Designation: E3281 − 21a

Standard Guide for NAPL Mobility and Migration in Sediments – Screening Process to Categorize Samples for Laboratory NAPL Mobility Testing[1]

This standard is issued under the fixed designation E3281; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide is designed for general application at a wide range of sediment sites where non-aqueous phase liquid (NAPL) is present or suspected to be present in the sediment. This guide describes a process to use field screening methods, specifically visual observations, and the results of shake tests, to categorize the relative amount of NAPL present in a sample. This categorization can then be utilized to select co-located sediment samples for laboratory testing to determine if the NAPL in the sample interval is mobile or immobile at the pore scale, or any other chemical or physical testing.

1.1.1 There is no current industry standard methodology to select sediment samples for laboratory NAPL mobility testing; the use of different methodologies is possible. This guide focuses on a selection process that uses visual observations and shake tests. This process has the advantage of being simple to use and, if applied in a disciplined manner, has been demonstrated to provide good results in the field.

1.2 This guide is intended to inform, complement, and support characterization and remedial efforts performed under international, federal, state, and local environmental programs but not supersede local, state, federal, or international regulations. The users of this guide should review existing information and data available for a sediment site to determine applicable regulatory agency requirements and the most appropriate entry point into and use of this guide.

1.3 ASTM International (ASTM) standard guides are not regulations; they are consensus standard guides that may be followed voluntarily to support applicable regulatory requirements. This guide may be used in conjunction with other ASTM guides developed for assessing sediment sites.

1.4 This guide does not address methods and means of sample collection (Guide E3163).

1.5 *Units*—The values stated in SI or CGS units are to be regarded as the standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)
D2488 Practice for Description and Identification of Soils (Visual-Manual Procedures)
D7203 Practice for Screening Trichloroethylene (TCE)-Contaminated Media Using a Heated Diode Sensor
E2531 Guide for Development of Conceptual Site Models and Remediation Strategies for Light Nonaqueous-Phase Liquids Released to the Subsurface
E2856 Guide for Estimation of LNAPL Transmissivity
E3163 Guide for Selection and Application of Analytical Methods and Procedures Used during Sediment Corrective Action
E3248 Guide for NAPL Mobility and Migration in Sediment – Conceptual Models for Emplacement and Advection
E3268 Guide for NAPL Mobility and Migration in Sediment—Sample Collection, Field Screening, and Sample Handling
E3282 Guide for NAPL Mobility and Migration in Sediments – Evaluation Metrics

[1] This guide is under the jurisdiction of ASTM Committee E50 on Environmental Assessment, Risk Management and Corrective Action and is the direct responsibility of Subcommittee E50.04 on Corrective Action.

Current edition approved Oct. 1, 2021. Published November 2021. Originally approved in 2021. Last previous edition approved in 2021 as E3281–21. DOI: 10.1520/E3281–21A

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

F2534 Guide for Visually Estimating Oil Spill Thickness on Water

3. Terminology

3.1 *Definitions:*

3.1.1 *immobile NAPL, n*—NAPL that does not move by advection within the connected void spaces of the sediment under specified physical and chemical conditions, as may be demonstrated by laboratory testing, or may be interpreted based on mathematical calculations or modeling. **E3248**

3.1.2 *mobile NAPL, n*—NAPL that may move by advection within the connected void spaces of the sediment under specific physical and chemical conditions, as may be demonstrated by laboratory testing, or as may be interpreted based on mathematical calculations or modeling. **E3248**

3.1.3 *non-aqueous phase liquid, NAPL, n*—chemicals that are insoluble or only slightly soluble in water that exist as a separate liquid phase in environmental media. **E3248**

3.1.3.1 *Discussion*—NAPL may be less dense than water (light non-aqueous phase liquid [LNAPL]) or more dense than water (dense non-aqueous phase liquid [DNAPL]).

3.1.4 *pore scale, n*—the scale of the connected void spaces within the sediment. **E3248**

3.1.5 *sediment(s), n*—a matrix of pore water and particles including gravel, sand, silt, clay, and other natural and anthropogenic substances that have settled at the bottom of a tidal or nontidal body of water. **E3163**

3.1.6 *sheen, n*—a silvery, rainbow, or dark rainbow film on the surface of the sediment sample or on a water surface.

4. Significance and Use

4.1 NAPLs (for example, chlorinated solvents, petroleum products, and creosote) can be emplaced in sediments through a variety of mechanisms (Guide E3248). Dense non-aqueous phase liquids (DNAPLs) are more dense than water, whereas light non-aqueous phase liquids (LNAPLs) are less dense than water.

4.2 Standardized guidance and test methods currently exist for assessing NAPL mobility at upland sites, from organizations such as ASTM (Guides E2531 and E2856), Interstate Technology & Regulatory Council (**1**)[3] and the American Petroleum Institute (**2, 3**).

4.3 Guide E3248 provides guidance regarding when a NAPL movement evaluation is warranted. After confirming that NAPL is present and evaluating nature and extent as appropriate, the next step in any NAPL movement evaluation is to evaluate if NAPL is mobile or immobile at the pore scale—this is done using tiered or weight of evidence (WOE) approaches. This guide provides a structured process to select samples to submit to the laboratory for NAPL mobility testing that is part of a NAPL movement evaluation.

4.4 This guide may be used by various parties involved in sediment corrective action programs, including regulatory agencies, project sponsors, environmental consultants, toxicologists, risk assessors, site remediation professionals, environmental contractors, and other stakeholders.

4.5 This guide should be used in conjunction with other reference material (refer to Section 2 and References) that direct the user in developing and implementing sediment assessment programs.

4.6 This guide is related to Guide E3163, concerning sediment analytical techniques used during sediment programs. This relates to Guide E3248, which discusses generic models for the emplacement and advection of NAPL in sediments. It is related to Guide E3268, which describes sample collection, field screening and sample handling considerations in NAPL movement evaluations. And this is related to Guide E3282, which describes evaluation metrics and frameworks to determine if NAPL is immobile or immobile at the pore scale, or if it is migrating or stable at the NAPL body scale.

4.7 This guide does not replace the need for engaging competent persons to evaluate NAPL emplacement and movement in sediments. Activities necessary to develop a conceptual site model should be conducted by persons familiar with NAPL-impacted sediment site characterization techniques, physical and chemical properties of NAPL in sediments, fate and transport processes, remediation technologies, and sediment evaluation protocols. The users of this guide should consider assembling a team of experienced project professionals with appropriate expertise to scope, plan, and execute sediment NAPL data acquisition activities.

4.8 This guide provides a framework based on overarching features and elements that should be customized by the user, based on site-specific conditions, regulatory context, and program objectives for a particular sediment site. This guide should not be used alone as a prescriptive checklist.

4.9 The user of this guide should review the overall structure and components of this guide before proceeding with use, including:

Section 1	Scope
Section 2	Referenced Documents
Section 3	Terminology
Section 4	Significance and Use
Section 5	Summary of the Process for Screening and Selection of Samples for Laboratory NAPL Mobility Testing
Section 6	Methods for Recording Visual Observations of Sheen and NAPL in Sediment Samples
Section 7	Methods for Performing Shake Testing of Sediment Samples
Section 8	Categorizing the Relative Presence of NAPL in Sediment
Section 9	Use of NAPL Categorization Results to Select Existing Samples or Identify Locations and Depths for Collecting Additional Undisturbed Samples for Laboratory NAPL Mobility Testing
Section 10	Other Methods to Select Samples for Laboratory NAPL Mobility Testing
Section 11	Keywords
Appendix X1	Recommended Procedure for Visually Characterizing Sediment for Sheen or NAPL Observations
Appendix X2	Recommended Procedure for a Sediment-Water Shake Test
Appendix X3	Case Study
References	

5. Summary of the Process for Screening and Selection of Samples for Laboratory NAPL Mobility Testing

5.1 One key factor that typically influences the potential for NAPL mobility of advectively emplaced NAPL is the NAPL

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

saturation (that is, the percentage of the total pore space that is filled with NAPL); the distribution of NAPL within the pores also has an effect on the mobility of the NAPL (that is, a relatively small amount in NAPL within the largest pores can produce mobility). Generally, the potential for NAPL mobility is greater in sediments containing relatively more NAPL and less in sediments containing relatively less NAPL; for depositionally emplaced NAPLs, the mobility is also strongly influenced by the degree of encapsulation of the NAPL. Therefore, this guide offers a process for qualitatively categorizing the relative amount of NAPL present in sediments. This information is then used to select locations and depth intervals for laboratory NAPL mobility testing.

5.2 There are two ways in which the categorization process presented in this guide can be used to select locations and depth intervals for laboratory NAPL mobility testing.

5.2.1 In the first method, often used in sediment investigations, grab samples of surface sediment and core samples of subsurface sediment are collected to determine the nature and extent of NAPL. Once the relative amount of NAPL in various areas and depths within the sediment has been categorized using the process described in this guide, NAPL mobility sampling is performed at targeted locations during a subsequent sampling event.

5.2.1.1 The advantage of this approach is that previously collected data can be used to select targeted locations and general depths for laboratory NAPL mobility testing, so the subsequent NAPL mobility sampling is focused and efficient.

5.2.1.2 A disadvantage to this approach is that multiple sampling events are necessary, which could extend the time required to complete the site investigation. In some cases, depending on site-specific conditions (for example, difficult access, small sampling area) and the number of cores to be collected, this approach could be more expensive than the second method.

5.2.2 In the second method, whose use depends on site-specific conditions, the approach is to collect multiple co-located samples at each sample location during a single sampling event. With this approach, one core is collected to determine the nature and extent of NAPL. Additional cores from the same sampling location are archived and preserved in the original core liners to provide co-located samples for subsequent laboratory NAPL mobility testing. Once the relative amount of NAPL in various areas and depths of sediment has been characterized, using the methods described in this guide, NAPL mobility sampling is performed at targeted locations and depths in the archived co-located samples.

5.2.2.1 One advantage of this approach is that laboratory NAPL mobility test results can be obtained more quickly, because sample material is already available. This approach generally also has the advantage of being performed in only one mobilization.

5.2.2.2 The disadvantage of this approach is that it requires collecting co-located samples that can be used for laboratory NAPL mobility testing at every sampling station during the initial investigation of the nature and extent of NAPL. Because many of the co-located samples would not undergo laboratory testing for NAPL mobility, this approach is less efficient and can add considerable expense to the investigation program.

5.3 The process for screening and selecting locations and general depths for laboratory NAPL mobility testing as presented in this guide consists of the four major steps summarized in Fig. 1 and discussed in detail in 5.4. This process is typically performed in the field, but there is nothing precluding the process being applied to sediment samples elsewhere (for example, the consultant's office or at a laboratory).

5.4 In this process, sediment samples are screened for the presence of NAPL using a standardized methodology consisting of visual observations (*Step 1*) and sediment-water shake

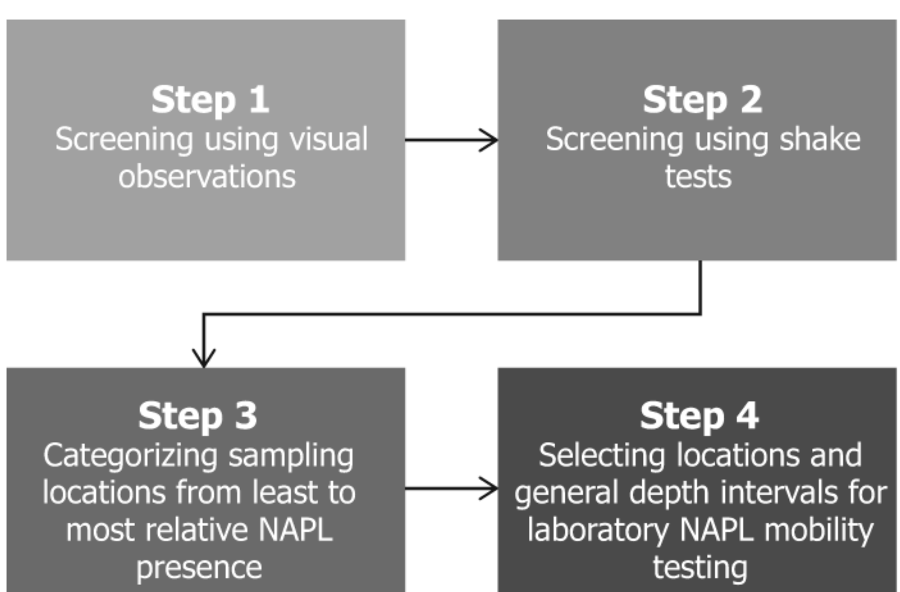

FIG. 1 Summary of the Process for Screening and Selection of Samples for Laboratory NAPL Mobility Testing

testing (*Step 2*); the relative amount of NAPL is then categorized in *Step 3*; finally, decisions regarding the selection of locations and general depths for laboratory NAPL mobility tests are made in *Step 4*. Exact depths of NAPL mobility test samples are usually selected based on detailed photography of cores collected specifically for NAPL mobility testing.

5.4.1 *Step 1*—A visual observation refers to the appearance of sheen or NAPL, if present, on and within the sediment sample. Methods and standard terminology for recording visual observations of sheen and NAPL in sediment samples are described in Section 6, and a recommended standardized visual observation procedure is provided in Appendix X1.

5.4.2 *Step 2*—A sediment-water shake test is a method for screening sediments for the presence of NAPL. Aliquots of sediment and water are placed in a clear container and gently shaken; the observation of sheen or NAPL is documented. Methods for performing shake tests to confirm the presence of sheen or NAPL in sediment samples and standard terminology for recording shake test results are described in Section 7, while a recommended standardized shake test procedure is provided in Appendix X2.

5.4.3 *Step 3*—Visual observations (*Step 1*) and shake test results (*Step 2*) are compiled to categorize the relative amount of NAPL present in each sediment core or grab sample, from least to most NAPL. Sediment cores/grabs are assigned NAPL categories, ranging from Category 1 (no sheen or NAPL present in the sample) through Category 4 (the greatest relative NAPL presence). The process of categorizing the relative presence of NAPL in sediment, based on shake test results, is described in Section 8.

5.4.4 *Step 4*—Based on the results of the NAPL categorization (Step 3), locations and general depth intervals are selected for laboratory NAPL mobility testing. This testing is performed either on cores collected during a subsequent targeted sampling program (5.2.1) or from co-located cores that were previously collected and archived in the original core liners (5.2.2). The use of NAPL categories to select locations and general depth intervals for laboratory NAPL mobility testing is discussed in Section 9.

5.4.4.1 The recommended approach is to evaluate NAPL mobility across a range of NAPL conditions, with sampling and testing biased towards locations and depths with relatively more NAPL (for example, Category 4), while also performing some testing at locations and depths with less NAPL (for example, Category 3) or even sheen only (that is, Category 2). For sites with more than one major sediment lithologic unit, ensure that sufficient representative samples are obtained from each unit.

5.4.4.2 The screening process described in this guide requires disrupting the sediment matrix, so sediment that has been used for visual observation of NAPL presence (for example, the sediment core has been split) or undergone a shake test cannot be used for NAPL mobility testing. Typically, a sediment core collected for NAPL mobility testing would include up to several meters of intact core material for submittal to the laboratory for core photography to select the specific depth(s) of the NAPL mobility test sample(s).

5.4.4.3 Differences in mudline elevation and percent recovery should be accounted for when trying to obtain samples from the same interval in co-located cores. Additionally, the sediment lithology should be examined to ensure that it is comparable for the two intervals from the co-located cores.

5.5 The screening process to categorize samples for laboratory NAPL mobility testing provided in this guide offers the following benefits:

5.5.1 The use of a standard method for screening sediment samples for the presence of NAPL reduces variability in the reporting of NAPL visual observation data and facilitates comparing NAPL presence and relative abundance in sediment across the sediment site. The use of a standard method for screening sediment samples for NAPL presence becomes particularly important when attempting to compare and interpret NAPL observation data collected throughout multiple investigations or by multiple parties.

5.5.2 The use of a standard shake test method to complement visual observations increases the validity of the visual observation data and provides a less qualitative measure of the relative amount of NAPL in each shake-test sample than visual observations alone.

5.5.3 Categorizing sediment sampling locations based on the relative amount of NAPL present in the sediment enables easy identification of areas with relatively more NAPL presence and relatively less NAPL presence when selecting areas for NAPL mobility sampling or existing samples for laboratory NAPL mobility testing. For sites with more than one sediment lithologic unit, it is preferable that the categorization results for all units be pooled, if possible. However, in some cases, it may be necessary to separately evaluate categorization results for each lithological unit.

6. Methods for Recording Visual Observations of Sheen and NAPL in Sediment Samples

6.1 This section summarizes methods for systematically describing the visible characteristics of sheen and NAPL in sediment and how to document those observations in a consistent manner using defined terminology.

6.1.1 The basic procedures for visually characterizing sheen and NAPL in sediment can be applied to sediment core or grab samples. A detailed methodology for visually characterizing and recording sheen and NAPL observations in sediment is included in Appendix X1.

6.1.2 These procedures are performed in addition to standard core logging, which includes (but is not limited to) a description of sediment lithology, moisture content, density/consistency of sediment, and color across the entire length of the core or sample (for guidance see Practices D2487 and D2488).

6.2 Visual observations of sheen and NAPL presence or absence, as well as the distribution of the visual observations within the sediment matrix, can be described using the terminology defined in Table 1.

6.3 Where sheen is observed in the sediment core or grab sample, the start and end depths for each unique observation are recorded, and the sheen color and relative amount of sheen

TABLE 1 Sheen and NAPL Observation Terminology

Standard Terminology	Appearance
No visual evidence of NAPL	No sheen or NAPL is observed.
Sheen	A sheen is present, but NAPL is not observed.
Blebs	Discrete droplets of NAPL are observed, but for the most part, the sediment matrix is not visibly contaminated or saturated. Typically, this is immobile NAPL.
Coated	Sediment grains are coated with NAPL. There is not sufficient NAPL present to saturate the pore spaces.
Saturated	The entirety of the pore space for a sample appears to be saturated with NAPL. Care should be taken to ensure that water saturating the pore spaces is not misinterpreted as NAPL when using this term (for example, use a paper towel to see if liquid wicks like water). Depending on the NAPL viscosity, NAPL may freely drain from a sediment sample.

observed are documented using the recommended methods and terminology provided in X1.4. When any aspect of a visual observation of sheen changes (for example, the relative amount observed changes), new start and end depths are recorded.

6.3.1 Sheen color can be described using the terminology provided in Table X1.1, which is modified from Guide F2534.

6.3.2 The relative amount of sheen observed in the sample is estimated by comparing the relative sample surface area with a sheen to standard comparison charts for visual estimates, such as those provided in Fig. 2 (**4**) and selecting the appropriate modifier from Table X1.2.

6.4 Where NAPL is observed in the sediment core or grab sample, the start and end depths for each unique observation are recorded; the NAPL color, viscosity, and relative amount are documented using the recommended methods and terminology provided in X1.5. When any aspect of a NAPL visual observation changes (for example, the relative amount observed changes), new start and end depths are recorded.

6.4.1 Relative NAPL viscosity can be described using the terminology provided in Table X1.4.

6.4.2 Similar to sheen observations, the relative amount of NAPL observed in the sample is estimated by comparing the relative sample surface area covered with NAPL to standard

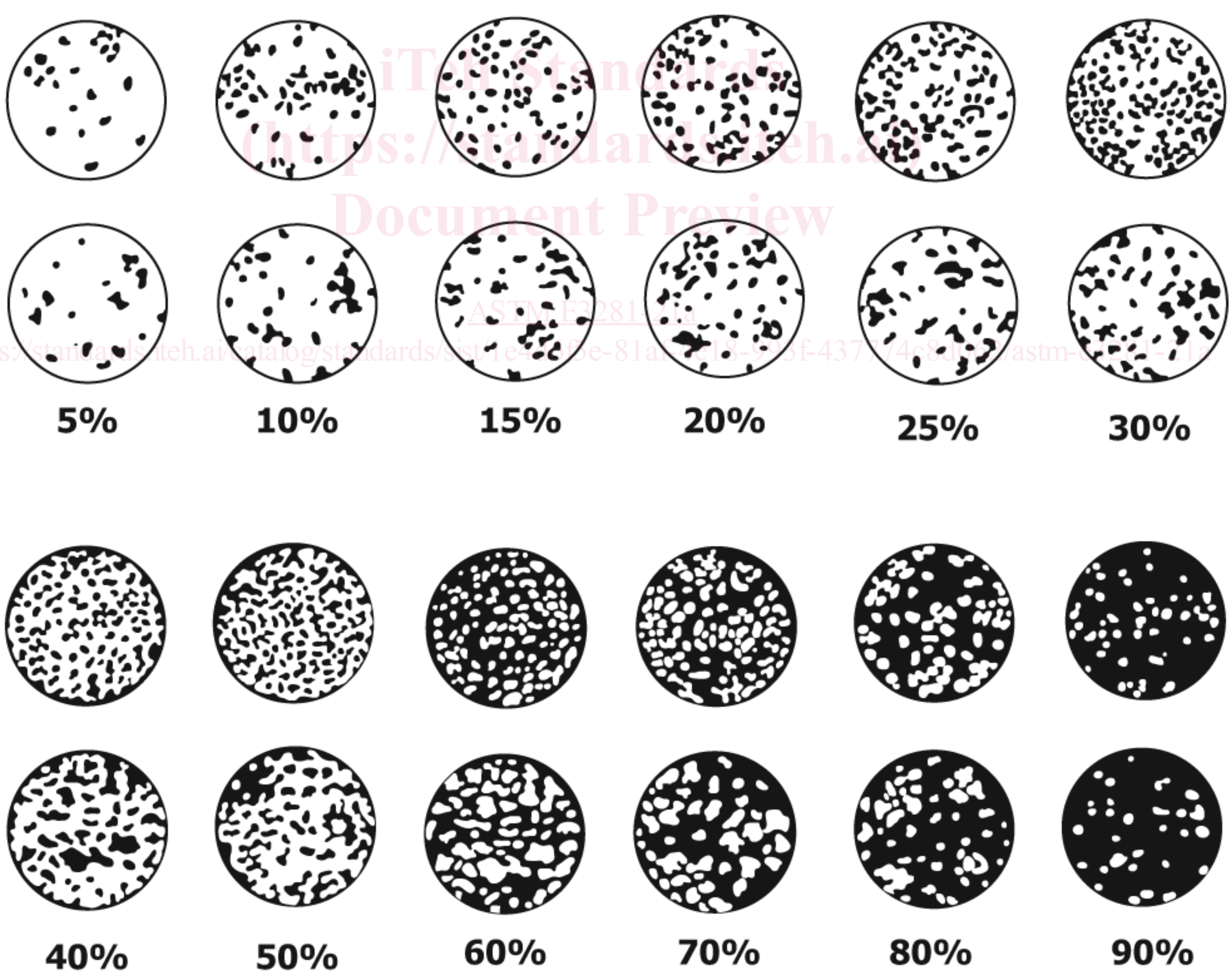

FIG. 2 Visual Estimate Guide

comparison charts for visual estimates, such as those provided in Fig. 2, and selecting the appropriate modifier from Table X1.2.

6.5 After the visual appearance of sediment is described, cores and grab samples are photographed for project documentation and later reference. Section X1.3 provides a standard method for photographing a sediment core; photographs of the core are taken in 0.3-m to 1.0-m increments (typically, the shorter increments are used to provide more detail), with a scale or tape measure placed next to the core/sample and included in the photograph to indicate core depth. It is recommended that photographs be taken straight on, with the core in the horizontal position and with the shallower depth on the left and the deeper depth on the right. It is recommended that sediment cores and grabs are photographed in a well-lit area with natural light.

6.6 For quality control purposes, sheen and NAPL visual observations, as well as the start and end depths of these visual observations, should be double-checked for accuracy and consistency by a second trained person.

7. Methods for Performing Shake Testing of Sediment Samples

7.1 This section provides an overview of sediment-water shake testing, including methods for selecting sample material to shake test, performing the shake test, and consistently documenting the shake test results using defined terminology. A sediment-water shake test is a method for screening sediments for the presence or absence of NAPL. Aliquots of sediment and water are added to a clear container and shaken, then the relative amount of sheen or NAPL is observed.

7.1.1 Shake testing is considered an effective indicator of NAPL presence in a sediment sample, because the NAPL that may be distributed in the sample, but is not observable, can accumulate as sheens, blebs, or layers (as a direct result of the sediment agitation) after the sediment matrix is disrupted. While uncommon, if the NAPL present is colorless, adding a hydrophobic dye to the sample before the shake test may be necessary.

7.1.2 Although a shake test is a useful indicator of NAPL presence and relative amount, a shake test is not a reliable indicator of NAPL density; either LNAPL or DNAPL typically forms a layer between the air and water or coats the walls of the sample container because of interfacial tension. Shake tests also are not a reliable indicator of NAPL mobility; the observed presence of NAPL in a shake test does not provide any evidence of whether NAPL is mobile at the pore scale in situ.

7.1.3 It is possible that shake test results may vary on a site-specific basis. One of the contributing factors to this could be the emplacement mechanism of the NAPL (for example, advective versus depositional emplacement).

7.1.4 A detailed procedure for conducting sediment-water shake tests is included in Appendix X2 and summarized in 7.2 through 7.10.

7.2 Upon retrieval of a sediment core or grab sample, visual observations of sheen or NAPL are documented as described in Section 6 and Appendix X1. Based on the visual observations, the depth at which to shake test is determined by selecting one or more sampling intervals with the most notable visual presence of sheen or NAPL using the following sequence (in increasing order):

7.2.1 silvery sheen < rainbow sheen < dark rainbow sheen < NAPL blebs < NAPL coated < NAPL saturated

7.3 If sheen or NAPL is not observed, select one or more depth intervals for shake tests based on other criteria, such as changes in lithology, sediment discoloration, or elevated photoionization detector (PID) readings. Due to the relatively small amount of sediment used when performing a shake test, the shake test should be performed at a specific depth and not over a wider depth interval.

7.3.1 If visual observations in a sediment core or grab sample consist of more than one visual observation type (that is, blebs, coated, saturated, or sheen), the recommended practice is to administer one shake test from a representative sample for each visual observation type in the interval where the most notable visual presence of sheen or NAPL for that observation type is observed (that is, one shake test in a sheen interval with the greatest degree of sheen, one shake test in a NAPL bleb interval with the greatest degree of NAPL blebs, one shake test in a NAPL-coated interval with the greatest degree of NAPL coating, and one shake test in a NAPL-saturated interval with the greatest degree of NAPL saturation). See X2.3 for additional detailed guidance for determining which intervals to perform shake tests on.

7.4 To perform a shake test, a consistent ratio of sediment and water (see X2.4) is added to a clear container, gently shaken, and allowed to equilibrate before documenting observations. For shake test results to be comparable, each shake test must be performed in the same way using the same type of jar (for example, size, shape, material, and lid), the same amounts of sediment and water, the same intensity and time of shaking, and the same amount of time for phase separation after shaking. A recommended methodology for shake testing is described in detail in Appendix X2.

7.5 To determine the shake test result, observe the shake test jar sidewalls and water surface for the presence of sheen, NAPL blebs, or a NAPL layer. Describe the shake test results using the standard terminology in Table 2. Note that non-NAPL material (for example, organic material, ash, miscellaneous flocculent) can float on the water surface or adhere to the sidewalls of the shake test container, potentially confounding the shake test result.

7.6 When sheen is observed, record the sheen color using the terminology for sheen color (Table X1.1) and estimate the relative amount of sheen present by comparing the relative surface area of the water surface covered with a sheen to standard comparison charts for visual estimates (Fig. 2).

7.7 When shake test blebs are observed, estimate the relative amount of NAPL present (that is, percent bleb coverage) by comparing the relative surface area of the jar sidewalls and water surface covered with NAPL to standard comparison charts for visual estimates (Fig. 2). Also, record the NAPL color.

TABLE 2 Shake Test Result Terminology

Shake Test Result	Appearance
Negative	No sheen or NAPL is observed. A negative shake test result indicates that sheen and NAPL are not present in the sample tested.
Shake test sheen	A sheen is present on the surface of the water, but no NAPL blebs or NAPL layer is observed. A sheen is a silvery, rainbow, or dark rainbow film on the surface of water.
Shake test blebs	Discrete droplets of NAPL are present on the sidewalls of the shake test jar, on the water's surface, or suspended in the water.
Shake test layer	NAPL appears as a distinct layer within the shake test jar, on the water surface. NAPL may also be present on the sidewalls of the shake test jar.

7.8 When a shake test layer is observed, estimate the NAPL thickness (if possible) and record the color. Due to the potential volume of NAPL generated in a shake test with a layer result, it may be difficult to see the NAPL layer, because the shake test jar walls may be covered by NAPL.

7.9 After describing the shake test results, shake tests are photographed for project documentation and later reference (including shake test bleb ranking, if performed, as described in 8.4). Section X2.5.5 provides a standard method for photographing the shake test container without the lid from two angles: vertically (that is, from the top, looking down into the shake test jar at the water surface) and horizontally (that is, from the side, looking at the shake test jar in profile). Photograph shake tests in a well-lit area, preferably with natural light. To create a clear contrast between the contents of the shake test jar and the background, photograph the shake test jar on a white surface with a white background.

7.10 After the shake test result has been described and photographed, compare visual observations from the representative sheen or NAPL observation interval to the shake test result. If the shake test result does not corroborate the visual observations for the sample depth interval, check the shake test result to confirm that the correct result was recorded. Then, review the core interval and ensure that the initial visual observation was correct; update if warranted.

7.11 Confirm a shake test was conducted each time a change in visual categorization (as defined in Table 1) was observed in the sediment core or grab.

7.12 After shake test results have been checked and photographed and the visual observations for the corresponding depth interval in the sediment sample have been corroborated against the shake test result, the shake test jar and sediment sample can be properly discarded. New jars should be used for subsequent shake tests.

7.13 For quality control purposes, conduct one shake test blank per day using only water to ensure that the shake test container batch is not biasing results. Field duplicate shake tests should be performed at a frequency of 5 % to 10 % of the shake tests. Field documentation of shake tests should be double-checked by a second trained person for accuracy and completeness.

8. Categorizing the Relative Presence of NAPL in Sediment

8.1 After completing the screening process described in Sections 6 and 7, shake test results are used to assign a NAPL category for each sampling location (Fig. 3), based on the relative amount of NAPL present (Table 3). The purpose of assigning NAPL categories is to readily identify areas of more, less, or no NAPL presence, to aid in the selection of locations

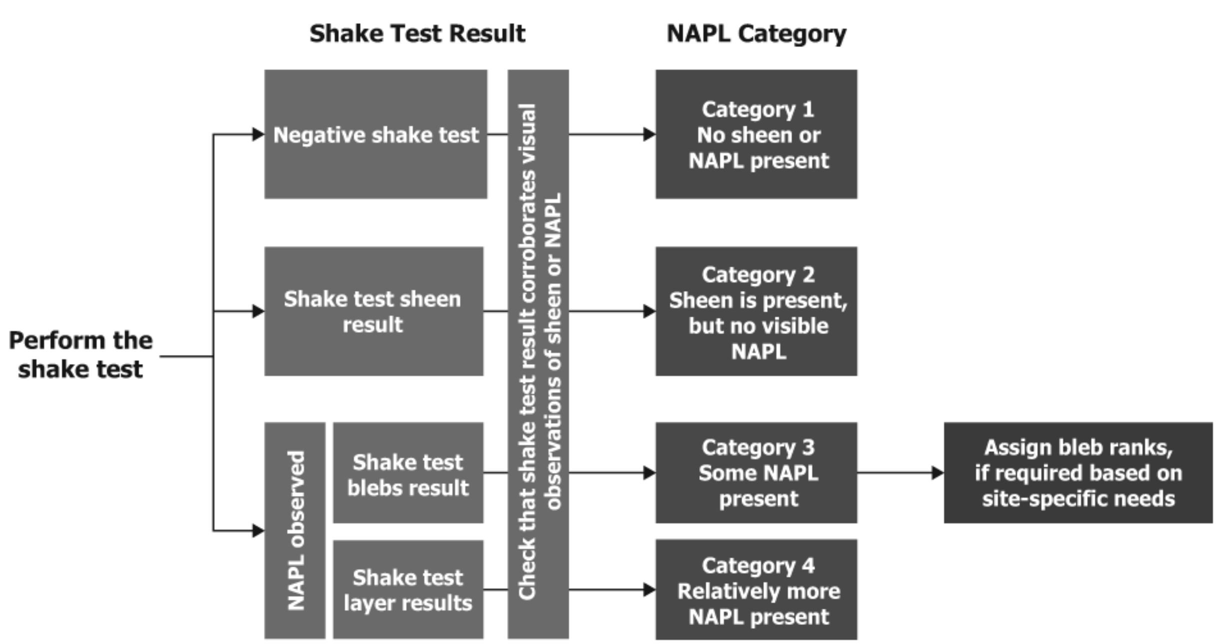

FIG. 3 Assigning NAPL Categories from Shake Test Results

TABLE 3 NAPL Shake Test Categories

NAPL Category	Criteria
Category 1	Sample locations with negative shake test results, indicating no sheen or NAPL is present
Category 2	Sample locations with shake test sheen results, indicating the presence of sheen, but no visible NAPL
Category 3	Sample locations with shake test bleb results, indicating the presence of some visible NAPL
Category 4	Sample locations with shake test layer results, indicating the presence of relatively more NAPL

and depth intervals to collect cores for laboratory NAPL mobility testing. These laboratory results can then be used in frameworks to determine if the NAPL is mobile at the pore scale (see Guide E3282).

8.2 NAPL categories can be assigned on a core or grab sample location basis, based on the specific lithology type (for example, recent deposits vs. glacial till), to a specific depth (for example, surface sediment vs. subsurface sediment), or based on any other site-specific metric.

8.3 In the example described in this section, NAPL categories are assigned based on the shake test result containing the greatest relative amount of NAPL for a given core or grab sample location, so that the NAPL category represents the greatest relative amount of NAPL at any depth at a given location. When assigning a NAPL category, select the shake test with the greatest relative amount of NAPL using the following sequence (in increasing order):

8.3.1 negative shake test < shake test sheen < shake test blebs (all ranks) < shake test layer

8.3.1.1 The NAPL categories, from least to greatest NAPL presence, are assigned as summarized in Fig. 3 and described in Table 3.

8.3.2 Given that the relative amount of NAPL present in a shake test jar with blebs can vary significantly, Category 3 cores/grabs can be further evaluated based on the degree of NAPL accumulation in the shake test jar (that is, shake test bleb ranking), from least to most NAPL. The shake test bleb ranking process is described in 8.4.

8.3.3 For example, consider a core with visual observations of none, sheen, and NAPL saturated at different depth intervals, with corresponding shake test results of negative, sheen, and layer, respectively. The visual observation containing the greatest relative amount of NAPL for this core is NAPL saturated. The shake test result containing the greatest relative amount of NAPL for this core is the shake test layer, which was associated with the NAPL saturated visual observation. The NAPL category for this core would be assigned based on the shake test layer result, which would result in this core being assigned to Category 4.

8.4 Depending on site-specific conditions, shake test bleb ranking is recommended to further evaluate Category 3 sample locations, based on the relative amount of NAPL present. Shake test bleb ranking consists of using estimates of the percent bleb coverage in the shake test jar to rank the percent coverage of blebs present in the shake tests and includes a check across the entire shake test dataset to confirm consistency in the ranking. Category 3 sample locations with relatively more NAPL presence can then be readily distinguished from Category 3 sample locations with relatively less NAPL presence. As shown in Fig. 3, shake test bleb ranking is performed after the field investigation is complete and NAPL categories have been assigned, so the shake test dataset can be evaluated as a whole. The need to further evaluate Category 3 sample locations based on the relative amount of NAPL present should be determined on a site-specific basis. The shake test bleb ranking process is described in 8.4.1 through 8.5.1.

8.4.1 Shake test bleb ranking will be useful if there are many Category 3 locations but no Category 4 locations (that is, shake testing yields many shake test blebs results, but no shake test layer results) or if the amount of NAPL observed in Category 3 shake tests varies significantly.

8.4.2 To perform shake test bleb ranking, at least two (but preferably three) trained personnel should review the shake test photographs, shake test field logs, and the sediment core or grab sample logs for Category 3 locations to estimate the percent coverage of blebs present on the shake test water surface and adhered to the jar walls (that is, percent bleb coverage) to the nearest 5 %. This is done using standard comparison charts for visual estimates, such as those provided in Fig. 2.

8.4.2.1 The percent bleb coverage recorded, when the shake test results were logged (see 7.6), can be used for one of the three estimates.

8.4.2.2 When reviewing shake test photographs, note that water in shake tests can range in appearance from clear to darkly colored or opaque, due to the composition of the shake tested sediment (for example, organic silt, clay, sand). Organic and anthropogenic material in sediment can appear as solids floating on the water in the shake test jar or adhered to shake test jar walls. Therefore, shake test photographs should be interpreted taking into consideration the sediment type, visual observations of sheen or NAPL in the sediment sample and the shake test results documented on the shake test field logs.

8.4.3 If all the bleb coverage estimates by the trained personnel are within 20 %, use the highest estimate to assign the bleb rank. If the bleb coverage estimates vary by more than 20 %, the reviewers should re-evaluate the shake test photographs and field information to develop consensus on the bleb coverage estimate. The consensus bleb coverage estimate is then used to assign the shake test bleb rank.

8.4.4 Before finalizing the shake test bleb ranks, the following quality assurance measures should be taken to establish consistency in rank estimates across the entire shake test dataset:

8.4.4.1 Compile and compare shake test photographs within each bleb rank for consistency.

8.4.4.2 Compile and compare shake test photographs between bleb ranks to confirm that increasing bleb ranks consistently reflect increasing quantities of bleb coverage.

8.4.5 See the case study (Appendix X3) for an example of shake test bleb ranking.

8.5 Depending on site-specific conditions or program objectives, it may be useful to develop a relationship between visual observations and shake test results, to infer the relative NAPL quantity in intervals that were not shake tested and had only visual observations made on them.

8.5.1 The relationship between visual observations and shake test results should be assessed on a site-specific basis, by reviewing the results of the visual observation and shake test screening data to confirm the presence of a consistent relationship. An example of the site-specific relationship between these two screening methods is presented in the case study (Appendix X3).

9. Use of NAPL Categorization Results to Select Existing Samples or Identify Locations and Depths for Collecting Additional Undisturbed Samples for Laboratory NAPL Mobility Testing

9.1 The results of the NAPL categorization discussed in Section 8 can be used to select locations and depth intervals for collection of undisturbed samples in a subsequent sampling program, or to choose existing samples for laboratory NAPL mobility testing; this testing will evaluate if NAPL present in the sediment sample is mobile or immobile at the pore scale. Objectives for laboratory NAPL mobility testing will vary based on site-specific conditions and data needs. However, the approach for characterizing the amount of NAPL in a sample (which provides some indication of the potential for NAPL mobility) in this guide is general, suitable for a range of sites, and adaptable to site-specific conditions and data needs.

9.2 As noted in Section 5, NAPL saturation (that is, the percentage of the total pore space that is filled with NAPL) influences the potential for NAPL mobility via advection in sediment. Generally, the potential for NAPL mobility is greater in sediments with relatively more NAPL and less in sediments with relatively less NAPL; in depositionally emplaced NAPLs, the mobility is also strongly influenced by the degree of encapsulation of the NAPL. Therefore, the recommended approach is to collect more samples for laboratory NAPL mobility testing at locations and depths where screening has indicated a higher potential for mobile NAPL (that is, locations and depths with relatively more NAPL, such as Category 4 locations) and fewer samples at locations and depths with lesser amounts of NAPL (that is, such as Category 3 and Category 2 locations), where NAPL is more likely immobile. Recommendations on designing a NAPL mobility sampling program to achieve these objectives are provided in 9.2.1 through 9.2.3.

9.2.1 It is recommended that NAPL mobility sampling locations be biased towards areas or depths with a higher potential for NAPL mobility.

9.2.2 NAPL mobility sampling locations should also provide spatial coverage across areas with a higher potential for mobile NAPL (for example, Category 4 areas, and if bleb ranking is performed, Category 3 areas with greater than 50 % bleb coverage) and areas with lesser amounts of NAPL where NAPL is likely immobile (for example, Category 3 areas; if bleb ranking is performed, Category 3 areas with less than 50 % bleb coverage; Category 2 areas).

9.2.2.1 If different types of NAPL (that is, different physical or chemical characteristics) are observed, sampling locations should provide spatial coverage of each.

9.2.3 The majority of NAPL mobility test samples should be collected from locations where cores were collected and processed for both visual observations and shake tests, because the results will enable a comparison between the NAPL categories assigned using this guide and NAPL mobility test results.

9.2.4 The screening process described in this guide requires disturbing the sediment matrix, so the sediment sample that has been used for visual observations and shake testing is not suitable for NAPL mobility testing. Typically, an intact co-located sediment core collected for NAPL mobility testing would be retained in the original core liner and would include up to several meters of core material. The core would be cut into increments suitable for shipping and submitted to the laboratory for core photography to select the specific depths for the NAPL mobility test samples.

9.3 Appendix X3 presents a case study illustrating the application of this selection process and a NAPL mobility investigation and evaluation designed using the approach outlined in this guide.

10. Other Methods to Select Samples for Laboratory NAPL Mobility Testing

10.1 Each of the technologies described in this section can be used to identify NAPL presence, and the magnitude of response can be used to interpret relative amount of NAPL in sediment. In general, locations and depths with the greatest relative amount of NAPL should be preferentially targeted for NAPL mobility testing. Additional NAPL mobility testing can be performed at locations and depths with less NAPL presence to provide NAPL mobility test results across a range of conditions.

10.2 *Ultraviolet (UV) Light Screening:*

10.2.1 Petroleum hydrocarbon, creosote, and coal tar NAPLs are partially composed of individual polycyclic aromatic hydrocarbons (PAHs) of varying concentrations. PAHs will generally fluoresce under excitation by UV light. The intensity of the fluorescence response may provide a qualitative indication of the relative magnitude of NAPL in a sample but can also vary depending upon other factors, including grain size and NAPL type. The color of fluorescence may provide an indication of PAH composition (PAH mixtures principally composed of two aromatic rings will generally appear blue shifted, whereas mixtures principally composed of five aromatic rings will generally appear red shifted) (**5**).

10.2.2 Because NAPL can be difficult to visually identify in dark-colored sediments, field or laboratory screening of a sediment core with UV light can provide useful information regarding the presence, distribution, and primary composition of NAPL within the sediment core. Practitioners should be aware that naturally occurring minerals (for example, calcite), shells, wood, algae, or organic-rich material (for example, peat) can also fluoresce when excited by similar wavelengths as NAPL-containing PAHs. If used, UV fluorescence responses should be combined with other lines of evidence to inform selection of sediment core intervals for NAPL mobility testing.

10.3 *Laser-Induced Fluorescence:*

10.3.1 Laser-induced fluorescence (LIF) is a semiquantitative technology that uses laser excitation to induce fluorescence of PAHs and other fluorescing material present in the sediment matrix and record the color and intensity of the fluorescence response. An increased magnitude of fluorescence response is typically associated with increased PAH concentrations in the sediment, which are in turn associated with an increased amount of NAPL in the sediment. The wavelengths of fluorescence emitted by fluorescing material in the sediment matrix vary and can be used to differentiate between different types of fluorescing material, such as NAPLs with different compositions.

10.3.2 When using a site-specific correlation, increased magnitude of fluorescence response can be correlated to the amount of NAPL in the sediment (that is, relative abundance), and the wavelengths emitted can be used to differentiate NAPL types and interferences. LIF with visible light excitation (usually a green wavelength) is typically used to identify NAPL dominated by larger PAH molecules, such as coal tars and creosotes. LIF with UV light excitation is typically used to identify petroleum NAPL, such as gasoline and diesel.

10.3.3 In practice, LIF probes are typically advanced using direct-push technology to provide a vertical log of fluorescence intensity in the sediment column versus depth. The fluorescence intensity by itself can be used to determine relative PAH concentration and varies proportionally with the amount of NAPL in the sediment and with the sediment particle size (that is, the same quantity of NAPL in sand will have a higher fluorescence response than in a silt or clay).

10.3.4 Similar to UV light screening, materials other than PAHs in the sediment may fluoresce (10.2.2). Fluorescence color will also vary depending upon the type of NAPL present. Thus, additional site-specific testing is required to correlate fluorescence intensity with the quantity of NAPL present and potential mobility.

10.3.5 LIF can be used to identify locations and depths where relatively more NAPL may be present in the sediment. This information can be used to inform selection of locations/depths for NAPL mobility sampling. Alternatively, if coupled with a sediment sampling and laboratory testing program, this information can be used to develop a correlation between fluorescence response and the amount of NAPL in the sediment; this has been used to correlate fluorescence response and NAPL saturation in a number of case studies (**6**, **7**, **8**).

10.4 *Hydrophobic Dye:*

10.4.1 Hydrophobic dye techniques involve adding a small quantity of hydrophobic dye to a jar containing a mixture of sediment and water. The jar is agitated; if NAPL is present, the dye dissolves into the NAPL, changing its color. Common dyes include Sudan IV and Oil Red O, both of which result in a bright red color when in contact with NAPL. Note that this test is qualitative and observing no color change does not mean NAPL is not present. Silts and clays commonly present in sediment samples may obscure results. Due to the potential for false negatives, some tests use two dyes, one hydrophobic dye for NAPL and one water-soluble dye. The water-soluble dye is intended to improve contrast and make the hydrophobic dye more visible. This field screening method may be useful for determining NAPL presence/absence, particularly for clear/colorless NAPLs, but it does not assess the relative potential for NAPL to be mobile at the pore scale.

10.5 *NAPL FLUTe*[4] *Fabric*:

10.5.1 NAPL Flexible Liner Underground Technologies (FLUTe[4]) is a color-reactive hydrophobic fabric. If present, NAPL wicks through the material and dissolves the dye stripes on the front side of the material. The NAPL carries the dye to the back side of the material. The stain on the back side of the material confirms that NAPL is in contact with the opposing side of the NAPL FLUTe[4] fabric. Although NAPL FLUTe[4] is effective at detecting various types of NAPL, practitioners should consult with the product manufacturer to verify compatibility with suspected NAPL types prior to using this method.

10.5.2 Traditionally, NAPL FLUTe[4] has been deployed within an open upland borehole with the goal of identifying the elevation and thickness of fractures or primary pore spaces containing NAPL. NAPL FLUTe[4] can also be gently pressed against sediment cores to qualitatively evaluate the presence of the NAPL on the exposed surface of the sediment core. A positive NAPL FLUTe[4] observation can be used to identify NAPL on the exposed sediment surface and select sediment samples for laboratory testing of pore-scale NAPL mobility. Standard procedures for the application of NAPL FLUTe[4] to assist in field logging of NAPL presence have not been developed. NAPL FLUTe[4] cannot be used to detect NAPL present within the sediment that is not exposed on the sediment surface. NAPL FLUTe[4] does not assess the relative potential for NAPL to be mobile at the pore scale. NAPL FLUTe[4] should be combined with other lines of evidence to inform selection of a sediment core interval for NAPL mobility testing.

10.6 *Photoionization Detector:*

10.6.1 Field screening with a PID is a common practice to assess the relative concentration of volatile organic compound (VOC) vapors emitting from the sediment core or sediment grab sample. An elevated PID response indicates comparatively greater VOC concentrations in the sediment core but should not be interpreted as a positive indicator of NAPL presence. This field screening method may be useful for determining NAPL presence/absence, but it does not assess the relative potential for NAPL to be mobile at the pore scale. Use of PID responses should be limited to identification of the intervals where additional assessment of potential NAPL or other organic chemicals could be completed using other methods outlined in this Guide. PID screening is described in greater detail in Practice D7203.

11. Keywords

11.1 contaminated sediments; field screening; laboratory NAPL mobility testing; mobility; NAPL; sediment; sediment corrective action; shake test

[4] Trademarked by Flexible Liner Underground Technologies.

APPENDIXES

(Nonmandatory Information)

X1. RECOMMENDED PROCEDURE FOR VISUALLY CHARACTERIZING SEDIMENT FOR SHEEN OR NAPL OBSERVATIONS

X1.1 Overview

X1.1.1 This recommended procedure describes how to visually characterize a sediment sample for the presence or absence of sheen or NAPL and (if present) qualitatively estimate the amount of sheen or NAPL present. The basic procedures for visually characterizing sediment for sheen or NAPL observations can be applied to sediment core samples or grab samples of surface sediment.

X1.2 Personnel Qualifications

X1.2.1 Personnel performing this procedure should read and be familiar with the requirements of the procedure, as well as work under the direct supervision of qualified professionals who are experienced in performing this type of work.

X1.2.2 To ensure objectivity and consistency in visual observations, it is recommended that initial (or refresher) training of this procedure be performed prior to the start of each new phase of field investigation. To the extent possible, training should include an opportunity for personnel to practice performing the procedure as a group under the supervision of experienced personnel.

X1.2.3 To the extent possible, the same personnel, or group of personnel, should perform sheen and NAPL visual characterizations and shake tests over the course of a project.

X1.3 Procedure

X1.3.1 For surface grabs, photograph the sediment surface in the grab sampler. Depending on the depth of the surface sediment grab, place sample material from the desired grab depth interval (for example, all the materials collected from each 0.15-m [15-cm] interval) into a clean stainless-steel bowl and photograph the material.

X1.3.2 For sediment cores, prior to processing, the core caps will be removed. For each section of the core, the core liner will be cut longitudinally using a circular saw, power shears, or a cutting tool (two longitudinal cuts 180° apart); care will be taken not to penetrate the sediment while cutting the liner. The sediment core will be split from the bottom of the core to the top with decontaminated stainless-steel utensils or wire to expose the center of the two halves for observation and core photography, while minimizing disturbance to the sediment. If significant visible sheen or NAPL is found on the utensils during cutting, they should be cleaned or replaced to prevent cross-contaminating sediment in other intervals. Core halves can be stabilized using corrugated roofing closure strips (or similar braces) to prevent movement.

X1.3.3 Prior to visually characterizing the freshly exposed undisturbed sediment surface for the presence of sheen or NAPL, take color photographs of the total core/sample length, using a whiteboard to record information about the core section being photographed.

X1.3.3.1 To ensure that sediment features and sheen and NAPL distribution are visible in core photographs, take photographs of the core in 0.3-m to 1.0-m increments (typically, the shorter increments are used to provide more detail), with a scale or tape measure placed next to the core/sample and included in the photograph, to indicate core depth. It is recommended that photographs be taken straight on, with the core in the horizontal position, with the shallower depth on the left and the deeper depth on the right. Depending on the lighting, the angle of the photograph may be altered to enhance the color, contrast, and detail of visible sediment features, as well as to reduce reflection of light that may reduce visible details.

X1.3.3.2 To ensure that sediment features and sheen and NAPL distribution are visible in core/sample photographs, take photographs under bright natural light. Additional light sources may be needed when photographing cores indoors or under low lighting (for example, LED flashlight or work light).

X1.3.3.3 If needed, additional close-up photographs should be taken of zones of particular interest, such as sheen- or NAPL-bearing zones.

X1.3.3.4 Review core/sample photographs prior to moving on to the next step in the procedure to check that photographs are in focus and bright. Retake photographs if needed.

X1.3.3.5 Examples of core photographs are provided in Fig. X1.1 and Fig. X1.2.

X1.3.4 Record a description of the core/sample, including the following components needed to determine the depths at which shake testing will be performed:

X1.3.4.1 Sediment type, moisture content, density/consistency of soil, and color along the entire length of the core/sample (for guidance, see Practices D2487 and D2488).

X1.3.4.2 Odors (for example, hydrogen sulfide or petroleum hydrocarbons).

X1.3.4.3 PID readings.

X1.3.4.4 Visual observations of sheen or NAPL, as described in X1.4 and X1.5.

X1.3.4.5 The name of the personnel making the observations and recording the descriptions.

X1.4 Visual Observations of Sheen

X1.4.1 Where sheen is observed in the sediment core sample, describe the observation as outlined in this section.

X1.4.2 Record the start and end depths for each unique observation.

X1.4.2.1 If any aspect of the sheen observation changes along the length of the sample—for example, if the relative