

Methods for Determining Inputs to Environmental Petroleum Hydrocarbon Mobility and Recovery Models

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1.0 Introduction

This manual describes methods used to obtain input parameters for models that evaluate the mobility and potential recovery of petroleum liquids in unconsolidated granular porous media. These models are valuable tools for improving system design because, unfortunately, the presence of petroleum liquids in a well is not itself a reliable indicator of the feasibility of free-product recovery or the need to continue recovery operations.

The feasibility and appropriate endpoint for free-product recovery can be addressed by evaluating the mobility of product in the sediment in which it is encountered. If the mobility of product is high, there is a potential for future adverse product migration. In addition, it is likely that properly designed recovery systems can effectively deplete a significant fraction of the mobile product. Conversely, if the mobility of the product is low, the risk associated with future migration is low, and efforts to deplete mobile product will be ineffective.

The intent of this document is to provide a reference for parties needing methods for determining inputs to product mobility, and volume models. The need for such data (in particular at large sites such as refineries) has increased dramatically in recent years. In large part this has been driven by broader use of multiphase flow models (e.g., Parker *et al.*, 1994) and spreadsheet tools for analysis of product mobility (e.g., Charbeneau *et al.*, 1999). Secondarily, it is recognized that for small sites the resources needed to conduct site specific mobility investigations may not be available. As such, it would be useful to generate a data base of study results (obtained using consistent methods) that can be used at these sites.

Standardization of methods is the ideal; unfortunately, it may not be possible in all cases. A number of factors frustrate rigid standardization. First, significant differences exist between sites. A direct consequence is that the best methods for many analyses are different for different sites. Secondly, only a few specialized laboratories conduct a number of the analyses (e.g., capillary pressure vs. saturation and relative permeability vs. saturation). The equipment and methods used by these laboratories are highly specialized and frequently different. As such, it may not be possible to have all sites conduct measurements using a single method. Reflecting these challenges, this document provides options as opposed to rigid standards.

The first section introduces nomenclature and parameters of concern. Individuals familiar with multiphase flow in porous media can skip this section. The second section addresses determination of fluid properties. The third section describes methods for analysis of porous media properties at a laboratory (column) scale. The fourth section addresses measurement of porous media properties at a field scale. Lastly, suggestions for reporting results are presented.

Throughout this document practices are described that involve the handling of potentially hazardous materials. This document does not address health, safety, and regulatory compliance issues. It is the responsibility of the user of this document to establish appropriate health and safety practices and to comply with relevant regulations.

2.0 Nomenclature and Parameters of Interest

A general conceptual model for petroleum hydrocarbon in granular porous media is presented in Figure 1. In this example, a surface release of product has percolated through the unsaturated zone. Below the capillary fringe an interval has formed in which product occurs as a continuous **non-wetting phase** in the porous media. The term non-wetting reflects product occupying the largest pore space and not directly contacting the porous media. The porous media is surrounded by a continuous water phase referred to as the **wetting phase**. Immediately above the capillary fringe, product forms a continuous **intermediate wetting phase** between the water (wetting phase) and the air (non-wetting phase). The critical feature to recognize is that two or three separate fluid phases (including product) coexist in the pore space where product is present.

Product that forms a continuous phase within the porous media is referred to as **potentially mobile product**. The qualifier "potentially" is included because movement is contingent on the presence of a driving force. Product that is not present as a continuous phase is referred to as **residual** or **immobile product**. A significant feature of residual product is that it is typically immobile under the typical range of driving forces associated with natural or induced groundwater flow.

The volumetric flux of product $q_o(L^3/L^2-T)$ at any point in the continuous product interval can be described as:

$$q_o = -\frac{kk_{ro}}{\mu_o} \left(\frac{dP_o}{dx} + \rho_o g \frac{dz}{dx} \right) \tag{1}$$

where:

k (L ²)	Intrinsic Permeability	a property of the porous media that describes its capacity to transmit a single fluid that fully saturates the porous media (saturation equal to 1).	
k_{ro} (Dimensionless)	Relative Permeability to Product	a ratio of permeability to product at a pore saturation less than 1 to the permeability at a pore saturation of 1. This describes reduced permeability associated with product occupying only a fraction of the total pore space.	
μ_o (M/LT)	Dynamic Viscosity	a measure of a fluid's resistance to shear.	
P_o (F/L ²)	Pressure	force per unit area in the product phase in the product or oil.	
<i>x</i> (L)	Distance	in the direction of flow.	
$ ho_{_o}$ (M/L 3)	Density	mass per unit volume of oil.	
g (L/T 2)	Gravitational Constant	acceleration imposed by gravity.	
z (L)	Elevation	position above a given datum.	
(Note: L = length, M = mass, T= time, F= force)			

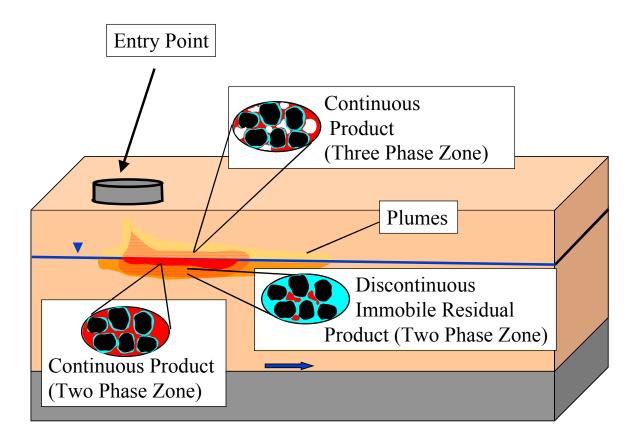


Figure 1 — General Conceptual Model

The **mobility** of the product is defined as:

$$M_o = \frac{kk_{ro}}{\mu_o} \tag{2}$$

Unfortunately, the fraction of pore space filled with product (S_o), and consequently the relative permeability to product (k_{ro}), changes with position. As such, mobility (M_o) is a function of position within the interval of continuous product. Variation in **product saturation** (S_o) reflects locally varying differences in pressure between the non-wetting and wetting phase pressures. This difference is defined as **capillary pressure** (F/L^2)

$$P_c = P_{nw} - P_w \tag{3}$$

Figure 2 illustrates pressure in the air, product, and water phases as a function of vertical position under the condition of no vertical flow. Recalling that pressure is linearly proportional to fluid density ($P = \rho gh$):

- Pressure in the air phase is essentially constant over the interval of concern due to the small density of air.
- With depth, pressure increases fastest in the water phase since water has the greatest density.
- As the product is less dense than water, pressure increases more slowly with depth in the product than in the water,

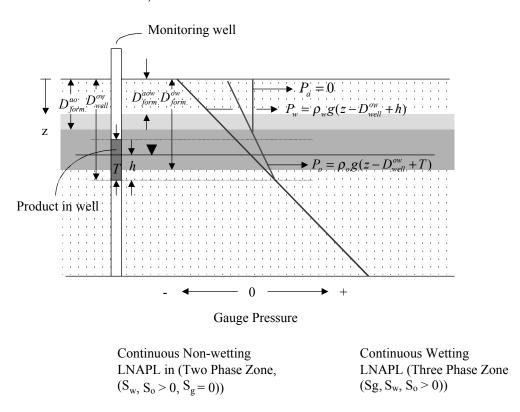


Figure 2 — Hydrostatic pressure in air, product, and aqueous phases (after Farr *et al.*, [1990], ao = air-oil, form. = formation, ow = oil-water, well = well, aow = air-oil-water, a = air, w = water, o = oil).

Building on the conditions defined in Figure 2, one can calculate both the volume and mobility of the continuous product present in the formation. Following Farr *et al.* (1990) and Lenhardt and Parker (1990), this process begins by using fluid levels in wells to characterize static pressures in the air, product, and aqueous phases (see Figure 2). Capillary pressures can be determined from the static pressures. Next, either the Brooks-Corey (1964) or van Genuchten (1980) models are used to estimate product saturation and relative permeability as a function of capillary pressure. These computations can be readily conducted using the spreadsheet model provided by Charbeneau *et al.* (1999).

As an introduction to the remaining parameters of interest, the following describes the Brooks-Corey and van Genuchten models. Both of these models are algebraic equations used to fit capillary pressure versus saturation data obtained from laboratory studies. For simplicity, the definition of physical parameters employed in describing both the models follows that used in Brooks and Corey (1964). Fitting parameters follow the definition presented in Brooks and Corey (1964) and van Genuchten (1980).

A typical capillary pressure-saturation relationship is presented in Figure 3. Procedures used to estimate fitting parameters from the capillary pressure data are described in Corey and Brooks (1999) and van Genuchten (1980). Alternatively, model parameters can be obtained by fitting the models to relative permeability versus saturation data obtained from laboratory studies. A typical relative permeability-saturation relationship is presented in Figure 4.

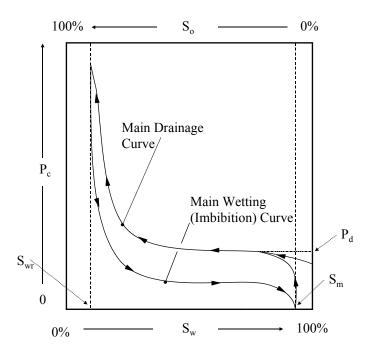


Figure 3 — Capillary pressure - saturation relationship for two-fluid system in porous media

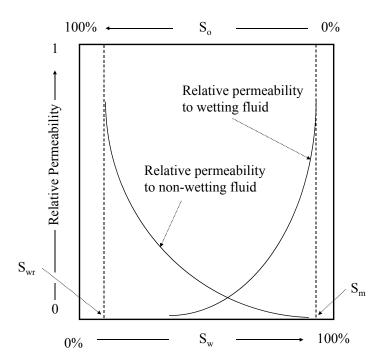


Figure 4 — Typical relationship between relative permeability and fluid saturation for a two-phase system under drainage.

The Brooks-Corey model is described in Equations (4) through (6).

$$S_e = \frac{S_w - S_{wr}}{1 - S_{wr}} = \left(\frac{P_d}{P_c}\right)^{\lambda} \quad \text{for } P_c \ge P_d \tag{4}$$

$$k_{ro} = \left[1 - \frac{S_{w} - S_{wr}}{1 - S_{wr}}\right]^{2} \left[1 - \left(\frac{S_{w} - S_{wr}}{1 - S_{wr}}\right)^{\frac{\lambda + 2}{\lambda}}\right]$$
 (5)

$$k_{rw} = \left(\frac{S_w - S_{wr}}{1 - S_{wr}}\right)^{\frac{2+3\lambda}{\lambda}} \tag{6}$$

where:

S_e (Dimensionless)	Effective Wetting Phase Saturation	wetting phase saturation as defined in (4)
$S_{\scriptscriptstyle w}$ (Dimensionless)	Wetting Phase Saturation	fraction of the pore space filled with wetting phase.
$S_{_{\it wr}}$ (Dimensionless)	Irreducible Wetting Phase Saturation	model fitting parameter. Roughly equal to the minimum wetting phase saturation at large capillary pressure (maximum product saturation).
P_d (F/L ²)	Displacement Pressure	model fitting parameter. Roughly equal to the minimum capillary pressure at which the non-wetting phase is present as continuous phase in the porous media. Brooks and Corey (1964) also refer to this parameter as bubbling pressure.
λ (Dimensionless)	Lambda	model fitting parameter. Generally a function of grain size distribution.
k_{ro} (Dimensionless)	Relative Permeability to the non-wetting phase	a ratio of permeability to the non-wetting phase at pore saturation less than 1 to the permeability at a pore saturation of 1.
k_{rw} (Dimensionless)	Relative Permeability to the wetting phase	a ratio of permeability to wetting phase at a pore saturation less than 1 to the permeability at a pore saturation of 1.

Following Parker *et al.*, (1987) and Charbeneau *et al.*, (1999), the van Genuchten model is described in Equations (7) through (9). Note the van Genuchten model parameter (N) is not included in Equations (7) through (9). This reflects the substitution N = 1/(1-M) proposed by van Genuchten (1980) when using Mualem's model for relative permeability (Mualem, 1976).

$$S_e \int \frac{S_w - S_{wr}}{1 - S_{wr}} = \left(\frac{1}{1 + (\alpha P_c)^{\frac{1}{1 - M}}}\right)^M \quad \text{for} \quad P_c \ge 0$$
 (7)

$$k_{ro} = \left(\frac{S_o}{1 - S_{wr}}\right)^{\frac{1}{2}} \left\{ \left[1 - \left(\frac{S_w - S_{wr}}{1 - S_{wr}}\right)^{\frac{1}{M}}\right]^M - \left[1 - \left(\frac{S_w + S_o - S_{wr}}{1 - S_{wr}}\right)^{\frac{1}{M}}\right]^M \right\}^2$$
(8)

$$k_{rw} = \left(\frac{S_w - S_{wr}}{1 - S_{wr}}\right)^{\frac{1}{2}} \left\{ 1 - \left[1 - \left(\frac{S_w - S_{wr}}{1 - S_{wr}}\right)^{1/M}\right]^M\right\}^2$$
(9)

where:

 $\begin{array}{ll} \textit{M} & \text{(Dimensionless)} & \textbf{M} & \text{van Genuchten fitting parameter.} \\ \alpha \text{ (L2/F)} & \textbf{Alpha} & \text{van Genuchten fitting parameter.} \end{array}$

Lastly, the parameters of concern are not always measured using site water and product. In some cases air or mercury are used in laboratory studies. To correct back to the fluids of concern, it is necessary to obtain estimates of air-product interfacial tension (σ_{ao}), air-water interfacial tension (σ_{aw}), and product-water surface tension (σ_{ow}). The units of interfacial and surface tension are force per unit area (F/L).

3.0 Fluid Properties

3.1 Sample Collection

Analysis of product properties begins with obtaining representative product samples. Typically this is accomplished by bailing free product from monitoring wells or collecting samples from active free-product recovery systems. Samples should be collected from a sufficient number of points to characterize the product present. Spatial variations in product properties commonly occur due to releases of different products and/or differential *in situ* weathering. A minimum volume of 250 mL of product (free of water) should be collected. Samples should be stored in brown glass containers with minimal head space and kept cool. No preservatives are required.

In all cases the *in situ* temperature of the product should be determined. This reflects the dependence of fluid properties on temperature. In the case of bailed product samples, temperature should be measured immediately after collection. In the case of active recovery systems, the temperature should be measured in fluids that are "fresh" from the well (as opposed to stagnant fluids from piping or tanks). A third option is to lower a thermometer in a secure housing down a well to the water table. In all cases the thermometer should have a minimum accuracy of 1-degree Celsius.

After sources of representative product samples have been identified, additional product and water samples should be collected for laboratory core studies. The volume of water and product needed to conduct core studies should be determined by asking the laboratory. Again, samples should be stored in brown glass containers with minimal head space and kept cool.

3.2 Density (ρ_{\circ})

Table 1 presents methods for determining the density of petroleum liquids. The order reflects common to uncommon use. Results can be reported in terms of density (M/L³) or as °API gravity. Calculations typically require results to be expressed in terms of density. The conversion from API gravity to density is:

$$\rho_{\circ} = \frac{141.5}{{}^{\circ}\text{API} + 131.5} \rho_{\text{w}} \tag{10}$$

In general, the density of site water should also be measured. This reflects the fact that both dissolved organic and inorganic compounds can effect water density. The density of water is significant in that capillary pressure is a function of the difference in density between water and product. Ideally, measurements should be made using liquids at their *in situ* temperature. Sensitivity to temperature is illustrated by considering fuel oil which varies in density by 2 percent over the range of 0 to 25 degrees Celsius (Charbeneau *et al.*, 1999).

Table 1 — Product density methods (ρ_\circ)

Method	Description	Comments
ASTM D1298-99 or ASTM D-287	Standard Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method	Suitable for field determination of density. Hydrometers can be purchased for a small cost. Accuracy (typically 0.0035 to 0.007 gm/cm3) is dependent on the range of the hydrometer used. Method requires ~ 200 mL sample.
ASTM D1217- 93(1998)	Standard Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer	Best for distillates that boil between 90 and 110°C. Precision is ~0.00001 gm/cm ³ .
ASTM D1481- 93(1997)	Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer	Oils more viscous than 15 cSt at 20°C (mm /s), and of viscous oils and melted waxes at elevated temperatures.
ASTM D1480- 93(1997)	Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer	Less accurate than ASTM D1217-93. Precision is ~0.00005 gm/cm ³ .
ASTM D4052-96	Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter	Suitable for materials that are liquids at 15-35°C. Liquids should be translucent. Precision is ~0.0001 gm/cm ³ .
ASTM D5002-99	Standard Test Method for Density and Relative Density of Crude Oils by Digital Density Analyzer	Appropriate for low vapor pressure crude oils.
ASTM D3505-96	Standard Test Method for Density or Relative Density of Pure Liquid Chemicals	Requires accurate knowledge of temperature expansion functions.

Further details regarding ASTM methods may found in Appendix A - Short Abstracts of Relevant ASTM Methods.

3.3 Viscosity (μ_{\circ})

Table 2 presents methods used to determine the viscosity of petroleum liquids. The order reflects common to uncommon use. Measurements should be made at *in situ* temperatures. Illustrating sensitivity to temperature, the viscosity of kerosene varies by 54% over the range of 0 to 25 degrees Celsius (Charbeneau *et al.*, 1999). Whenever possible, measurements should be made using liquids collected from the site of concern. Prolonged exposure of petroleum liquids to soil and water can have contact significant effects on the viscosity of petroleum liquids.

Table 2 — Viscosity methods (μ_{\circ})

Method	Description	Comments
ASTM D445-97	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity).	Common wet chemistry technique.
Brookfield, Pfann, or Rotational Viscometer	Principle of rotational viscosity measurement employs a spindle immersed in the fluid sample to sense torque resistance when running at constant speed.	Common instrument technique.
ASTM D4486-91 (1996)e1	Standard Test Method for Kinematic Viscosity of Volatile and Reactive Liquids.	Best for volatile and reactive liquids.

3.4 Surface Tension and Interfacial Tension ($\sigma_{\rm ao}$, $\sigma_{\rm ow}$, and $\sigma_{\rm aw}$)

Table 3 presents methods used to measure surface and interfacial tension. Key parameters of concern are air-oil interfacial tension, air-water surface tension, and oil-water surface tension. The order reflects common to uncommon use. Measurements should be made at temperatures close to *in situ* conditions. As with viscosity and density, measurements should be made using liquids from the site of concern. Prolonged interactions between water, petroleum liquids and soil can have significant effects on fluid properties.

Table 3 — Surface tension and interfacial tension methods ($\sigma_{\rm ao}$, $\sigma_{\rm ow}$, and $\sigma_{\rm aw}$)

Method	Description	Comments
ASTM D971-99a (e.g., DuNouy Tensiometer)	Standard Test Method for Interfacial Tension of Oil Against Water by the Ring Method	(Gas-Liquid & Liquid-Liquid) ±0.1 mN/m.
Spinning Drop Tensiometer	Relies on centripetal forces which replace gravity in other types of tensiometers	(Gas-Liquid & Liquid-Liquid) 0.005 mN/m ~ 500 mN/m, measuring range good for crude oil, surfactant solutions.
ASTM D3825- 90(1995)	Standard Test Method for Dynamic Surface Tension by the Fast-Bubble Technique	(Gas-Liquid) Suitable for liquids with viscosity less than 4 centipoise, not suitable for opaque DNAPL.
ASTM D1331- 89(1995)	Standard Test Methods for Surface and Interfacial Tension of Solutions of Surface-Active Agents	(Liquid-Liquid) Suitable to surface-active agents. Also applicable to nonaqueous solutions and mixed solvent solutions.
ASTM D2285-99 (Pendant Drop)	Standard Test Method for Interfacial Tension of Electrical Insulating Oils of Petroleum Origin Against Water by the Drop-Weight Method	Applicable to viscous solutions.

4.0 Porous Media Properties - Laboratory-Scale

The following outlines procedures for determining physical properties of porous media that relate to product mobility. In general, the methods described reflect decades of research in the fields of petroleum, soil science, and groundwater hydrology.

4.1 Sample Collection

Sediments - Analysis of soil properties at a laboratory scale begins with obtaining representative soil samples. Ideally, soil samples should be undisturbed. This reflects the fact that parameters of interest are dependent on the geometry of pore bodies and throats. Preservation of these features or reproducing these features represents a significant challenge.

At one extreme, soil samples can be recovered using hollow stem auger and split spoon sampling equipment [see ASTM D1586-99 or ASTM D1452-80(1995)e1]. After extraction of product, samples can be dried and sieved to remove larger materials (e.g., gravel) that would block flow in a standard core holder (diameters of a few centimeters). Lastly, samples can be placed in coreholders with confining pressure similar to the *in situ* overburden pressure. The assumption associated with this approach is that the procedure will result in pore bodies and throats representative of the *in situ* conditions. In general, this approach should be avoided when possible due to potential biases associated with disturbing the architecture of the sediments.

Alternatively, soil samples can be collected in liners. This can be accomplished using hollow stem auger drilling systems equipped with continuous sampling systems or using direct push drilling systems (see ASTM D6282-98) or thin-walled sampling tubes (see ASTM D1587-94). The liners serve as core preservation systems. Immediately after recovery the core should be frozen on site. This has been accomplished using liquid nitrogen or dry ice. Assuming the pore space is fully saturated with water (worst case), this will effect an approximate 5 percent expansion of the pore space. This may fracture the plastic sleeve. Even if this occurs, it is hoped that the general geometry of the particles will be preserved. Note: further complication is added by the fact that petroleum liquids shrink at reduced temperatures. The primary assumption with this approach is that compaction during sample collection and freeze-thaw do not impose significant biases.

Beyond the above, options are limited. Within the crude oil production industry, a number of special coring systems have been developed, including pressure retaining cores, sponge-lined coring systems, full-closure coring systems, rubber sleeve core barrels, and side wall coring systems [see API (1998)]. Unfortunately, these systems are generally not available for collection of shallow unconsolidated soils. Notable exceptions are described by:

- Durnford et al., (1991) A prototype sampler is described that cools soil at the bottom end
 of a sampler to near freezing. Cooling is achieved by allowing compressed carbon dioxide
 to expand to atmospheric pressure in situ at the bottom end of the sampler. The principle
 benefit is retention of liquids in the soil core. Samples are frozen upon retrieval to the
 surface.
- McElwee et al., (1991) A prototype sampler that closes in situ is described. Nitrogen gas
 is used to inflate a bladder located in the drive shoe of the sampler. The primary advantage
 noted is improved recovery of sands and gravels.

• Murdoch et al., (2000). – A sampling tool is described that collects horizontal sidewall soil core up to 15 centimeters in length and 4 cm in diameter. A hydraulic pushing mechanism advances and retracts a sidewall sample tube. Promising aspects include direct collection of horizontal soil cores (potentially less disturbed) and collection of short samples which are less prone to drainage of interstitial liquids. A difficulty is that direct observation of the sample may not be possible prior to testing due to the sample tube.

The user should determine the commercial availability of the above three techniques.

Residual Liquids – Often it is desirable to obtain measurements of product and water saturation from cores. In such instances it is necessary to address the issue of whether the residual fluids present in samples after collection are representative of liquid saturations present *in situ*. Using conventional split spoon or direct push techniques, significant biases can be introduced, including:

- Driving sampling equipment through sediments may compress the samples and displace fluids.
- Fluids that accumulate in sampling devices above the sample (e.g., water in portions of the sampler above the sample) will drain through the sample as it is brought to the surface. As this occurs, water and product saturations within the sample may be altered significantly.
- As the sample is brought to the surface, air can invade the pore space. If this occurs, product and/or water will drain out.

The severity of the above problems will increase with the hydraulic conductivity of the sediments and the length of the sample collected.

Methods outlined by Durnford *et al.*, (1991) or Murdoch *et al.*, (2000) hold promise to overcome the noted biases. Unfortunately, their availability and effectiveness is not well documented. Alternatively, using conventional equipment, samples should not be collected from below the water table as this limits fluid flushing through the sample. In addition, core lengths should be less than the height of capillary rise in the soil of interest. This will limit drainage of liquids from the samples as they are collected. McWhorter (1996) describes an empirical approach to estimating air-water displacement pressure. Using this method and the assumption that only water liquid is present in the pore space, estimates of capillary rise (maximum sample height) as a function of sediment hydraulic conductivity can be obtained (See Figure 5).

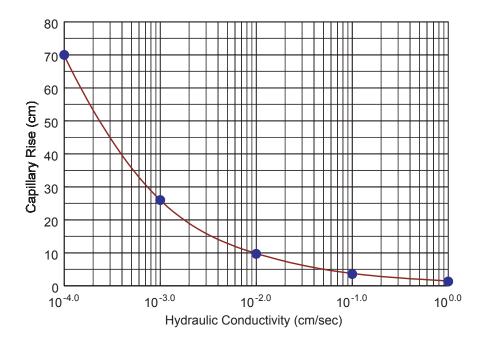


Figure 5 — Capillary rise as function of hydraulic conductivity (After McWhorter, 1996)

4.2 Core Preservation (Field)

Objectives of core preservation are to:

- Accurately document the sample location and depth
- Preserve the mechanical integrity of the sample
- Retain liquids present in the cores

Disturbed samples should be placed in labeled glass jars and kept cool. Plastic containers should not be used in instances where the plastic may be degraded by the hydrocarbons in soil. Soil samples in sleeves should be:

- 1. Capped and taped at both ends to limit expansion within the sleeve
- 2. Sealed using aluminum foil or a dip coating to limit losses of volatile compounds and liquids
- 3. Labeled as to location, interval, date, and orientation (top-bottom)
- 4. Placed in a horizontal position and frozen immediately to stabilize sediments and prevent loss of liquids. Again, freezing of the core could result in fracturing of a plastic liner sleeve. Even if this does occur, pore geometry will be kept as close to a native state as possible.

Approaches used to freeze samples include dry ice or liquid nitrogen at the drilling site. Once the samples are frozen they should be shipped (in a frozen state) to the laboratory conducting the core analyses. Ideally this should be done using an overnight courier so that samples remain frozen during shipping. At the laboratory, samples should continue to be kept frozen until used in specified tests. Additional details regarding preservation of unconsolidated samples can be found in *Recommended Practices for Core Analysis*, API (1998).

4.3 Core Screening and Preparation (Laboratory)

Steps involved in laboratory screening and preparation of soil samples include:

- 1. Logging the cores
- 2. Selecting intervals of interest
- 3. Collecting subcores (horizontal or vertical orientation)
- 4. Mounting subcores in holders for hydraulic testing
- 5. Extracting residual liquid (water and product) from the core

Steps one through three are typically a joint activity conducted by the site geologist and laboratory staff. In steps two and three, representative subcores (vertical or horizontal orientation) are identified and cut from the frozen field core in a laboratory. The diameter of subcores is typically a few centimeters, conforming to the size of standard core holders. Subcores should be cut and sleeved frozen for laboratory manipulation. Step four typically involves placing the core in a sleeve and applying a representative overburden pressure. Overburden pressure is useful in reproducing *in situ* conditions and in controlling short-circuit flow of liquids at the boundary between the sample and core holder. Care should be taken not to apply excess overburden pressure as this will collapse the pore space and alter the porous media properties of interest. Step five is required for determining permeability, capillary pressure as a function of saturations, and relative permeability as a function of saturation. Steps four and five are not required for samples that will be used for determining residual liquid saturations.

Some hydraulic tests result in destruction of the sample (e.g., residual liquids determination and capillary pressure tests using mercury). Therefore, it is often necessary to obtain multiple core samples from intervals of interest. Comprehensive discussion of core screening and preparation (including attention to frozen unconsolidated samples) is presented in Recommended Practices for Core Analysis, API (1998).

4.4 Porosity (ϕ)

Standard methods for measuring porosity are presented in Table 4. Methods are listed in order from most common to least common. Since porosity is a function of pore space architecture, it is desirable to use intact core samples with representative overburden pressures. Porosity is typically reported as a fraction of the bulk soil volume. Alternatively, it can be presented as a percentage of the bulk soil volume.

Table 4 — Porosity methods (ϕ)

Method	Description	Comments	
API (1998) Sec. 5.3.2.2.3	Liquid Saturation Method – Direct pore volume measurement	Suitable for jacketed unconsolidated samples, accuracy to 0.005 (fraction of bulk soil volume).	
API (1998) Sec. 5.3.2.2.1	Boyle's Law Double Cell Method – Direct pore volume measurement	Suitable for jacketed unconsolidated samples.	
API (1998) Sec. 5.3.2.2.2	Summation of Fluids – Porosity is computed by measuring and summing product, air, and water present in fresh cores.	Involves displacement of core liquids with mercury, destroys core, requires permeability measurement to be made on a separate sample.	
ASTM D4404- 84(1998)e1	Standard Test Method for Determination of Pore Volume and Pore Volume Distribution of Soil and Rock by Mercury Intrusion Porosimetry	Not well suited to high permeability sediments; at high pressures may alter pore geometry.	
ASTM F1815-97	Standard Test Method for Saturated Hydraulic Conductivity, Water Retention, Porosity, Particle Density, and Bulk Density of Putting Green and Sports Turf Root Zones	More suited to agronomic soils.	

4.5 Permeability (k)

Techniques for measuring permeability are outlined in Table 5. The order follows the general preference of the author. Since permeability is dependent on the geometry of pore bodies and throats, use of representative samples (under representative overburden pressures) is critical to obtaining meaningful estimates. In all cases, core samples must be free of residual liquids.

Common units for permeability are micrometers squared (μm^2) and Darcys. One Darcy is equal to 0.987 μm^2 .

Permeability can be measured using gas or liquid under steady or unsteady flow conditions. In addition, flow can be driven along the axis of the core (axial flow) or radially about the axis of the

core (radial flow). As such, there are twelve possible approaches that can be used. Each of these approaches has advantages and disadvantages. The techniques listed in Table 5 reflect:

- Obtaining an effective seal at the edge of unconsolidated samples is more difficult when the
 test fluid is a gas. Furthermore, gas measurements are prone to significant high velocity
 inertial resistance in high permeability sediments. As such, preference is given to liquid
 measurement techniques (see order).
- In general, constant head techniques are simpler and more reliable. Typically, use of unsteady measurement techniques is driven by a need to measure extremely low permeability. Unsteady methods are not included in Table 5.
- The noted ASTM methods are focused on geotechnical and agronomic applications and, therefore, are less applicable and listed last.

Note: Whenever possible, site water should be used for liquid permeability determinations. Use of different water may cause non-representative reactions with the matrix materials, such as swelling or shrinking of clays.

Table 5 — Permeability methods (k)

	Table 5 — Permeability methods (%)			
Method	Description	Comments		
API (1998) Sec. 6.3.2.2 or 6.3.2.1 Liquid Axial Flow Steady State	Gravity flow of liquids through high permeability cylindrical cores; sensor may be manometers or electric pressure transducers	Simple, low cost, difficult to ensure zero gas in core, 0.0001-100 Darcy.		
API (1998) Sec. 6.3.1.4 Liquid Radial Flow Steady State	Radial flow of liquid in a large sample.	Larger sample size yields an averaged value, more difficult to prepare samples, 0.000002-0.25 Darcy.		
API (1998) Sec. 6.3.1.1. Gas Axial Flow Steady State	Axial flow of gas through cylindrical cores; sensor may be manometers or electric pressure transducers.	Historically a common approach, relatively simple, labor intensive, 0.0001-10 Darcy.		
API (1998) Sec. 6.3.1.4 Gas Radial Flow Steady State	Radial flow of gas in a large sample.	Large sample yield an averaged value; difficult to prepare samples, 0.00001-0.25 Darcy.		
ASTM D5856-95	Standard Test Method for Measurement of Hydraulic Conductivity of Porous Material Using a Rigid-Wall, Compaction-Mold Permeameter.	Appropriate for samples that are compacted in the laboratory.		
ASTM D5084- 90(1997)	Standard Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter.	Suitable for undisturbed samples.		
D2434-68(1994)e1	Standard Test Method for Permeability of Granular Soils (Constant Head).	Suitable for disturbed unconsolidated coarse sediment.		

4.6 Capillary Pressure vs. Saturation (P_c vs.S)

Typically, input parameters to either the Brooks-Corey model (Equations 4-6) and van Genuchten model (Equations 7-9) are obtained using capillary pressure vs. saturation data (See Figure 3). Methods for measuring capillary pressure as a function of saturation are listed in general order of preference in Table 6. All methods require that the samples be sleeved or placed in a confining apparatus.

Both the Brooks-Corey and van Genuchten models can be fit to drainage or wetting (imbibition) curves. Where the additional cost is not prohibitive, it is useful to obtain both drainage and wetting data. Since water table fluctuations cause drainage and wetting, both conditions are plausible under field conditions. Use of both data sets allows estimation of bounding conditions. Due to higher product saturation, for any given capillary pressure, wetting data will provide higher (more conservative) estimates of product mobility than drainage data. Note: the above statement is predicated on the assumption that water is the wetting phase.

Table 6 — Methods for capillary pressure as a function of saturation (P_c vs. S)

Method	Description	Comments
Porous Diaphragm Welge <i>et al.</i> , (1947)	Samples are placed on a diaphragm that only transmits the wetting fluid. Wetting phase saturation is determined as a function of pressure applied in the non-wetting phase.	Suitable for both drainage and wetting (imbibition) curves, superior in that actual wetting and non-wetting liquids can be used, slow procedure (days to months).
Centrifuge Method Slobod et al., (1951)	Samples are rotated at a range of constant speeds. Wetting phase saturation is determined as a function of applied force and resultant capillary pressure.	Suitable for both drainage and wetting (imbibition) curves, superior in that actual wetting and non-wetting liquids can be used, procedure is fast (hours), agreement with diaphragm method is good, applied force may cause adverse sample compaction.
Dynamic Method Brown (1951)	Simultaneous steady-state flow of two liquids is established. Capillary pressure is measured as the difference between the pressure in the two liquids. Saturation is controlled by flow rates.	Suitable for both drainage and wetting (imbibition) curves, superior in that actual wetting and non-wetting liquids can be used, procedure more complicated than the above.
Mercury Injection Purcell (1949)	The volume of mercury in a sample is determined as a function of the pressure used to displace air from the sample	Analyses can be conducted quickly, disadvantages include difference in wetting properties and loss of the cores.

4.7 Relative Permeability vs. Saturation (k, vs. S)

Table 7 lists methods for measuring relative permeability as a function of saturation. Two general categories of methods are steady-state and unsteady state. Significant debate has been given to which of these approaches is best (e.g., Honarpour *et al.,* (1986) and Rose (1987)). While it can be argued that steady-state methods are more rigorous, it is the author's opinion that the potential for better results is not significant when one considers the magnitude of uncertainty introduced by the heterogeneity of the subsurface environment. As such, Table 7 gives preference (order follows preference) to unsteady state methods due to their advantage of lower cost. It is noted that several of the reviewers of this document stated a preference for steady-state methods due to greater accuracy. In light of the lack of clear consensus, the best approach should be evaluated within the context of the objectives of individual projects.

The rationale for determining permeability as a function of saturation includes:

- The data can be used to identify whether the Brooks-Corey model (as presented in Equations 4-6) or the van Genuchten model (as presented in Equations 7-9) provides the best results.
- Insight can be gained into the wetting properties of the matrix.
- Relative permeability data can be used to directly obtain inputs to relative permeability models.

Table 7 — Methods for relative permeability as a function of saturation ($k_{\scriptscriptstyle \rm T} {\it vs.} S$)

Method	Description	Comments
Unsteady-State – Jones and Rosselle (May 1978)	Water is injected into core saturated with both oil and water. The oil saturation decrease and change in pressure is monitored.	Method allows for easier data interpretation by calculating the saturations and pressures at the core outlet.
Unsteady-State-JBN Method, Johnson <i>et al.</i> , (1959)	Water is injected into core saturated with both oil and water. The oil saturation decrease and change in pressure is monitored.	Method is quick and inexpensive, data interpretation is not straight forward.
Unsteady-State – Kerig and Watson (1987)	Water is injected into core saturated with both oil and water. The oil saturation decrease and change in pressure is monitored.	Uses cubic spline functional representation instead of exponential functionalities to develop data. Not as widely used as the Jones and Rozelle model.
Steady-State – Penn-State Method (Honarpour et al., (1986))	Two fluids are simultaneously pumped through a core at a range of flow rates, saturations are determined at steady-state conditions by weight, end effects are overcome by mounting the sample between two similar samples.	Historically a popular approach; a day or more may be required to achieve steady-state conditions for a given saturation; costly due to slow time to steady state.
Steady-State – Single Sample Dynamic Method (Honarpour <i>et al.,</i> (1986))	Two fluids are simultaneously pumped through a core at a range of flow rates, saturations are determined for steady-state conditions by weight, end effects are overcome using high flow rates.	Problems may occur at high flow rates due to nonlaminar flow; costly due to slow time to steady state.
Steady-State – Stationary Fluid Method (Corey et al. 1956)	Permeability to the non-wetting phase (e.g., air) is measured at a fixed wetting phase saturations.	Fast, typically done with air-water; requires correction for water-product systems.
Steady-State Hassler Method (Honarpour et al., (1986)) Original Patent is US 2,345,9.535 (April, 1944)	Two fluids are simultaneously pumped through a core at a range of flow rates, saturations are determined for steady-state conditions by weight, end effects are overcome using semi-permeable membranes that keep liquids separate at the inlet and outlet.	Potential for best results; generally difficult and time consuming to apply.

4.8 Water and Product Saturation (S_w and S_o)

In the case of representative field samples, product saturation values can be used to verify product saturations measured in laboratory studies and predictions developed using models. In the case of cores used in laboratory studies, saturation measurements provide direct determinations of irreducible water (S_{wr}) and irreducible product (S_{or}). Table 8 presents methods for determining product and water saturations in sediments. The order reflects the author's general preference.

Table 8 methods provide product concentration on a weight basis (e.g., mass of product per mass of dry weight soil). Following Feenstra *et al.*, (1991) and Brost and DeVaull (2000) weight basis soil concentrations, C_{soil} (M/M), can be transformed to product saturation using

$$S_o = \frac{C_{soil} (1 - \phi) \rho_{matrix} - C_w \phi S_w - C_w K_{oc} f_{oc} (1 - \phi) \rho_{matrix} - C_w H \phi S_a}{\phi \rho_o}$$
(11)

where:

 $ho_{\it matrix}$ (M/L³) - density of the minerals comprising the solid phase of the samples $C_{\it w}$ (M/L³) - dissolved concentration of product components in the aqueous phase $S_{\it w}$ (dimensionless) - water saturation

 K_{oc} (L³/M) - organic carbon - water partition coefficient for product

 $f_{\it oc}$ (dimensionless) - mass fraction of organic carbon in soil

H (dimensionless) - Henry's law coefficient for product

 S_a (dimensionless) - air saturation

∅ (dimensionless) – porosity – volume of the soil void space divided by the bulk soil volume

Unfortunately, products consist of a complex mixture of hydrocarbons. As such, estimates of K_{oc} and H are difficult to obtain. Ignoring mass stored in the aqueous, sorbed, and air phases, oil saturation can be estimated as

$$S_o = C_{soil} \left(\frac{(1 - \phi)\rho_{matrix}}{\phi \rho_o} \right)$$
 (12)

Assuming a product containing 10% benzene and 90% insoluable hydrocarbons, Equation 12 overestimates saturation by 7 and 0.7 percent, at product saturations of 1 and 10 percent, respectively. Note: use of Equation 12 requires an estimate of the density of the minerals comprising the solid phase and the product density. Where the solid matrix is dominated by quartz, it is reasonable to use the density of quartz for the matrix density (ρ_{matrix} = 2.65 gm/cm³). Otherwise, the bulk density of the soil ρ_b can be measured directly (ASTM D4564-93) and

converted to $\rho_{\it matrix}$ by dividing by $(1-\phi)$. Values for product density (ρ_{\circ}) can be obtained from site product samples or tables of typical values for known products (e.g., Mercer and Cohen [1990] and Charbeneau [1999]).

Table 8 — Water and Product Saturation Methods (S_w and S_o)

Method	Description	Comments
Dean-Stark, API (1998) Sec. 4.3	Distillation extraction method (reflux) using toluene; water is distilled from the sample; water content is measured by volume or weight of water condensate; hydrocarbon fraction is extracted; hydrocarbon content is determined by gravimetric difference. Hydrocarbon content can be determined by spectroscopy for some hydrocarbon types such as crude oil or creosote.	Yields accurate water content; relatively simple; applicable to a wide range of petroleum hydrocarbons; hydrocarbon volume may be inaccurate due to loss of solids or water of hydration.
EPA 418.1(USEPA 1986) or SW846-9071 (USEPA 1983) Total Petroleum Hydrocarbon	Extraction of hydrocarbons using Freon (CFC 113).	Inexpensive; accuracy should be sufficient for high concentrations associated with residual hydrocarbon (>1000 mg/kg), limited to C ₁₀ - C ₅₀ , less desirable for light hydrocarbons with significant fractions less than C ₁₀ (e.g., gasoline).
SW846-8015M (USEPA 1986) Gasoline Range Organics	Purge and Trap GC-FID procedure.	Only suitable if the product's hydrocarbons are in the gasoline range C ₆ -C ₁₂ .
SW846-8015M (USEPA, 1986) Diesel Range Organics	Solvent extraction followed by GC-FID procedure.	Only suitable if the product's hydrocarbons are in the diesel range C_{10} - C_{25} .
Retort Method Hansel, W.M. Jr (1974)	Retort method that distills product and water from soil. Distilled fluids are collected in a graduated receiving tube in a two stage distillation.	Lithologies with minerals rich in water are difficult to differentiate between water of hydration and crystalized water. Inexpensive but can be destructive.

4.9 Determination of Brooks-Corey Model Parameters (λ , P_d , and S_{wr})

Brooks-Corey model parameters λ , P_d , and S_{wr} are obtained by plotting either effective saturation (as defined in Equation 4) or relative permeability as a function of capillary pressure or capillary head. Capillary head is defined as

$$h_c = \frac{P_c}{\rho_w g} \tag{13}$$

This procedure is illustrated in Figures 6 and 7. The irreducible wetting phase saturation S_{wr} is obtained by iteratively testing different values of S_{wr} until the data forms a best fit to a straight line. According to Corey and Brooks (1999), "values of S_{wr} that are too small cause the curve to bend upward, and values that are too large cause the curve to bend downward". The remaining parameters are obtained graphically as shown in Figures 6 and 7. An advantage to the approach shown in Figure 7 is that the model is fit directly to relative permeability data, the parameter of interest. Reflecting on this, the value of λ developed in Figure 7 will provide a better estimate of relative permeability than the λ value generated in Figure 6. Other useful references regarding the Brooks-Corey model are Brooks and Corey (1964), Brooks and Corey (1966), Corey (1986), and Corey and Brooks (1999).

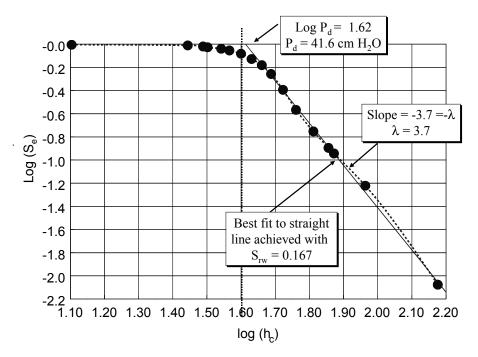


Figure 6 — Graphic format for analysis of Brooks-Corey parameters using effective saturation versus capillary head data (data from Brooks and Corey [1964], fine sand)

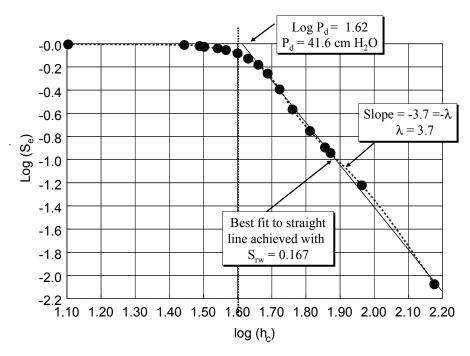


Figure 7 — Graphic format for analysis of Brooks-Corey parameters using relative permeability saturation versus capillary head data (data from Brooks and Corey [1964], fine sand)

4.10 Determination of van Genuchten Parameters (S_{rw} , M , and lpha)

Fitting parameters for the van Genuchten model (as introduced in equations (7) through (9)) are S_{rw} , M, and α . Values for these variables are determined by plotting capillary pressure vs. wetting water content (θ) as shown in Figure 8 (note $\theta = S\phi$). The irreducible wetting phase saturation S_{rw} is obtained by estimating water content at large capillary pressure (See Figure 8). The variable M is determined by identifying a point on the curve halfway between the maximum and minimum water content. Next, the dimensionless slope through this point S_p is determined and M is estimated as

$$M = \begin{vmatrix} 1 - \exp(-0.8S_p) & 0 < S_p \le 1 \\ 1 - \frac{0.5755}{S_p} + \frac{0.1}{S_p^2} + \frac{0.025}{S_p^3} & \text{for} \\ S_p > 1 \end{vmatrix}$$
 (14)

Next, α is determined using

$$\alpha = \frac{1}{h_p} (2^{\frac{1}{M}} - 1)^{1-M} \tag{15}$$

where h_n is the head value associated with the point P (See Figure 8).

Further detail regarding estimating parameters for the van Genuchten model are presented in van Genuchten (1980). Alternatively a program titled RETC (van Genuchten [1991]) can be used to obtain van Genuchten model parameters. The program permits one to fit analytical functions simultaneously to observed water retention and hydraulic conductivity data. Access to the program can be obtained through the Salinity Laboratory, Agricultural Research Service, U.S. Department of Agriculture (http://www.ussl.ars.usda.gov/Models/Modelsmenu.htm).

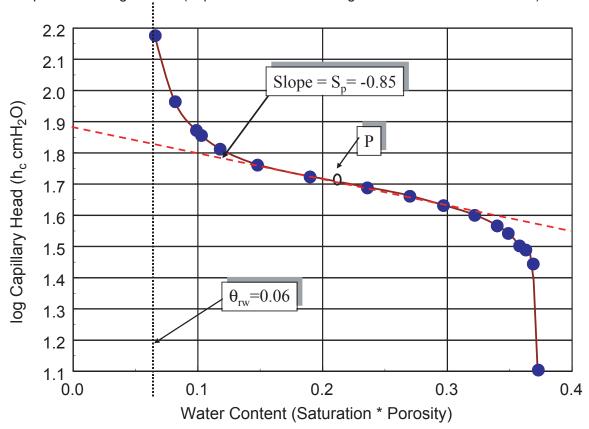


Figure 8 — Graphic format of analysis for van Genuchten parameters using log capillary pressure vs. saturation data (data from Brooks and Corey [1964], fine sand)

5.0 Porous Media Properties – Field-Scale

Discussions in the previous sections address determination of porous media properties at a scale of a few centimeters. Using centimeter-scale values to address field-scale problems, it is necessary to assume that centimeter-scale properties are representative of field-scale properties. Reflecting on the heterogeneous nature of geologic materials, extrapolation of centimeter-scale properties to field-scale may not always be appropriate. To overcome this limitation, porous media properties can be measured at a field scale. This is analogous to measuring hydraulic conductivity in a laboratory core test versus measuring hydraulic conductivity through an aquifer test. The core study provides a point value. The aquifer test provides a volume-averaged result for the portion of the aquifer stressed by the test. While field-scale determinations are appealing, their development for analysis of product mobility has been limited. The following describes use of baildown tests and hydrocarbon production data to address product mobility.

5.1 Baildown Tests

Baildown tests involve instantaneously removing product from a conventional monitoring well that is screened across the water table. Responses of the air-product and product-water interfaces are measured through time. The procedure is roughly analogous to a slug test.

Baildown tests have been used to obtain qualitative evidence of the potential for free-product recovery (e.g., Testa and Winegardner [1991]) and estimates of specific product volume (e.g., Lundy and Zimmerman [1996] and Lundy *et al.*, [1998]). It has also been proposed that baildown tests can be used to determine formation transmissivity to product (e.g., Lundy and Zimmerman [1996] and Huntley [2000]). Techniques for estimating product transmissivity using baildown tests are outlined in Table 9.

Table 9 — Baildown test methods for estimation of product transmissivity (field-scale T_{o})

Method	Description	Comments
Huntley (2000) – Modified Bouwer Rice Slug Test	Air-product and product-water interfaces are measured through time after rapid depletion of product. Data is analyzed using a modified Bouwer-Rice Slug Test procedure.	Appropriate for conditions where the corrected potentiometric surface remains near constant through the test; computationally simple.
Huntley (2000) – Based on Cooper- Jacob equation	Air-product and product-water interfaces are measured through time after rapid depletion of product. Three computational approaches are outlined; greater accuracy is achieved with more complex computational procedures.	Appropriate for conditions where the corrected potentiometric surface does not remain constant through the test; computationally more demanding.
Lundy and Zimmerman (1996) – Modified Bouwer and Rice Slug Test	Changes in LNAPL thickness, in a well from which a slug of LNAPL has been withdrawn, are analyzed with standard slug test methods to obtain vertically averaged LNAPL conductivity	Best for condition where the water table is not significantly depressed by removal of the LNAPL slug, computational methods are relatively simple.

5.2 Production Tests

Theim Solution

Where free-product recovery systems are employed, it is common to track production as a function of time. Typically this is accomplished using product flow meters or by tracking volume in product storage tanks. Following the derivation presented in Appendix B, production rates (Q_o) can be used to estimate vertically averaged relative permeability to product \overline{k}_{ro} (dimensionless), vertically averaged product conductivity to product \overline{K}_o (L/T), and transmissivity to oil T_o (L²/T)

$$\bar{k}_{ro} = \frac{Q_o \mu_o \ln\left(\frac{r_2}{r_1}\right)}{2\pi b_o k \rho_o g(h_2 - h_1)}$$
(16)

$$\overline{K}_{o} = \frac{k\overline{k}_{ro}\rho_{o}g}{\mu_{o}} = \frac{Q_{o}\ln\left(\frac{r_{2}}{r_{1}}\right)}{2\pi b_{o}(h_{2} - h_{1})}$$
(17)

$$T_o = \overline{K}_o b_o = \frac{Q_o \ln \left(\frac{r_2}{r_1}\right)}{2\pi (h_2 - h_1)}$$
 (18)

Variables h_1 and h_2 are the water heads measured at distances r_1 and r_2 from the recovery well. Variable b_o is the height of continuous product in the formation at the time of the measured product recovery rate (Q_o).

As an example, Figure 9 presents production data (circles) from a dual phase recovery well located in an alluvial aquifer at a former refinery. Initially recovery rates are low due to startup constraints. After 1989 the recovery well was operated at a near constant water production rate of 100 gal/min. Using data after 1989 and Equation 18, Figure 10 presents estimates of transmissivity to oil as a function of time.

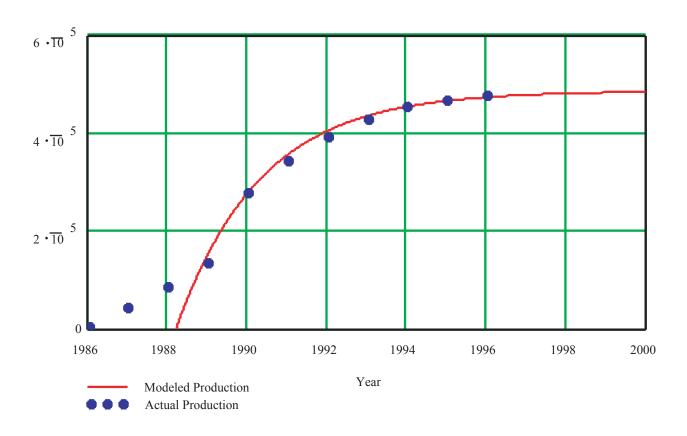


Figure 9 — Dual Phase Recovery Well Production Data

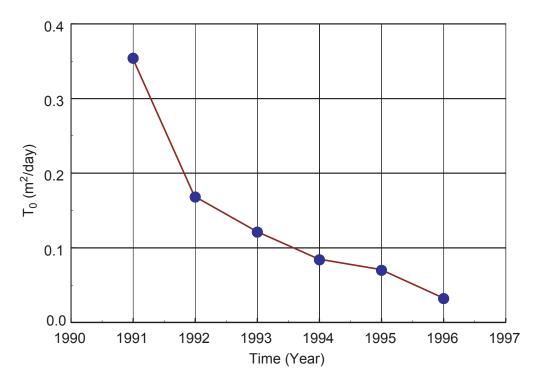


Figure 10 — Transmissivity to products as a function of time developed using well production data from Figure 9 and Equation (18)

Decline Curve Analysis

Building on petroleum industry methods, production data can be used to estimate future production [e.g., Frick and Taylor (1962)]. A decline curve analysis technique (building on the assumption of first order decay) is presented in Equations (19) through (21)

$$Q_o(t) = \alpha V_m e^{-\alpha t} \tag{19}$$

$$V_p(t) = V_m(1 - e^{-\alpha t})$$
 (20)

$$t_{1/2} = \frac{0.693}{\alpha} \tag{21}$$

where:

$Q_o(t)$ (L ³ /T)	Production Rate	volumetric recovery rate at time t.
α (1/T)	Alpha	a fitting parameter.
$V_m(L^3)$	Maximum Potential Volume	maximum or ultimate volume of product that can be recovered.
$V_p(t)$ (L ³)	Volume Produced	cumulative volume recovered at time t .
<i>t</i> _{1/2} (T)	Half Life	time required to deplete half of the remaining mobile product.

A derivation for Equations (19) through (21) is presented in Appendix C.

Figure 11 illustrates a graphic approach for estimating α and V_m . As shown, production rate is plotted as a function of cumulative production. Typically, late data will fit a straight line. The primary condition here is that the system is operated in a near consistent mode (near constant rate of total fluid production). The slope of the straight line is α . The x intercept of the line is V_m . The modeled results presented in Figure 9 (see solid line) illustrate that the technique provides a reasonable match to cumulative production data. Sale and Applegate (1997) describe use of decline curve analysis to estimate the maximum recoverable volume and operation of free product recovery systems to an endpoint of 95% of the maximum recoverable volume.

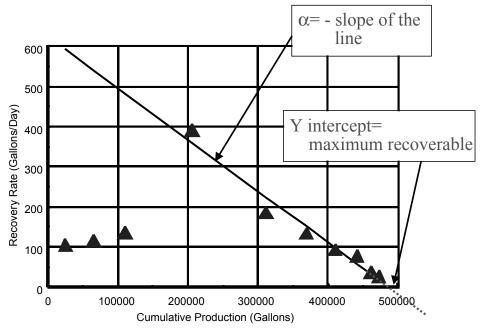


Figure 11 — Graphical format for decline curve analysis

6.0 Reporting

The requirements of individual projects vary. As such, it is inappropriate to proscribe a fixed format for reports. Nevertheless, in the interest of generating catalogues of results, it would be useful if reports included as many of the following elements as possible:

- 1. An introduction to the hydrologic setting and nature of the hydrocarbon released.
- 2. Documentation of procedures used in collecting liquids.
- 3. A description of methods used to drill and collect sediment samples.
- 4. Presentation of site core preservation, laboratory screening, and laboratory core preparation techniques.
- 5. For liquids measurements, a description of methods, the temperature at which measurements were made, and the *in situ* temperature of liquids.
- 6. A lithologic description of the core materials following ASTM D5434-97.
- 7. For porous media laboratory measurements, a description of methods and notation as to the temperature at which the measurements were made.
- 8. Tabulation of Brooks-Corey and/or van Genuchten model parameters.
- 9. For field measurements, a description of methods, data analyses procedures, and tabulation of results.
- 10. Comments as to the efficacy of the methods employed.
- 11. Conclusions reached through the analyses.

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Liquid Density

D3505-96 Standard Test Method for Density or Relative Density of Pure Liquid Chemicals

1. Scope

- 1.1 This test method describes a simplified procedure for the measurement of density or relative density of pure liquid chemicals for which accurate temperature expansion functions are known. It is restricted to liquids having vapor pressures not exceeding 600 mm Hg (0.8 atm) at the equilibration temperature, and having viscosities not exceeding 15 cSt at 20°C (60°F).
- 1.2 Means are provided for reporting results in the following units: Density g/cm at 20°C Density g/ml at 20°C Relative density 20°C/4°C Relative density 60°F/60°F (15.56°C/15.56°C) Commercial density, lb (in air)/U.S. gal at 60°F Commercial density, lb (in air)/U.K. gal at 60°F.

Note 1-This test method is based on the old definition of 1 L = 1.000028 cm). In 1964 the General Conference on Weights and Measures withdrew this definition of the litre and declared that the word "litre" was a special name for the cubic decimetre, thus making 1 mL = 1 cm exactly. Note 2-An alternative method for determining relative density of pure liquid chemicals is Test Method D4052.

- 1.3 The following applies to all specified limits in this test method: for purposes of determining conformance with this test method, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E29.
- 1.4 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 and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in 7.1.
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D1298-99 Standard Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

1. Scope

1.1 This practice covers the laboratory determination, using a glass hydrometer, of the density, relative density (specific gravity), or API gravity of crude petroleum, petroleum products, or mixtures of petroleum and nonpetroleum products normally handled as liquids, and having a Reid vapor pressure (Test Method D323, or IP 69) of (179 kPa) 26 lb or less. Values are measured on a hydrometer at convenient temperatures, readings of density being reduced to 15°C, and readings of relative density (specific gravity) and API gravity to 60°F, by means of international standard tables. By means of these same tables, values determined in any one of the three systems of measurement are convertible to equivalent values in either of the other two so that measurements may be made in the units of local convenience.

- 1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see 9.1.
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D1481-93(1997) Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer

- 1. Scope
- 1.1 This test method covers the determination of the density of oils more viscous than 15 cSt at 20°C (mm/s), and of viscous oils and melted waxes at elevated temperatures, but not at temperatures at which the sample would have a vapor pressure of 100 mm Hg (13 kPa) or above.
- Note 1-To determine the densities of less viscous liquids at 20 or 25°C use Test Method D941, or Test Method D1217.
- 1.2 This test method provides a calculation procedure for converting density to relative density (specific gravity).
- 1.3 The values stated in acceptable SI units are to be regarded as the standard.
- 1.4 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
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D1217-93(1998) Standard Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

1. Scope

- 1.1 This test method covers the measurement of the density of pure hydrocarbons or petroleum distillates boiling between 90 and 110°C that can be handled in a normal fashion as a liquid at the specified test temperatures of 20 and 25°C.
- 1.2 This test method provides a calculation procedure for conversion of density to relative density (specific gravity).
- 1.3 The values stated in SI units are to be regarded as the standard.
- 1.4 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Notes 1, 2, and 3.
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D1480-93(1997) Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer

1. Scope

1.1 This test method describes two procedures for the measurement of the density of materials which are fluid at the desired test temperature. Its application is restricted to liquids of vapor pressures below 600 mm Hg (80 kPa) and viscosities below 40,000 cSt (mm/s) at the test temperature. The method is designed for use at any temperature between 20 and 100°C. It can be used at higher temperatures; however, in this case the precision section does not apply.

Note 1-For the determination of density of materials which are fluid at normal temperatures, see Test Method D941 or where greater precision is desired see Test Method D1217.

- 1.2 This test method provides a calculation procedure for converting density to specific gravity.
- 1.3 The values stated in acceptable SI units are to be regarded as the standard.
- 1.4 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 and health practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements see Notes 1, 2, and 3.
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D4052-96 Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter

1. Scope

- 1.1 This test method covers the determination of the density or relative density of petroleum distillates and viscous oils that can be handled in a normal fashion as liquids at test temperatures between 15 and 35°C. Its application is restricted to liquids with vapor pressures below 600 mm Hg (80 kPa) and viscosities below about 15 000 cSt (mm /s) at the temperature of test.
- 1.2 This test method should not be applied to samples so dark in color that the absence of air bubbles in the sample cell cannot be established with certainty. For the determination of density in crude oil samples use Test Method D5002.
- 1.3 The accepted units of measure for density are grams per millilitre or kilograms per cubic metre.
- 1.4 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 and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Notes 1 and 2.
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D5002-99 Standard Test Method for Density and Relative Density of Crude Oils by Digital Density Analyzer

- 1.1 This test method covers the determination of the density or relative density of crude oils that can be handled in a normal fashion as liquids at test temperatures between 15 and 35°C. This test method applies to crude oils with high vapor pressures provided appropriate precautions are taken to prevent vapor loss during transfer of the sample to the density analyzer.
- 1.2 This test method was evaluated in round robin testing using crude oils in the 0.75 to 0.95 g/mL range. Lighter crude oil can require special handling to prevent vapor losses. Heavier crudes can require measurements at higher temperatures to eliminate air bubbles in the sample.
- 1.3 The accepted units of measurement of density are grams per millilitre and kilograms per cubic metre.
- 1.4 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 and

health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Notes 1, 2, and 3.

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Viscosity

D445-97 Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)

- 1. Scope
- 1.1 This test method specifies a procedure for the determination of the kinematic viscosity, [nu], of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, [eta], can be obtained by multiplying the measured kinematic viscosity by the density, [rho], of the liquid.

Note 1-For the measurement of the kinematic viscosity and viscosity of bitumens, see also Test Methods D2170 and D2171.

- 1.2 The result obtained from this test method is dependent upon the behavior of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behavior). If, however, the viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behavior, have been included.
- 1.3 The values stated in SI units are to be regarded as the standard.
- 1.4 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 and health practices and determine the applicability of regulatory limitations prior to use.
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D4486-91(1996)e1 Standard Test Method for Kinematic Viscosity of Volatile and Reactive Liquids

- 1. Scope
- 1.1 This test method covers the measurement of kinematic viscosity of transparent, Newtonian liquids which because of their reactivity, instability, or volatility cannot be used in conventional capillary kinematic viscometers. This test method is applicable up to 2 X 10 N/m (2 atm) pressure and temperature range from -53 to +135°C (-65 to +275° F).

- 1.1.1 For the measurement of the kinematic viscosity of other liquids, see Test Method D445.
- 1.2 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 and health practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements, see 6.2, 6.3, and 6.4.
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Surface and Interfacial Tension

D971-99a Standard Test Method for Interfacial Tension of Oil Against Water by the Ring Method

- 1. Scope
- 1.1 This test method covers the measurement of, under nonequilibrium conditions, the interfacial tension of mineral oils against water.
- 1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
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D3825-90(1995) Standard Test Method for Dynamic Surface Tension by the Fast-Bubble Technique

- 1. Scope
- 1.1 This test method covers the determination of the specific free energy of a liquid-gas surface a short time after formation of the surface.
- 1.2 It is applicable to liquids with vapor pressures up to 30.0 kPa (225 torr) and kinematic viscosities up to 4.0 mm /s (4.0 cSt) at the test temperature. Higher viscosities have not yet been investigated.
- 1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.
- 1.4 This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements, see 7.3, 7.4, and 7.5.

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D1331-89(1995) Standard Test Methods for Surface and Interfacial Tension of Solutions of Surface-Active Agents

1. Scope

- 1.1 These test methods cover the determination of surface tension and interfacial tension of solutions of surface-active agents, as defined in Definitions D459. Two methods are covered as follows: Method A-Surface Tension. Method B-Interfacial Tension.
- 1.2 Method A is written primarily to cover aqueous solutions of surface-active agents, but is also applicable to nonaqueous solutions and mixed solvent solutions.
- 1.3 Method B is applicable to two-phase solutions. More than one solute component may be present, including solute components that are not in themselves surface-active.
- 1.4 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.
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D2285-99 Standard Test Method for Interfacial Tension of Electrical Insulating Oils of Petroleum Origin Against Water by the Drop-Weight Method

- 1.1 This test method covers a comparatively rapid procedure particularly applicable for field use for measuring, under nonequilibrium conditions, the interfacial tensions of electrical insulating oils of petroleum origin against water. This test method has been shown by experience to give a reliable indication of the presence of hydrophilic compounds. This test method may not be applicable for highly viscous insulating fluids.
- 1.2 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 and health practices and determine the applicability of regulatory limitations prior to use.
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Porosity and Pore Volume Distribution

D4404-84(1998)e1 Standard Test Method for Determination of Pore Volume and Pore Volume Distribution of Soil and Rock by Mercury Intrusion Porosimetry

- 1.1 This test method covers the determination of the pore volume and the pore volume distributions of soil and rock by the mercury intrusion porosimetry method. The range of apparent diameters of pores for which this test method is applicable is fixed by the operant pressure range of the testing instrument. This range is typically between apparent pore entrance diameters of about 100 [mu]m and 2.5 nm (0.0025 [mu]m). Larger pores must be measured by another method.
- 1.2 Mercury intrusion porosimetry is useful only for measuring pores open to the outside of a soil or rock fragment; mercury intrusion porosimetry will not give the volume of any pores completely enclosed by surrounding solids. This test method will give only the volume of intrudable pores that have an apparent diameter corresponding to a pressure within the pressurizing range of the testing instrument.
- 1.3 The intrusion process proceeds from the outside of a fragment toward its center. Comparatively large interior pores can exist that have smaller pores as the only means of access. Mercury intrusion porosimetry will incorrectly register the entire volume of these "inkbottle" pores as having the apparent diameter of the smaller access pores. In a test sample, inter-fragment pores can exist in addition to intra-fragment pores (see Section 3 for definitions). The inter-fragment pores will vary in size and volume depending on the size and shape of the soil or rock fragments and on the manner in which the fragments are packed together. It is possible that some inter-fragment pores can have the same apparent diameter as some intra-fragment pores. When this occurs this test method cannot distinguish between them. Thus, the test method yields an intruded pore volume distribution that is in part dependent upon the packing of multifragment samples. However, most soils and rocks have intra-fragment pores much smaller than the inter-fragment pores. This situation leads to a bi-modal pore size distribution and the distinction between the two classes of pores can then be made (see Figs. 1 and 2).
- 1.4 Mercury intrusion may involve the application of high pressures to the sample. This may result in a temporary, or permanent, or temporary and permanent alteration in the pore geometry. Generally, soils and rocks are composed of comparatively strong solids and are less subject to these alterations than certain other materials. However, the possibility remains that the use of this test method may alter the natural pore volume distribution that is being measured.
- 1.5 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific precaution statements, see Section 8.
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F1815-97 Standard Test Method for Saturated Hydraulic Conducitivity, Water Retention, Porosity, Particle Density, and Bulk Density of Putting Green and Sports Turf Root Zones

1. Scope

- 1.1 These test methods cover the measurements of saturated hydraulic conductivity, water retention, porosity (including distribution of capillary and air-filled porosity at a known matrix potential), and particle and bulk densities on root zone mixes to be used for construction and top dressing of golf course putting greens including United States Golf Association (USGA) recommended greens or other highly trafficked turfgrass areas.
- 1.2 Water retention is not a required measurement for USGA Recommended greens. Its inclusion in these test methods is for the benefit of those who wish to continue to obtain such data. Likewise, bulk density is no longer a physical parameter required in the evaluation of USGA root zone mixes, but is must be determined for calculation of total and capillary porosity.
- 1.3 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.
- 1.4 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 and health practices and determine the applicability of regulatory limitations prior to use.
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Permeability

D2434-68(1994)e1 Standard Test Method for Permeability of Granular Soils (Constant Head)

- 1.1 This test method covers the determination of the coefficient of permeability by a constant-head method for the laminar flow of water through granular soils. The procedure is to establish representative values of the coefficient of permeability of granular soils that may occur in natural deposits as placed in embankments, or when used as base courses under pavements. In order to limit consolidation influences during testing, this procedure is limited to disturbed granular soils containing not more than 10% soil passing the 75-[mu]m (No. 200) sieve.
- 1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
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D5856-95 Standard Test Method for Measurement of Hydraulic Conductivity of Porous Material Using a Rigid-Wall, Compaction-Mold Permeameter

1. Scope

- 1.1 This test method covers laboratory measurement of the hydraulic conductivity (also referred to as coefficient of permeability) of laboratory-compacted materials with a rigid-wall, compaction-mold permeameter.
- 1.2 This test method may be used with laboratory-compacted specimens that have a hydraulic conductivity less than or equal to 1 X 10 -5 m/s. The hydraulic conductivity of compacted materials that have hydraulic conductivities greater than 1 X 10 -5 m/s may be determined by Test Method D2434.
- 1.3 The values stated in SI units are to be regarded as the standard, unless other units are specifically given. By tradition in U.S. practice, hydraulic conductivity is reported in centimetres per second, although the common SI units for hydraulic conductivity are metres per second.
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D5084-90(1997) Standard Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter

- 1.1 This test method covers laboratory measurement of the hydraulic conductivity (also referred to as coefficient of permeability) of water-saturated porous materials with a flexible wall permeameter.
- 1.2 This test method may be utilized with undisturbed or compacted specimens that have a hydraulic conductivity less than or equal to 1 X 10-5 m/s (1 X 10-3 cm/s).
- 1.3 The hydraulic conductivity of materials with hydraulic conductivities greater than 1 X 10-5 m/s may be determined by Test Method D2434.
- 1.4 The values stated in SI units are to be regarded as the standard, unless other units are specifically given. By tradition in U.S. practice, hydraulic conductivity is reported in centimetres per second, although the common SI units for hydraulic conductivity are metres per second.
- 1.5 This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
- 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 and health practices and determine the applicability of regulatory limitations prior to use.

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Capillary Pressure

D3152-72(1994)e1 Standard Test Method for Capillary-Moisture Relationships for Fine-Textured Soils by Pressure-Membrane Apparatus

1. Scope

1.1 This test method covers the determination of capillary-moisture properties of fine-textured soils as indicated by the moisture content - moisture tension relationships determined by pressure-membrane apparatus using tensions between 1 and 15 atm (101 and 1520 kPa). Moisture tension (matrix suction) is defined as the equivalent negative gage pressure, or suction, in soil moisture. The test result is a moisture content which is a measure of the water retained in the soil subjected to a given soil - water tension (or at an approximately equivalent height above the water table).

Note 1-For determination of capillary-moisture relationships of coarse- and medium-textured soils, refer to Test Method D2325.

- 1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
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D2325-68(1994)e1 Standard Test Method for Capillary-Moisture Relationships for Coarse- and Medium-Textured Soils by Porous-Plate Apparatus

1. Scope

1.1 This test method covers the determination of capillary-moisture relationships for coarse- and medium-textured soils as indicated by the soil-moisture tension relations for tensions between 10 and 101 kPa (0.1 and 1 atm). Under equilibrium conditions, moisture tension is defined as the equivalent negative gage pressure, or suction, corresponding to a soil moisture content. This test method determines the equilibrium moisture content retained in a soil subjected to a given soil-water tension. This test method is not suitable for very fine-textured soils.

Note 1-For determination of capillary-moisture relationships for fine-textured soils, refer to Test Method D3152.

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D425-88(1994) Standard Test Method for Centrifuge Moisture Equivalent of Soils

- 1. Scope
- 1.1 This test method covers the determination of the moisture equivalent of soil in the laboratory by means of a centrifuge technique.
- 1.2 This test method is limited to disturbed specimens of coarse-grained soils having fines of low plasticity such as SP, SW, SC-SM, or SM soils. The test is limited to soils passing the 2.00-mm sieve or that fraction of a soil passing a 2.00-mm sieve.

Note 1-Test Method D3152 or Test Method D2325 should be used to evaluate the capillary-moisture relations of fine-grained soils and coarse-grained soils having fines of medium to high plasticity, undisturbed soils, and soils at specific desired units weights.

- 1.3 The test method is temperature-dependent, and consistent comparable results can be obtained only if the tests are performed under a constant-temperature condition.
- 1.4 The values stated in SI units are to be regarded as the standard.
- 1.5 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
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Sampling

D4696-92e1 Standard Guide for Pore-Liquid Sampling from the Vadose Zone

- 1. Scope
- 1.1 This guide discusses equipment and procedures used for sampling pore-liquid from the vadose zone (unsaturated zone). The guide is limited to *in-situ* techniques and does not include soil core collection and extraction methods for obtaining samples.
- 1.2 The term "pore-liquid" is applicable to any liquid from aqueous pore-liquid to oil. However, all of the samplers described in this guide were designed, and are used to sample aqueous pore-liquids only. The abilities of these samplers to collect other pore-liquids may be quite different than those described.
- 1.3 Some of the samplers described in this guide are not currently commercially available. These samplers are presented because they may have been available in the past, and may be encountered at sites with established vadose zone monitoring programs. In addition, some of these designs are particularly suited to specific situations. If needed, these samplers could be fabricated.

- 1.4 The values stated in SI units are to be regarded as the standard.
- 1.5 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
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Bulk Density of Soil

D4564-93 Standard Test Method for Density of Soil in Place by the Sleeve Method

- 1. Scope
- 1.1 This test method covers the determination of the density of soil in place by the sleeve method.
- 1.2 The sleeve method of determining the density of soil in place is used for cohesionless, granular soils for which other methods of determining the density (sand cone, test pit, and the like) may not be practical. Typically, the sleeve method is applicable for soils that are predominantly fine gravel size, with a maximum of 5% fines, and a maximum particle size of 3/4 in. (19.0 mm).
- Note 1-There have been other methods developed for testing cohesionless soils. Compared to other methods, this procedure is convenient for field construction control testing because smaller and lighter equipment is used and the test can be performed in a smaller area.
- 1.3 A calibration equation is necessary in the application of this test method to obtain a reliable value of the in-place density of the soil (see Annex A1). The calibration equation is used to calculate the density of the soil in place from the mass of dry soil per inch of test hole measured by the sleeve method.
- 1.3.1 The calibration equation is predetermined for a particular soil type that is to be tested. When the soil changes significantly in either gradation or particle angularity, the calibration equation may have to be adjusted or redefined before the sleeve method can be used.
- 1.3.2 There may be certain soils meeting the general description in 1.2 for which a calibration equation may not be appropriate due to unsatisfactory correlation of the data. The sleeve method would not be applicable for these soils.
- 1.3.3 There may be certain soils meeting the description in 1.2 for which the calibration equation may be applicable only for a certain range of densities. The sleeve method will give reliable values of the density in place only within that range of densities.
- 1.4 It is common practice in the engineering profession to concurrently use pounds to represent both a unit of mass (lbm) and a unit of force (lbf). This implicitly combines two separate systems of units; that is, the absolute system and the gravitational system. It is scientifically undesirable

to combine the use of two separate sets of inch-pound units within a single standard. This standard has been written using the gravitational system of units when dealing with the inch-pound system. In this system the pound (lbf) represents a unit of force (weight). However, the use of balances or scales recording pounds of mass (lbm), or the recording of density in lbm/ft should not be regarded as nonconformance with this test method.

- 1.5 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
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D2937-94 Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method

- 1.1 This test method covers the determination of in-place density of soil by the drive-cylinder method. The test method involves obtaining a relatively undisturbed soil sample by driving a thin-walled cylinder and the subsequent activities for the determination of in-place density. When sampling or in-place density is required at depth, Test Method D1587 should be used.
- 1.2 This test method is not appropriate for sampling organic soils which can compress upon sampling, very hard natural soils and heavily compacted soils which cannot be easily penetrated by the drive sampler, soils of low plasticity which will not be readily retained in the cylinder, or soils which contain appreciable amounts of gravel (particles coarser than 4.75 mm). The presence of particles coarser than 4.75 mm may introduce significant errors in density measurements by causing voids along the wall of the cylinder during driving, and when coarse materials have to be dislodged by the trimming of the sample obtained by the cylinder.
- 1.3 This test method is limited to the procedures necessary for obtaining specimens suitable for determining the in-place density and water content of certain soils. The procedures and precautions necessary for selecting locations and obtaining undisturbed samples suitable for laboratory testing or otherwise determining engineering properties is beyond the scope of this test method.
- 1.4 It is common practice in the engineering profession to concurrently use pounds to represent both a unit of mass (lbm) and a unit of force (lbf). This implicitly combines two separate systems of units, that is, the absolute system and the gravitational system. It is scientifically undesirable to combine the use of two separate sets of inch-pound units within a single standard. This test method has been written using the gravitational system of units when dealing with the inch-pound system. In this system the pound (lbf) represents a unit of force (weight). However, the use of balances or scales recording pounds of mass (lbm), or the recording of density in lbm/ft should not be regarded as nonconformance with this test method.
- 1.5 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 and health practices and determine the applicability of regulatory limitations prior to use.

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Soil Sampling

D6282-98 Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations

- 1.1 This guide addresses direct push soil samplers, which also may be driven into the ground from the surface or through pre-bored holes. The samplers can be continuous or discrete interval units. Samplers are advanced by a combination of static push, or impacts from hammers, or vibratory methods, or a combination thereof, to the depth of interest. The guide does not cover open chambered samplers operated by hand such as augers, agricultural samplers operated at shallow depths, or side wall samplers. This guide does not address single sampling events in the immediate base of the drill hole using rotary drilling equipment with incremental drill hole excavation. Other sampling standards, such as Test Methods D 1586 and D 1587 and Practice D 3550 apply to rotary drilling activities. This guide does not address advancement of sampler barrel systems with methods that employ cuttings removal as the sampler is advanced. Other drilling and sampling methods may apply for samples needed for engineering and construction applications.
- 1.2 Guidance on preservation and transport of samples, as given in Guide D 4220, may or may not apply. Samples for chemical analysis often must be subsampled and reserved for chemical analysis using special techniques. Practice D 3694 provides information on some of the special techniques required. Additional information on environmental sample preservation and transportation is available in other references (1, 2). Samples for classification may be preserved using procedures similar to Class A. In most cases, a direct push sample is considered as Class B in Practice D 4220 but is protected, representative, and suitable for chemical analysis. The samples taken with this practice do not usually produce Class C and D (with exception of thin wall samples of standard size) samples for testing for engineering properties, such as shear strength and compressibility. Guide D 4700 has some information on mechanical soil sampling devices similar to direct push techniques, however, it does not address most direct push sampling methods. If sampling is for chemical evaluation in the Vadose Zone, consult Guide D 4700 for any special considerations.
- 1.3 Field methods described in this guide, include the use of discreet and continuous sampling tools, split and solid barrel samplers and thin walled tubes with or without fixed piston style apparatus.
- 1.4 Insertion methods described include static push, impact, percussion, other vibratory/sonic driving, and combinations of these methods using direct push equipment adapted to drilling rigs, cone penetrometer units, and specially designed percussion/direct push combination machines. Hammers providing the force for insertion include drop style, hydraulically activated, air activated and mechanical lift devices.
- 1.5 Direct push soil sampling is limited to soils and unconsolidated materials that can be penetrated with the available equipment. The ability to penetrate strata is based on hammer

energy, carrying vehicle weight, compactness of soil, and consistency of soil. Penetration may be limited or damage to samplers and conveying devices can occur in certain subsurface conditions, some of which are discussed in 5.5. Successful sample recovery also may be limited by the ability to retrieve tools from the borehole. Sufficient retract force must be available when attempting difficult or deep investigations.

- 1.6 This guide does not address the installation of any temporary or permanent soil, ground water, vapor monitoring, or remediation devices.
- 1.7 The practicing of direct push techniques may be controlled by local regulations governing subsurface penetration. Certification, or licensing requirements, or both, may need to be considered in establishing criteria for field activities.
- 1.8 The values stated in SI units are to be regarded as standard; however, dimensions used in the drilling industry are given in inch-pound units by convention. Inch-pound units are used where necessary in this guide.
- 1.9 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 and health practices and determine the applicability of regulatory limitations prior to use.
- 1.10 This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.
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D6169-98 Standard Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations

- 1.1 This guide covers guidance for the selection of soil and rock sampling devices used with drill rigs for the purpose of characterizing *in situ* physical and hydraulic properties, chemical characteristics, subsurface lithology, statigraphy, and structure, and hydrogeologic units in environmental investigations.
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D1452-80(1995)e1 Standard Practice for Soil Investigation and Sampling by Auger Borings

1. Scope

- 1.1 This practice covers equipment and procedures for the use of earth augers in shallow geotechnical exploration. This practice does not apply to sectional continuous flight augers.
- 1.2 This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
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D1587-94 Standard Practice for Thin-Walled Tube Geotechnical Sampling of Soils

- 1. Scope
- 1.1 This practice covers a procedure for using a thin-walled metal tube to recover relatively undisturbed soil samples suitable for laboratory tests of structural properties. Thin-walled tubes used in piston, plug, or rotary-type samplers, such as the Denison or Pitcher, must comply with the portions of this practice which describe the thin-walled tubes (5.3).
- Note 1-This practice does not apply to liners used within the above samplers.
- 1.2 The values stated in both inch-pound and SI units are to be regarded as the standard.
- 1.3 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 and health practices and determine the applicability of regulatory limitations prior to use.
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D1586-99 Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils

- 1.1 This test method describes the procedure, generally known as the Standard Penetration Test (SPT), for driving a split-barrel sampler to obtain a representative soil sample and a measure of the resistance of the soil to penetration of the sampler.
- 1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a

specific precautionary statement, see 5.4.1. The values stated in inch-pound units are to be regarded as the standard.

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NAPL Saturation

D5369-93(1998) Standard Practice for Extraction of Solid Waste Samples for Chemical Analysis Using Soxhlet Extraction

- 1. Scope
- 1.1 This practice describes standard procedures for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sediments, sludges, and granular wastes using Soxhlet extraction.
- 1.1.1 The sample must be suitable for being mixed with the sample drying agent, sodium sulfate or magnesium sulfate, to provide drying of all sample surfaces.
- 1.2 This practice, when used in conjunction with Test Method D5368 is applicable to the determination of the total solvent extractable content (TSEC) of a soil, sediment, sludge, or granular solid waste and depends upon the solvent chosen for extraction.
- 1.3 This practice is limited to solvents having boiling points below the boiling point of water at ambient pressure.
- 1.4 The solvent extract obtained by this practice may be analyzed for total or specific nonvolatile and semivolatile organic compounds, but may require sample clean-up procedures prior to specific compound analysis.
- 1.4.1 This practice provides sample extracts suitable for analysis by various techniques such as gas chromatography with flame ionization detection (GC/FID) or gas chromatography with mass spectrometric detection (GC/MS).
- 1.5 This practice is recommended only for solid samples that can pass through a 10-mesh sieve (approximately 2-mm openings), or are less than 2 mm in thickness.
- 1.6 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific precautions see Section 9.
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D5368-93 Standard Test Methods for Gravimetric Determination of Total Solvent Extractable Content (TSEC) of Solid Waste Samples

1. Scope

1.1 These test methods describe standard procedures for gravimetrically determining the total nonvolatile and semi-volatile organic content of solvent extracts from soils or solid wastes. The following methods are included:

Section Method A---Micro-Determination of TSEC 11-13 Method B---Evaporating Dish Procedure 14-16 Method C---Boiling Flask Procedure 17-19

- 1.2 These methods are used after a solvent extract is obtained from a soil or solid waste. For these methods to be applicable, the extraction solvent must have a boiling point less than that of water at ambient pressure.
- 1.3 The total solvent extractable content (TSEC) of a soil, sediment, sludge, or solid waste depends upon the solvent and method used for the extraction procedure.
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Appendix B — Derivation of Methods for Estimating Relative Permeability, Average Conductivity to Oil, and Transmissivity to Oil from Production Data

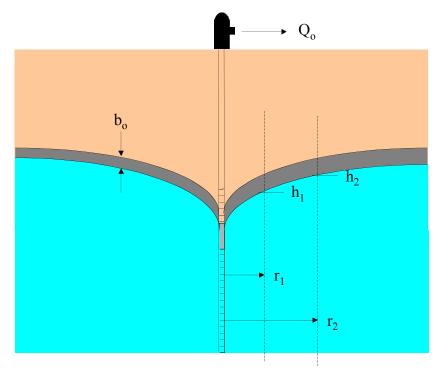


Figure B-1 — Basis for derivation of production equations*

Darcy's equation for flow

$$q_o = -\frac{kk_{ro}}{\mu_o} \left(\frac{dP_o}{dr} + \rho_o g \frac{dz}{dr} \right) \tag{B-1}$$

From the definition of capillary pressure $P_{o}=P_{c}+P_{w}$, by substitution

$$q_o = -\frac{kk_{ro}}{\mu_o} \left(\frac{dP_c}{dr} + \frac{dP_w}{dr} + \rho_o g \frac{dz}{dr} \right)$$
 (B-2)

From the definition of hydraulic head $P_{_{\!w}}=\rho_{_{\!w}}gh_{_{\!w}}-\rho_{_{\!w}}gz$, by substitution

$$q_o = -\frac{kk_{ro}}{\mu_o} \left(\frac{dP_c}{dr} + \rho_w g \frac{dh_w}{dr} - \rho_w g \frac{dz}{dr} + \rho_o g \frac{dz}{dr} \right)$$
 (B-3)

Recognizing $\frac{dh_w}{dr} = \frac{dz}{dr}$ at the water table and $\frac{dP_c}{dr}$ to be small

$$q_o = -\frac{kk_{ro}\rho_o g}{\mu_o} \frac{dh_w}{dr}$$
 (B-4)

Area = circumference ($2\pi r$) times height of continuous product (b_{o}) \bar{k}_{ro} = average relative permeability over the height of continuous product

$$Q_o = \frac{2\pi r b_o k \bar{k}_{ro} \rho_o g}{\mu_o} \frac{dh_w}{dr}$$
 (B-5)

Note by convention production at a well (\mathcal{Q}_o) is positive; therefore there is a sign change in (B-5).

Input Parameters for Sample Calculation

$$Q_0=100~{\rm gal/day} \qquad \mu_o=0.62~{\rm centipoise} \qquad r_{\rm I}=10~{\rm feet} \qquad r_2=100~{\rm feet}$$

$$b=1~{\rm foot} \qquad g=9.807~{\rm m/s^2} \qquad h_{\rm I}=95~{\rm feet} \qquad h_2=100~{\rm feet}$$

$$\rho_o=0.8~{\rm gm/cm^3} \qquad k=10^{-7}~{\rm cm^2}$$

Following Theim (1906)

$$\frac{Q_o \mu_o}{2\pi b_o k \bar{k}_{ro} \rho_o g} \frac{I}{r} dr = dh_w$$
 (B-6)

$$\frac{Q_{o}\mu_{o}}{2\pi b_{o}k\bar{k}_{ro}\rho_{o}g}\int_{r_{1}}^{r_{2}}\frac{1}{r}dr=\int_{h_{1}}^{h_{2}}dh_{w} \tag{B-7}$$

$$\frac{Q_o \mu_o}{2\pi b_o k \overline{k}_{r_o} \mu_o g} ln \frac{r_2}{r_I} = h_2 - h_I$$
(B-8)

$$\bar{k}_{ro} = \frac{Q_o \mu_o}{2\pi b_o k \rho_o g(h_2 - h_1)} ln \left(\frac{r_2}{r_2}\right)$$
(B-9)

 \overline{k}_{ro} Sample calculation (Equation B-9)

$$\bar{k}_{ro} = 0.027$$

The average oil conductivity is

$$\overline{K}_{o} = \frac{k\overline{k}_{ro}\rho_{o}g}{\mu_{o}} = \frac{Q_{o}ln\left(\frac{r_{2}}{r_{I}}\right)}{2\pi b_{o}(h_{2} - h_{I})}$$
(B-10)

 $\overline{K}_{\scriptscriptstyle o}$ Sample calculation (Equation B-10)

$$\overline{K}_o = 3.5 \times 10^{-4} \frac{cm}{sec}$$

The Transmissivity to oil is

$$T_o = \overline{K}_o b_o = \frac{Q_o ln \left(\frac{r_l}{r_2}\right)}{2 \left(h_l - h_2\right)}$$
(B-11)

 T_o Sample Calculation (Equation B-11)

$$T_o = 0.091 \frac{m^2}{day}$$

Appendix C — Derivation of Decline Curve Method

Note the following derivation is based on production in terms of mass per time. This solution is more general than that presented in Section 4.2. To convert the following to the volume per time solutions presented in Section 4.2 divide contaminant mass by liquid density.

Consider a finite three-dimensional subset of the subsurface environment that contains a released contaminant (Figure C-1). The mass per unit volume porous media (concentration) at any point in the source zone is:

$$\rho_{T}(\overline{x}) = \rho_{w}(\overline{x}) + \rho_{v}(\overline{x}) + \rho_{s}(\overline{x}) + \rho_{n}(\overline{x})$$
 (C-1)

where ρ_w , ρ_v , ρ_s and ρ_m is the mass per unit volume porous media present in the water, air, sorbed, and nonaqueous phases, respectively. The vector (\bar{x}) describes the location of the center of a representative element of volume located in the source zone.

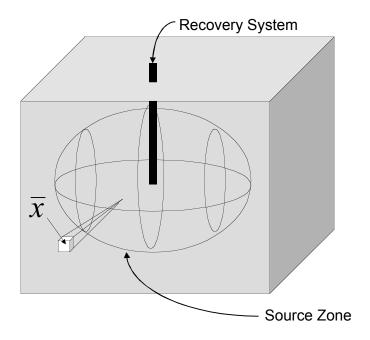


Figure C-1 — Hypothetical subsurface source zone

The total mass in the source zone at time (t) is defined as:

$$M_T(t) = \int_{V_s} \rho_T(t) dV$$
 (C-2)

where V_{s} is a volume subset of the subsurface greater than or equal to the source zone volume.

Next, consider a sink that extracts either air or an aqueous phase and has a capture zone that includes the entire source zone. The rate of mass removal from the source zone via the sink at time (t) is referred to as production and is defined as:

$$P(t) = -\frac{dM_T(t)}{dt}$$
 (C-3)

Integration of the production rate from the initiation of the remediation (t = 0) to a time of interest (t) yields (4), the cumulative mass produced at time t.

$$M_P(t) = \int_0^t P(t)dt \tag{C-4}$$

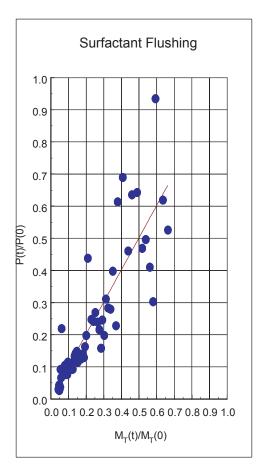
P(t) and $M_P(t)$ are quantities that can be readily measured. In the case of a soil gas extraction system, P(t) is determined as the product of the air flow rate and the concentration of contaminants. In the case of a free product recovery system, $M_R(t)$ is determined as product of the accumulated volume of free product and the density of the free product. By mass balance it follows that the contaminant mass produced from a source zone $M_P(t)$ can be defined as:

$$M_P(t) = M_T(0) - M_T(t)$$
 (C-5)

Next, mathematical expressions for P(t) and $M_P(t)$ are developed. As a general observation it is noted that as contaminant mass is recovered, production rates typically decay. Furthermore, it is observed that production rates are a function of the mass present in the source zone (e.g., large releases have the potential for larger production rates). As such, it is proposed that production rates follow a first order decay equation:

$$\frac{dM_T}{dt} = -\alpha M_T \tag{C-6}$$

Alpha (α) is a rate constant (1/T) that characterizes mass transfer processes from a given source zone (without additional releases) using a given cleanup technology operated in a constant mode (e.g., flow rate, number of recovery wells). To test this hypothesis, Figure C-2 plots the normalized production rate (P(t)/P(0)) as a function of the normalized fraction of the contaminant mass remaining in the source zone ($M_T(0)-M_P(t))/M_T(0)$). Figure C-2 shows normalized results from soil vapor extraction, and surfactant flushing remedies. Principle attributes of these systems include: 1) they address the entire release, 2) they were operated in near constant modes, 3) no additional releases were occurring, and 4) the dominant mechanisms governing mass transfer in the source zone largely remained constant.



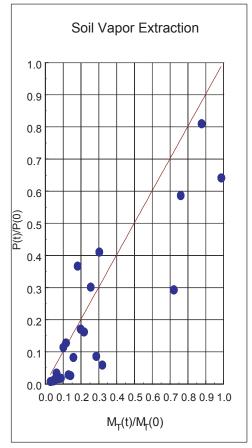


Figure C-2 — Normalized production data vs. normalized mass in source zone

Based on Figure C-2 data it is concluded that the assumption of production rates linearly proportional to the mass present is valid for the data sets presented. From this it follows that:

$$P(t) = \alpha M_T(0)e^{-\alpha t} \tag{C-7}$$

$$M_P(t) = M_T(0) - M_T(0)e^{-\alpha t}$$
 (C-8)

$$M_T(0) = -\frac{P(0)}{\alpha} \tag{C-9}$$

$$t_{1/2} = \frac{0.693}{\alpha} \tag{C-10}$$

The half life $(t_{1/2})$ describes the time required to deplete half of the mass remaining in a source zone. The half-life is a characteristic property of a given recovery system, applied to a given source zone. The half life $(t_{1/2})$ can be used to compare remedies applied at different sites. Methods to estimates α and $M_T(0)$ are presented in Section 4.2.

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