM) (latest version). For a list of current NELAP accredited labs, please see <http://www.nelac-institute.org/>.

(a) Any laboratory performing chemical analysis must provide their self declaration and supporting documentation to the applicable MM DC in order to be approved by that MM DC. The determination of qualifications of the laboratory should be at the discretion of the MM DC Project Chemist. If the laboratory fails to meet project-specific requirements at any time, the Contracting Officer (CO) or Contracting Officer's Representative (COR) may request use of the laboratory be discontinued and analytical services be procured from another qualified laboratory that can meet project-specific requirements. Samples may not be subcontracted to another laboratory without the approval of the MM DC PDT. The subcontracted laboratory must meet all requirements for the contract laboratory.

(3) Accuracy and Precision of Sample Locations. The personnel performing the MC investigation must have the ability to accurately and precisely locate a sample location to other known points, preferably using a common survey grid and/or datum. Sample locations should be recorded to within 3 feet of the actual survey location.

b. If any of the above three components is lacking, the overall MC process may be unable to meet the project's objectives. Therefore it is important to carefully plan and integrate all aspects of an MC investigation and not to start fieldwork prematurely.

10-4. Sampling and Analysis Considerations.

a. Sampling and analysis requirements will vary based upon site-specific conditions and must be addressed during TPP activities. Safety concerns must be addressed. If sampling is performed in a potential MEC environment, all requirements from EP 75-1-2, MEC Support during HTRW and Construction Activities, apply unless sampling is performed during intrusive MEC operations. If that is the case, the procedures for sampling should be included in the Work Plan along with other MEC operations procedures.

b. Further considerations that may affect sampling and analysis activities include:

(1) MEC Depth. If MEC items are located on the surface, generally, initial sampling should be surficial. Research data has shown the most secondary explosives are found in the top 2” of soil. The sample depth that constitutes “surface” soils should be defined during the TPP taking this information, as well as data use, into consideration, as the definition of what constitutes surface soils varies. Alternate depths would be appropriate in conditions of shifting sands, erosion, etc. If MEC items are also found in the subsurface, initial sampling should also be taken from subsurface soil near the identified MEC location.

(2) MEC Item Composition. Analytical requirements for MC should be based on the anticipated MEC item composition, if known. If unknown, some assumptions may be made regarding typical composition to establish the analytical requirements for MC. In either case, the anticipated MEC items, along with fill information, if available, should be tabulated in the Work Plan. Information on MEC item composition is available from the MIDAS database (available at <https://midas.dac.army.mil/>; access requires registration and is restricted to DoD personnel and DoD contractors), various Technical Manuals, and the Common Range Operations Reports (contact HTRW CX - CENWO-HX-M - for more information). An ammunition composition database for FUDS era munitions is also in development by USACE (contact HTRW CX - CENWO-HX-M - for more information). Many types of filler used in MEC items are composition explosives, consisting of two or more explosive compounds mixed to produce an explosive with more suitable characteristics for a particular application. Some typical examples are listed in Table 10-1. Exact compositions vary; they are documented in TM 9-1300-214, Military Explosives.

Table 10-1. Composition Explosive Makeup (1)

Composition Explosive	Explosive Compounds	Other Ingredients (2)
Amatol	Ammonium nitrate and TNT	
Composition A (A, A2, A3, A4, A5, A6)	RDX	Beeswax, synthetic wax, desensitizing wax, stearic acid, or polyethylene
Composition B (Cyclotol, B, B2, B3)	RDX and TNT	Wax, calcium silicate
Composition C (C, C2, C3, C4)	RDX, explosive plasticizer (C2 contained nitrotoluenes, dinitrotoluenes, trinitrotoluene, nitrocellulose, dimethylformamide; C3 contained nitrotoluenes, dinitrotoluenes, TNT, tetryl,	Nonexplosive oily plasticizer (included lecithin) or polyisobutylene, may also contain lead chromate, and

	and nitrocellulose)	lamp black
Octol	HMX and TNT	
Pentolite	PETN and TNT	
Picratol	Ammonium picrate and TNT	
Tetrytol	Tetryl and TNT	
Tritonal	TNT	Flaked aluminum
HBX (HBX-1, HBX-3, HBX-6)	RDX, TNT (3), nitrocellulose	Calcium chloride, calcium silicate, aluminum, wax, and lecithin
Minol	TNT and ammonium nitrate	Aluminum
Torpex	RDX and TNT	Aluminum powder and wax

(1) Source: TM 9-1300-214

(2) Varies by type, may contain any or all other ingredients listed

(3) HBX-6 does not contain TNT

(3) Background Conditions. In some locations, native or anthropogenic background concentrations of metals, perchlorate, or PAHs may exceed non-site specific risk based screening levels or regulatory limits that are commonly used for screening purposes or response action decision making. If these parameters are analyzed and no appropriate regional or site-specific background data are available for the project property, background samples should be collected and analyzed.. Some available resources for background condition evaluation include:

(a) Guidance for Environmental Background Concentration Analysis Volume I: Soil (NAVFAC UG-2049-ENV, April 2002) <https://portal.navy.mil/>

(b) Guidance for Environmental Background Concentration Analysis Volume II: Sediment (NAVFAC UG-2054-ENV, April 2003) <https://portal.navy.mil/>

(c) Guidance for Environmental Background Concentration Analysis Volume III: Groundwater (NAVFAC UG-2059-ENV, April 2004) <https://portal.navy.mil/>

(d) Guidance for Comparing Background and Chemical Concentrations in Soil for CERCLA Sites (EPA 540-R-01-003 OSWER 9285.7-41, September 2002)
<http://www.epa.gov/oswer/riskassessment/pdf/background.pdf>

(4) Regulatory Requirements. Varying state and local requirements and requests for sampling and analysis may exist. These should be considered and addressed during TPP and the development stage of overall project objectives and Data Quality Objectives (DQOs).

(5) Chemical-Specific Screening Levels, Applicable or Relevant and Appropriate Requirements (ARARs) and To Be Considereds (TBCs). Chemical-specific screening levels, ARARs, and TBCs can impact the choices of the appropriate analytical methodology as part of the DQO process. Anticipated criteria should be established during the planning process to ensure proper sampling procedures can be applied; appropriate analytical methodologies can be utilized; meaningful data can be collected; and DQOs can be achieved. These should be documented in planning documents along with the reporting limits/method detection limits specific to the project laboratory to allow comparison/confirmation that methodology is adequate.

(6) Site Hydrology. If significant releases of MC are believed to have occurred, groundwater sampling should be considered. The decision to sample groundwater should be made based on depth to groundwater and its susceptibility to contamination from surface releases, potential receptors, the magnitude of the suspected MC release, and the type of MC suspected at the site. If surface water is located on or near the project property and receives runoff from suspected MC source areas, surface water/sediment sampling should be considered.

c. Collecting a Representative Soil Sample from a Range

(1) Cold Regions Research Engineering Laboratory (CRREL), a USACE Engineering Research and Development Center (ERDC) laboratory, has conducted numerous studies to determine the best means to collect a representative sample on testing and training ranges. These studies have been conducted at primarily active or BRAC sites as part of a Research and Development (R&D) effort. Their current recommendations are documented in full in the Field Analytic Technologies Encyclopedia (FATE) Explosives Module located at <http://clu-in.org/char/technologies/exp.cfm> and in Appendix A of SW8330B located at <http://www.epa.gov/epaoswer/hazwaste/test/new-meth.htm>. It should be noted that sampling performed under these studies to date have included nitroaromatic/nitramines/nitrate ester explosives, but not metals or other MC, with the exception of one limited study that did include metals.

(2) All research in the area of secondary explosives contamination at ranges has supported the use of composite sampling (also referred to as multi-increment sampling) rather

than discrete sampling. The recent update of SW8330B specifically includes multi-increment sampling. As the performance capability and regulatory acceptance of SW8330B increase, this method is expected to become the standard for evaluating secondary explosives contamination at ranges.

(3) SW8330B recommends collecting a 1000 g of soil and sieving and grinding the entire sample prior to subsampling. The sieving and grinding may occur in the field or in the laboratory. Grinding samples that will be analyzed for metals is not recommended at this time. For additional information on laboratory subsampling, see Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples, EPA/600/R-03/027, http://www.clu-in.org/download/char/epa_subsampling_guidance.pdf.

(4) Typically, vegetation (grass, sticks, leaves, moss, etc.) is removed from soil samples prior to laboratory processing, frequently during actual field sampling. SW8330B recommends retaining the vegetation within the processed sample in order to account for any particles that may cling to the vegetation. Depending upon the concentrations of concern and the laboratory's chromatographic separation, this may be problematic for the analysis. For FUDS site characterization projects, this is not recommended, given the time elapsed between the distribution of the explosives and the characterization. For post-BIP samples, this would be appropriate, but it may not be feasible analytically.

(5) SW8330B also recommends sieving samples with #10 (2 mm) sieves rather than the 30 mesh sieves specified in SW8330. It also recommends processing 10 grams of soil rather than 2 grams. For FUDS, this portion of the method should be implemented even if SW8330B is not implemented in full.

(6) The compositing scheme, degree of processing, vegetation inclusion/exclusion, and sieve size must be discussed by the PDT, contractor (if applicable), the laboratory, and the applicable regulatory agencies to ensure acceptance of data to the data users. The regulatory acceptance should be documented to ensure future acceptance of the data.

d. General Guidance for Sampling to Determine Presence or Absence of MC Contamination.

(1) Analysis should be based on MEC fill, if known.

(2) Sampling requirements should be determined by development of clear project objectives, definition of data needs, and establishing specific data quality objectives through the TPP process. An appropriate sampling design, including the type and number of samples, should be developed based on those project-specific objectives.

(3) Soil samples should be collected from each area suspected to contain MC, such as known target impact areas, firing lines, open burn/open detonation areas, hand grenade courts, and areas with high concentrations of MEC.

(4) Sample representativeness should be maximized to the extent practical. Multi-increment sampling and sample processing IAW SW8330B, Appendix A, should be implemented for secondary explosives, unless there are state or local requirements to the contrary. If the MIS approach is not implemented, the rationale for its lack of implementation should be documented. If sampling is to be conducted in a high density MEC environment, MC sampling density must be evaluated relative to safety issues for sampling personnel.

(5) If the site Conceptual Site Model indicates potentially complete pathways, collecting surface water, sediment, and/or groundwater sampling should be considered.

e. General Guidance for Sampling during Blow in Place or Consolidated Shot Operations.

(1) This type of sampling is typically required during site characterization efforts that require ordnance disposal (more likely at the RI/FS stage during intrusive operations) and during removal/remedial actions.

(a) Analysis should be based on MEC fill, if known.

(b) Before and/or after (pre-and post-detonation) soil samples should be collected at the location of each specific type of MEC destroyed.

(c) Pre-detonation samples should be composite samples located as near to the identified MEC to be detonated as is safe and feasible unless there are state or local requirements to the contrary. Pre-detonation samples are used for comparison with post-detonation samples to determine whether any residual MC is due to existing contamination or contamination left due to the detonation.

(d) Post-detonation samples should be biased multi-increment samples unless there are state or local requirements to the contrary. Sample representativeness should be maximized to the extent practical.

10-5. Types of MC Analyses.

a. There are several types of constituents that may require analyses. The actual selection of MC for analysis should be based upon anticipated or known MEC items, as discussed in Section 10.4. Potential MC include, but are not limited, to primary explosives, nitrogen-based explosives, perchlorate, chemical warfare agents (CWAs) and agent breakdown products

(ABPs), white phosphorous (WP), and metals. Primary explosives are of concern primarily at manufacturing sites, so they are not discussed further here.

b. For sampling to determine the presence or absence of MC contamination, fixed laboratory sampling is typically used, but project requirements may make field laboratory methods more cost-effective. Field laboratory methods may be used, but it is recommended that at least 10 percent of analyses be confirmed by fixed laboratory methods.

c. Nitrogen-Based Explosives. Commonly evaluated nitrogen-based explosives, co-contaminants, and breakdown products are shown in Table 10-2. Nitrocellulose (NC), nitroguanidine (NQ), pentaerythritol tetranitrate (PETN), ammonium picrate (AP), picric acid, and RDX breakdown products (typically hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine (MNX); hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine (DNX); and hexahydro-1,3,5-trinitroso-1,3,5-triazine (TNX)) may be required, but are not part of current methods published by the EPA. Each of these analytes except NC can be analyzed with a modification to either method SW8330 or SW8321; however, ammonium picrate is typically reported based on the analysis of picric acid. If analytes that are not part of methods published by the EPA are included in the project, proposed methodology must be accepted by the PDT and stakeholders and documentation regarding any method modifications or unpublished methods should be provided in the project SAP.

(1) Field Tests. Field tests for nitrogen-based explosives are shown in Table 10-3. Fate and transport properties of the analytes should be considered prior to the use of field tests, particularly if the use of TNT or RDX as an indicator compound is intended. It is anticipated that for a range that has been out of use for a substantial period of time, most, if not all TNT, would have broken down due to photodegradation and biodegradation. RDX is less likely to have broken down but may not be an appropriate indicator compound depending upon the age of the range.

(a) Immunoassays have been developed for 2,4,6-trinitrotoluene (TNT) and hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX). The commercially available tests have little cross-reactivity with other nitroaromatic/nitramines explosives.

(b) Colorimetric analyte-specific tests are commercially available for TNT, RDX, and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX). They may be used to analyze for other analytes but require documentation of method modifications used to acquire the other analytes. Additionally, one colorimetric test for general analyte classes is available (EXPRAY™). EXPRAY™ may be used in the field or in the laboratory to determine whether nitroaromatic explosives, nitramine and nitrate ester explosives, or inorganic nitrates are present. It is typically used qualitatively, although it can be used semi-quantitatively with sufficient expertise, as documented in SW8330B and in ERDC/CRREL TN-05-2, Pre-Screening for

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Explosives Residues in Soil Prior to HPLC Analysis Utilizing Expray™
(http://www.crrel.usace.army.mil/techpub/CRREL_Reports/reports/TN05-2.pdf).

(2) Fixed Laboratory Tests.

(a) Several technologies are used to analyze for nitroaromatic/nitramine explosives. Currently available methods are provided in Table 10-4. A version of SW8330 is typically used unless significant interferences are anticipated. Some laboratories are unable to perform quantitative second column confirmation for explosives per DoD QSM/EM 200-1-3/SW8000C (i.e., five-point calibrations must be performed for each target analyte for the primary and confirmatory columns and quantitative results for each column must be reported). This requirement should not be waived for MC projects. Based upon project requirements, exceptions may be considered for the following co-eluting pairs: 2-amino-4,6-dinitrotoluene (2-Am-DNT)/4-amino-2,6-dinitrotoluene (4-Am-DNT), 2-nitrotoluene (2-NT)/4-nitrotoluene (4-NT), and 2,4-dinitrotoluene (2,4-DNT)/2,6-dinitrotoluene (2,6-DNT), but the exception should be evaluated based upon review of relevant ARARs and TBCs. SW8095 may be recommended if lower reporting limits are required, but it is not widely available commercially. SW8321 is typically used for complex matrices where there is concern regarding confirmation of positive results. It may also be used by laboratories with coelution problems for SW8330; however, routine use of liquid chromatography/mass spectrometry (LC/MS) confirmation to compensate for the laboratory's failure to properly execute SW8330 should not incur additional cost to the government. For all aqueous samples, sample preparation should be performed in accordance with SW3535A solid phase extraction (SPE) rather than by the SW8330 salting out procedure unless a reasonable technical rationale (i.e. SPE disk clogging) is documented.

Table 10-2. Common Nitrogen-Based Explosives, Co-Contaminants, and Breakdown Products

Compound	Description (1)	Abbreviation	CAS Number (2)
Octahydro-1, 3, 5, 7-tetranitro-1,3,5,7-tetrazocine	Nitramine explosive; also RDX co-contaminant	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	Nitramine explosive; also HMX co-contaminant	RDX	121-82-4
1,3,5-Trinitrobenzene	TNT co-contaminant and breakdown product	1,3,5-TNB	99-35-4
1,3-Dinitrobenzene	DNT breakdown product and TNT co-contaminant	1,3-DNB	99-65-0
Methyl-2,4,6-trinitrophenylnitramine	Nitramine explosive	Tetryl	479-45-8
Nitrobenzene	DNT co-contaminant	NB	98-95-3
2,4,6-Trinitrotoluene	Nitroaromatic explosive	2,4,6-TNT	118-96-7
4-Amino-2,6-dinitrotoluene	TNT breakdown product	4-Am-DNT	1946-51-0
2-Amino-4,6-dinitrotoluene	TNT breakdown product	2-Am-DNT	355-72-78-2
2,4-Dinitrotoluene	Nitroaromatic explosive/ propellant; also TNT co-contaminant	2,4-DNT	121-14-2
2,6-Dinitrotoluene	Nitroaromatic explosive/ propellant; also TNT co-contaminant	2,6-DNT	606-20-2
2-Nitrotoluene (o-Nitrotoluene)	DNT co-contaminant	2-NT	88-72-2
3-Nitrotoluene (m-Nitrotoluene)	DNT co-contaminant	3-NT	99-08-1
4-Nitrotoluene (p-Nitrotoluene)	DNT co-contaminant	4-NT	99-99-0
Nitroglycerine	Nitrate ester explosive/propellant	NG	55-63-0
Ammonium Picrate	Nitroaromatic explosive	AP	131-74-8
Picric Acid	Nitroaromatic explosive	PA	88-89-1
Pentaerythritol Tetranitrate	Nitrate ester explosive	PETN	78-11-5
Hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine	RDX breakdown product	MNX	5755-27-1
Hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine	RDX breakdown product	DNX	80251-29-2
Hexahydro-1,3,5-trinitroso-1,3,5-triazine	RDX breakdown product	TNX	13980-04-6
Nitroguanidine	Nitroaromatic/nitramine explosive/ propellant	NQ	556-88-7

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Compound	Description (1)	Abbreviation	CAS Number (2)
3,5-Dinitroaniline	TNB breakdown product	3,5-DNA	618-87-1

1 Information gathered from TM 9-1300-214, Military Explosives; ATSDR Toxicological Profiles for 2,4- and 2,6-Dinitrotoluene and for 2,4,6-Trinitrotoluene (located at <http://www.atsdr.cdc.gov/toxpro2.html>) and the Hazardous Substances Data Bank (located at <http://toxnet.nlm.nih.gov/>).

2 Chemical Abstracts Service registry number.

Table 10-3. Field Tests for Nitrogen-Based Explosives

Method No.	Title
SW4050	TNT Explosives in Soil by Immunoassay
SW4051	RDX in Soil by Immunoassay
SW8515	Colorimetric Screening Method for TNT in Soil
SW8510	Colorimetric Screening Procedure for RDX and HMX in Soil
N/A	Expray™

Table 10-4. Fixed Laboratory Tests for Nitrogen-Based Explosives, Co-Contaminants, and Breakdown Products

Method No.	Title
SW8330B	Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography (HPLC)
SW8332	Nitroglycerine by HPLC
SW8095	Explosives by Gas Chromatography (GC)
SW8321A (1)	Explosives by HPLC/Mass Spectrometry (MS)

Method No.	Title
EPA 529	Determination of Explosives and Related Compounds in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)

1 This method is typically cited for HPLC/MS of explosives. However, no published version includes explosives. An effort is underway to update SW8321 that would address explosives.

d. Perchlorate. Perchlorate (CAS Number 14797-73-0) is the anion of perchloric acid. Two salts of primary concern are Ammonium Perchlorate (CAS Number 7790-98-9, NH_4ClO_4) and Potassium Perchlorate (CAS Number 7778-74-7, KClO_4). The latest perchlorate policies and guidance can be found at <http://www.dodperchlorateinfo.net/>. Current guidance includes:

- (1) Policy on DoD Required Actions Related to Perchlorate, January 26, 2006
- (2) DoD Perchlorate Handbook, March 2006
- (3) Interim Army Guidance on Perchlorate for Restoration/Cleanup Activities, May 25, 2006
- (4) EPA Assessment Guidance for Perchlorate, January 26, 2006

e. Additional information on perchlorate is available from the Interstate Technology Regulatory Council (ITRC) Perchlorate Team (http://www.itrcweb.org/teampublic_Perchlorate.asp), to include Perchlorate: Overview of Issues, Status, and Remedial Options (September 2005), available at <http://www.itrcweb.org/Documents/PERC-1.pdf>.

(1) Field Tests. Field tests based on an ion-selective electrode (ISE), colorimetry, capillary electrophoresis, and ion mobility/mass spectroscopy exist for perchlorate, but they have not been widely used at this time. The ISE method is documented in Perchlorate Screening Study: Low Concentration Method for the Determination of Perchlorate in Aqueous Samples Using Ion Selective Electrodes: Letter Report of Findings for the Method Development Studies, Interference Studies, and Split Sample Studies, including Standard Operating Procedure, available at http://www.clu-in.org/programs/21m2/letter_of_findings.pdf. The colorimetry test is documented in CRREL TR 04-8, Field Screening Method for Perchlorate in Water and Soil, available at http://www.crrel.usace.army.mil/techpub/CRREL_Reports/reports/TR04-8.pdf.

(2) Fixed Laboratory Tests. All fixed laboratory tests for perchlorate are based on ion chromatography or liquid chromatography. The DoD Perchlorate Handbook requires that

detections of perchlorate above reporting levels be confirmed with mass spectrum confirmation. Fixed laboratory tests for perchlorate are shown in Table 10-5.

Table 10-5. Fixed Laboratory Tests for Perchlorate

Method No.	Title	DoD Perchlorate Handbook Status
EPA 314.0	Determination of Perchlorate in Drinking Water by Ion Chromatography	Not recommended. Only allowed for existing NPDES permits.
EPA 314.1	Determination of Perchlorate in Drinking Water Using Inline Column Concentration/Matrix Elimination Ion Chromatography with Suppressed Conductivity Detection	Not recommended. All results above the method reporting limit <i>must</i> be confirmed using MS.
Draft SW9058	Determination of perchlorate using ion chromatography with chemical suppression conductivity detection	Not recommended. All results above the method reporting limit <i>must</i> be confirmed using MS.
EPA 331.0	Determination of Perchlorate in Drinking Water by Liquid Chromatography Electrospray Ionization Mass Spectrometry	Recommended for drinking water
EPA 332.0	Determination of Perchlorate in Drinking Water by Ion Chromatography with Suppressed Conductivity and Electrospray Ionization Mass Spectrometry	Recommended for drinking water
SW6850	Perchlorate in Water, Soils and Solid Wastes Using High Performance Liquid Chromatography/ Electrospray Ionization/Mass Spectrometry	Recommended for drinking water, groundwater, soil, and wastewater
SW6860	Perchlorate In Water, Soils And Solid Wastes Using Ion Chromatography/ Electrospray Ionization/Mass Spectrometry	Recommended for drinking water, groundwater, soil, and wastewater

f. CWAs and ABPs. CWAs and ABPs are listed in Table 10-6. No methods published by EPA exist for CWAs or ABPs. Methods available have primarily been developed by Edgewood Chemical Biological Center (ECBC). Analyses are performed based on ECBC (or commercial laboratory) standard operating procedures. Most are based on GC/MS or GC/Flame Photometric Detection (FPD). Several ABP methods are in development by HPLC and Capillary Electrophoresis. CWA analysis must go to either ECBC or a commercial laboratory with a Bailment Agreement. Additional requirements for sampling and analysis related to

CWAs and ABPs are found in EP 75-1-3. Note that if CWA-contaminated soil is suspected, the Chemical Warfare Materiel (CWM) Design Center should be contacted, as a Chemical Safety Submission for DoD Explosives Safety Board (DDESB) review and concurrence may be required.

g. White Phosphorus. WP (CAS 7723-14-0, P₄) reacts with air and requires special handling for sampling and analysis. Typically, if significant levels of WP are present in soil that is excavated, visible smoke will be observed. If visible smoke is observed, notify contract laboratory and confirm willingness to accept for analysis.

(1) Field Tests. No field tests have been developed for WP, although the fixed laboratory test has been used on a limited basis in the field, to include use of Solid-phase micro-extraction (SPME) as discussed in SW7580.

(2) Fixed Laboratory Tests. Fixed laboratory tests for WP are all based on gas chromatography. The only published method for WP is SW7580, a GC method with a nitrogen-phosphorus detector (NPD). A GC/MS method is also available, but is not published. Due to increased regulation of WP by the Drug Enforcement Agency, the standard is currently unavailable. Therefore, analytical capabilities for this compound are very limited. Contact the MM CX for methodology recommendations.

(3) Other Considerations. If dewatering in an identified WP area or decontamination of WP contaminated equipment is required, water may need to be collected and analyzed prior to disposal. Appropriate disposal procedure should be followed according to the analytical results. WP is considered a Resource Conservation and Recovery Act (RCRA) reactive waste; therefore, careful planning is required prior to conducting an investigation. Planning considerations, to include disposal options, should be discussed in the Work Plan

Table 10-6. Chemical Warfare Agents and Agent Breakdown Products

Compound	Description	Abbreviation	CAS Number ⁽¹⁾	Analytical Technology
<i>Chemical Warfare Agents</i>				
Sulfur Mustard (bis(2-chloroethyl)sulfide)	Blister Agent	H, HS, HD	505-60-2	GC/MS
Lewisite (Dichoro(2-chlorovinyl)arsine)	Blister Agent	L	541-25-3	GC/MS ⁽²⁾
Nitrogen Mustard (bis(2-chloroethyl)ethylamine)	Blister Agent	HN-1	538-07-8	GC/MS

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Compound	Description	Abbreviation	CAS Number ⁽¹⁾	Analytical Technology
Nitrogen Mustard (tris(2-chloroethyl)amine)	Blister Agent	HN-3	555-77-1	GC/MS
Tabun (Ethyl n, n- dimethylphosphoramidocyanidate)	Nerve Agent	GA	77-81-6	GC/MS
Sarin (Isopropyl methylphosphonofluoridate)	Nerve Agent	GB	107-44-8	GC/MS
Soman (Pinacolyl methylphosphonofluoridate)	Nerve Agent	GD	96-64-0	GC/MS
o-Ethyl S-(2-diisopropylaminoethyl) Methylphosphonothiolate)	Nerve Agent	VX	50782-69-9	GC/MS
<i>Agent Breakdown Products</i>				
1,4-Dithiane	HD ABP		505-29-3	GC/MS
1,4-Thioxane	HD ABP		15980-15-1	GC/MS
Thiodiglycol	HD ABP	TDG	540-63-6	GC/MS or HPLC
2-Chlorovinyl Arsenous Acid	L ABP	CVAA	85090-33-1	GC/MS ⁽²⁾
2-Chlorovinyl Arsenous Oxide	L ABP	CVAO	3088-37-7	GC/MS ⁽²⁾
Triethanolamine	HN-3 ABP	TEA	102-71-6	CE
Ethyl-diethanolamine	HN-1 ABP		139-87-7	CE
Isopropyl methyl phosphonic acid	GB	IMPA	1832-54-8	IC
Methylphosphonic Acid	GB, GD, and VX ABP	MPA	993-13-5	IC
Dimethyl methylphosphonate	GB simulant and precursor	DMMP	756-79-6	GC
Ethyl methylphosphonic acid	VX ABP	EMPA	1832-53-7	IC
Diisopropyl methylphosphonate	GB ABP	DIMP	1445-75-6	GC
Pinacolyl methylphosphonic acid	GD ABP	PMPA	616-52-48	IC
S-(2-diisopropylaminoethyl)- methylphosphonothioic acid	VX ABP	EA2192	73207-98-4	GC/MS

1 Chemical Abstracts Service registry number.

2 L, CVAA, and CVAO must be derivatized and form the same derivative. They are analyzed and reported together.

h. Metals. Metals are found in all military munitions. Certain munitions only contain metals (i.e., incendiaries). Metal analyses may be based on a limited list if the type(s) of ordnance are known or can be reasonably assumed. If not, it is recommended to analyze for the 23 Total Analyte List (TAL) metals (aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc), unless a state-specific list exists. Depending upon munitions used on the site, zirconium, titanium, and strontium may also be potential metals of concern. If metals are analyzed, establishing background conditions should be discussed by the PDT and stakeholders during TPP. For additional discussion of background considerations, see 10-4b(3).

(1) Field Tests. There are two published field tests available for metals: SW4500, Mercury in Soil by Immunoassay and SW6200, Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment. SW6200 is appropriate for some, but not all of the metals of interest. Other field tests may be used on munitions response projects, if appropriate, but their use must be approved by the MM-DC.

(2) Fixed Laboratory Tests. There are several published methods for metals other than mercury. Currently available tests for metals are shown in Table 10-7. Determination of the appropriate method should depend upon the established DQOs. For soil analysis, SW6010B is typically appropriate, although it may require the use of “Inductively Coupled Plasma (ICP) trace” rather than ICP. For lower reporting limits, SW6020 or SW7000 series (to be replaced by SW7010) may be required.

Table 10-7. Fixed Laboratory Tests for Metals

Method Number	Title
SW6010C	Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)
SW6020A	Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)
SW7010	Graphic Furnace Atomic Absorption (GFAA) Spectrophotometry
SW7000 series	Individual Metals by GFAA
SW7470A/ SW7471A	Mercury by Cold Vapor Atomic Absorption (CVAA)

(3) Small arms-specific Considerations. There has been a considerable amount of study performed at small arms ranges. These studies have focuses on where the contamination is likely to be and on how best to measure it. One key aspect to characterizing soils at a small arms range is reaching consensus on whether to sieve the soil samples prior to analysis. One of the primary reasons to sieve is to remove bullet fragments. Retaining bullet fragments would yield a higher concentration of lead; however, the lead in the fragments would not be readily available to receptors. This subject is recommended for discussion at project TPP sessions. If additional sample preparation is planned, it should be thoroughly described in project work plans. Prior to conducting site characterization or remediation at small arms ranges, review of the following publications is recommended.

(a) Army Environmental Center (AEC) software/documentation for small arms ranges, available through AEC:

- “REST” (Range Evaluation Software Tool)
- “ASAP” (Army Sampling and Analysis Plan)

(b) ITRC Guidance: Characterization and Remediation of Soils at Closed Small Arms Firing Ranges, available at <http://www.itrcweb.org/Documents/SMART-1.pdf>

(c) EPA Region 2 Guidance: Best Management Practices for Lead at Outdoor Shooting Ranges, available at <http://www.epa.gov/region02/waste/leadshot/>

(d) TRW Recommendations for Performing Human Health Risk Analysis on Small Arms Shooting Ranges (OSWER #9285.7-37), available at <http://www.epa.gov/superfund/programs/lead/products/firing.pdf>

10-6. Sampling and Analysis Plan (SAP). Prior to initiating field activities, a SAP should be prepared. The SAP may be a stand-alone document or be an appendix of the Work Plan. It describes the project requirements for all sampling and analysis activities that should take place during a munitions response project. The SAP must consist of the Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP) when sampling for MC as required by ER 200-3-1. A SAP Review Checklist is provided in Appendix J of EM 200-1-3.

a. SAP Requirements. The SAP should:

- (1) Address each requirement as identified in ER 1110-1-263.
- (2) Be prepared in accordance with (IAW) EM 200-1-3.

(a) Additional reference material on QAPPs may be found in the Intergovernmental Data Quality Task Force Uniform Federal Policy for QAPPs – QAPP Manual, located at <http://www.epa.gov/fedfac/documents/qualityassurance.htm>

(3) Include the laboratory Quality Assurance/Quality Control (QA/QC) plan and applicable Standard Operating Procedures as an appendix (Compact Disk (CD) submittal preferred).

(4) Clearly identify any DoD QSM requirements that a laboratory cannot meet.

(5) Document DoD QSM self declaration of compliance

b. Previously prepared Work Plans for the project property should be used as much as possible in the preparation of the SAP. As a minimum, the level of data quality and QC requirements should be equivalent to what is required in the existing Work Plans with the addition of any new requirements that have been added to improve the defensibility of the data quality since the last work plan submittal.

c. The laboratory must meet all of the requirements specified in the DoD QSM, unless approved in advance in the SAP. As noted above, the requirement for the laboratory to provide quantitative second column confirmation for explosives per DoD QSM/EM 200-1-3/SW8000C should not be waived.

d. SAP Review and Approval. The SAP should be submitted to the Life Cycle Project Manager (LCPM) at the FUDS Geographic District and the MM DC. The MM DC should route the plan to the appropriate MM DC technical staff for review, comment, and approval. For FUDS, SAPs must be submitted to the lead regulatory agency for notice and opportunity to comment IAW ER 200-1-3. For other projects, this is recommended also. Once approved by the CO, the SAP represents the standard to which all sampling and analysis activities will be compared to assure compliance for the project.

10-7. Data Interpretation, Validation, Reporting, and Decision Making.

a. Data Interpretation. After a project property undergoes sampling and analysis, it is necessary to carefully interpret all data and determine if project objectives have been met. Project related information such as possible MEC composition (if available) and donor explosive composition should be provided as part of data interpretation. If numeric DQOs, such as screening levels, have been identified for the project, a comparison of those DQOs must take place. Environmental Data Management System (EDMS) software is available to USACE personnel and contractors for DQO comparison. Data gaps may exist and should be identified and explained. Data gaps may require additional action as part of the remedial response.

b. Data Review. The contractor should perform data review according to their approved SAP requirements. Review procedures should be based on EM 200-1-10, Guidance for Evaluating Performance-Based Chemical Data; the latest versions of the CLP National Functional Guidelines (EPA 540-R-99-008 and EPA 540-R-04-004, available at <http://www.epa.gov/oerrpage/superfund/programs/clp/guidance.htm>); and any applicable state or regional requirements. During TPP, the amount of review should be coordinated with regulatory agencies. The review should be documented in the draft and final engineering reports. Review documentation should address review of laboratory and field QC results. Persons performing the data validation should have appropriate experience as determined by their contractual requirements.

c. Data Reporting. Laboratories and contractors each have data reporting responsibilities.

(1) Laboratories must provide data reporting elements for definitive data IAW DoD QSM Appendix DoD-A – Reporting Requirements”. They should report all analytical results greater than the Method Detection Limit (MDL) that, in the analyst’s professional judgment, are believed to be reliably detected. Concentrations reported between the MDL and the Practical Quantitation Limit (PQL) must be flagged as estimated. PQLs must be at least 3 times MDLs for all analytes, as required by the DoD QSM. Non-detect results should be reported to the PQL unless the laboratory has demonstrated the ability to report non-detects to smaller concentrations by means such as detection limit check samples. Data packages should be organized and assembled such that the analytical results are reported on a per-batch basis.

(2) Contractors should submit the complete data packages to the MM DC and reference them as part of the large study report. They should include the analytical data in the draft and final engineering reports in tabular data summary table format. There should be, at a minimum, two types of data summary tables. The first should include all analytical results for all samples collected. The second should include all analytical results greater than the MDL for all samples collected. Both tables should include for each analyte, medium of concern, and study area, the decision limits (e.g., risk based screening limits and background thresholds, if any), the MDL, the reporting limit for non-detects, and the PQL (if different from the reporting limit for non-detections). Both tables should be sorted by sample field ID, method, analyte, and include appropriate data flags resulting from laboratory review and contractor’s data validation. Results on all tables should be reported with an appropriate number of significant figures, e.g., J-qualified results below the PQL should be reported to one significant figure. If there are PQLs that exceed the applicable decision limit, these should be annotated.

(3) The analytical data should also be provided electronically to the MM DC by the Contractor in the Staged Electronic Data Deliverable (SEDD) format for all FUDS projects. The SEDD stage and specification version required should be stated in project Statements of Work (SOWs)/Performance Work Statements (PWSs). Other project-specific Electronic Data Deliverable requirements should be documented in project SOWs/PWSs. For more

information on the SEDD format, see <http://www.epa.gov/superfund/programs/clp/sedd.htm>. The SEDD formatted deliverable can be evaluated by the Automated Data Review (ADR) software. ADR software is intended to automate certain data review functions that are strictly comparisons to numeric criteria (i.e., holding time compliance, comparison to recovery/relative percent difference limits, etc.) Use of the ADR software will require that the contractor develop a comprehensive library file for all of the methods to be analyzed under the SOW/PWS. The library file should accurately reflect all of the analytical quality requirements as documented in the final SAP for the project and should be provided to both MM DC and the subcontract lab for use in screening Electronic Data Deliverable (EDD) submittals. The electronic deliverable must include appropriate data flags resulting from laboratory review and contractor's data validation. All electronic data submitted by the contract laboratory is required to be error-free, and in complete agreement with the hardcopy data. Data files are to be delivered IAW contract requirements. They should be submitted with a transmittal letter from the laboratory that certifies that the file is in agreement with hardcopy data reports and has been found to be free of errors using the latest version of ADR evaluation software provided to the laboratory. The contract laboratory, at their cost, should correct any errors identified by MM DC. The contractor is responsible for the successful electronic transmission of field and laboratory data. The laboratory is responsible for archiving the electronic raw data, associated software, and sufficient associated hardcopy data (e.g., sample login sheets and sample preparation log sheets) to completely reconstruct the analyses that were performed for the period specified after completion of the applicable contract. If no period is specified, laboratories should keep data for 10 years.

d. Decision Making. The sampling and analysis data and evaluations are usually incorporated into a larger study (e.g., SI, Engineering Evaluation/Cost Analysis (EE/CA), RI/FS, Site Characterization, etc.) and the USACE PDT, contractors, and project stakeholders are involved in making decisions regarding future work to be performed.

10-8. Quality Management.

a. Data Quality. The contractor must provide data quality of a level sufficient to support the project's objectives as defined in the SAP. The contractor must provide QC of the various analytical tasks performed. The contractor is responsible for achieving data quality as defined in the SAP. Analytical data that does not meet QC requirements may be rejected by the government. Re-sampling and re-analysis may be required, with contract type determining whether there are additional costs to the government.

b. Quality Control. It is recommended that field duplicates be collected. The PDT should determine the rate per matrix per analysis per sampling event. Each project sample designated for a field duplicate must be homogenized thoroughly, and then divided equally (if sampling and analysis of volatile organic compounds is required for an MC site, the duplicate should be collocated). Both portions should be sent to the contractor's laboratory, but the

identity of the duplicate should not be provided to the laboratory. The QC samples should include all sample matrices and analytical parameters except disposal parameters (i.e., Toxicity Characteristic Leaching Procedures (TCLP), reactivity, corrosivity, and ignitability). The contractor should administer all QC sample handling and custody requirements in a similar manner to that used for the environmental samples.

c. Coordination with QA Laboratory. If contractual requirements include collection of QA samples, the contractor must provide coordination and QA samples (collected and transported by the contractor) to the QA laboratory identified in the SOW/PWS. The PDT should determine the rate per matrix per analysis per sampling event for the QA splits. The contractor should provide sample containers, shipping, etc. for QA samples. QA samples should be taken as splits of the same samples as QC duplicates (i.e., sample should be homogenized and split in triplicate) (if sampling and analysis of volatile organic compounds is required for an MC site, the QA split should be collocated). The QA split samples should include the same matrices and parameters as QC duplicate samples. The QA laboratory should be provided a list of measurement quality objectives (MQOs). The MQOs should include, but should not be limited to, identification of extraction and analysis method numbers and a list of analytes with required limits. All QA sample handling and custody requirements should be administered by the contractor similar to the environmental samples. The QA samples should be sent to the QA Laboratory by overnight delivery for government contract compliance monitoring. See EM 200-1-6 for additional guidance.