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CALIBRATION TECHNIQUE FOR THE NEUTRON SURFACE
MOISTURE MEASUREMENT SYSTEM

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CALIBRATION TECHNIQUE FOR THE NEUTRON SURFACE MOISTURE MEASUREMENT SYSTEM

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Abstract: A technique for calibrating the response of a surface neutron moisture measurement probe to material moisture concentration has been devised. Tests to ensure that the probe will function in the expected in-tank operating environment are also outlined.

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CALIBRATION TECHNIQUE FOR THE NEUTRON SURFACE MOISTURE MEASUREMENT SYSTEM

1.0 INTRODUCTION

The Surface Moisture Measurement System (SMMS) is designed to measure the moisture concentration near the surfaces of the wastes located in the Hanford Site tank farms. This procedure details how moisture standards will be built, controlled, and utilized both to demonstrate that the moisture probe meets requirements in the *Design Requirements Document (DRD) for the Surface Moisture Measurement System* (Stokes et al. 1995) and to calibrate, or benchmark, computer models that predict probe detector responses to moisture. Planned tests of detector responses to changes in probe temperature and to ambient gamma ray fields are also explained. The primary purpose of the experimental tests described in this procedure is to make possible interpretation of in-tank moisture data using computer modeled predictions of detector responses to waste properties.

The surface moisture measurement system functions by emitting high energy neutrons that are moderated or slowed in the surrounding waste, primarily through interactions with hydrogen nuclei (Hearst and Carlson 1994). The SMMS consists of a radioactive Cf-252 neutron source and three neutron detectors. These thermal and epithermal neutron detectors detect fractions of the moderated neutrons at rates related to the hydrogen concentration of the surrounding waste. Calibrations and modeling then are used to obtain estimates of the moisture concentration of the underlying waste. It is important that the calibration standards be constructed and maintained so that their elemental composition, moisture concentration, bulk density, and physical dimensions are well known. These parameters will be inputs to the computer models used to predict probe responses so that errors in each of these will likely translate to errors in adjustable parameters associated with the computer model of the probe. Because this model of the probe will be used with best estimates of tank waste composition and density to determine calibration curves for tank data interpretation, it is important to develop a probe model that contains minimal systematic uncertainty.

2.0 STANDARD PREPARATION

In order to interpret data from the SMMS one must have a way of determining the response of the instrument to moisture in material similar to the waste encountered in the tanks. It is not possible to perform the calibrations on actual waste because the waste is radioactive and the actual moisture content is not controlled. The waste will be simulated using a neutron transport computer code, Monte Carlo N-Particle (MCNP), in which the different waste configurations can be easily modeled (LANL 1993). The first step in this process is to validate the computer models of the detectors and geometry of the SMMS. To accomplish this task the response of the SMMS to mixtures of sand and hydrated alumina will be measured. The weights of these components, along with their chemical compositions can be used to establish the hydrogen concentrations of the mixture. Since the slowing down of neutrons is dominated by the hydrogen content of the mixture, these samples will be similar to actual tank configurations as far as the neutron transport is concerned. MCNP will then be used to calculate the detector responses for these same configurations. Verification of the computer code and the detector responses will be accomplished when the measured and calculated measurements agree. This agreement must be within the combined uncertainty in the makeup of the standards.

This section will discuss the preparation of the moisture standards. The procedures will be given in detail so that in the future sets of moisture standards equivalent to this set of standards could be constructed.

2.1 BACKGROUND

Results of initial probe design development using both computer modeling and prototype testing may be found in *Results of Modeling and Experimental Measurements for the Design of a Neutron Surface Moisture Measurement System* (Watson, et al. 1995). A prototype probe, very similar to the design used in the calculations performed by the computer modeling, was built and tested on multiple moisture test beds. These test beds were comprised of different size sand and gravel mixtures used to create matrices of different porosity. Each matrix, when saturated with water, contained a different water concentration by weight. The tests described in the design development report were meant to provide experimental confirmation that the computer model predictions of probe responses were reasonable so that probe construction could continue. The results also show that the neutron moderation technique, using the baseline design should produce the desired moisture measurement results.

Several minor unknowns and potential problems existed with the use of these water saturated simulants, as described in the Watson report. First, low moisture concentrations were impossible to simulate unless very fine, very dense sand was used. The density of this type of configuration would be much higher than the expected density of the Hanford waste. Second, water

evaporated from the samples, over long periods of time, so that remeasurements could not be made without refilling the containers. The weight of the sample after refilling was difficult to measure. There was additional uncertainty about the equivalent water concentration near the surfaces of the standards because the matrix uniformity was not well controlled at the surface. Entrained air bubbles were also found to be present in some of the tests beds after being filled with water. Nonuniform, or possibly changing distribution or amount of trapped air in the matrix would cause problems with relative moisture concentration determinations. Also, it was difficult to remove samples of the water saturated gravel mix for elemental analyses and moisture concentration measurements. Finally, it was not possible to adequately simulate geometric surface irregularities while maintaining water saturation of the matrix and air-filled void spaces around the irregularities. In spite of these potential problems, the water concentrations of the water saturated samples were estimated to be accurate to about 1% by weight for freshly made samples.

The verification standards for the calibration tests will be made using mixtures of sand, hydrated alumina ($Al_2O_3 \cdot 3(H_2O)$), and boron carbide. Water molecules bound to the alumina replace the free water found in the water saturated models, making it possible to simulate partially saturated conditions in different configurations. The hydrated alumina is chemically and physically stable and can be obtained in a grit size similar to that of fine sand. After mixing and compaction, the distributions of the two components are not likely to change. No loss of chemically bound water will occur at temperatures below 320 °F, and the chemical composition of the mixture as a function of depth can be obtained by sampling.

2.2 PURPOSE OF STANDARDS

The verification standards are designed to allow scientists to verify that the SMMS modeling results, obtained with MCNP, agree with SMMS experimental measurements made on each standard. This will ensure that the modeled configuration of the system (i.e. source, detectors, shielding) is correct and will demonstrate that this model can be used to predict detector responses to many varied moisture conditions. These standards are designed to provide a calibration of the input parameters of the computer code rather than a calibration of the SMMS. The computer code will then be used to provide libraries of data for a set of expected tank configurations. The assumed tank waste compositions will be used in the code along with different moisture concentrations and moisture gradients to produce expected responses for each of the detectors for all of the tank configurations. Comparison of these calculated data and data measured in the tanks will then allow an accurate determination of the average moisture concentration in the waste near the surface, and an estimate of the average profile of the moisture concentration to be determined for the actual tanks.

2.3 MATERIALS, SIZES, GEOMETRY, & LIST OF STANDARDS

The standard vessels are cylindrical aluminum barrels (about 0.5 cm wall thickness) with inner diameters of 76 cm and heights of 46 cm. Each of the vessels has a removable circular aluminum lid (76 cm OD, 0.08 cm thick) that can be placed in the barrel at the fill-height of the moisture matrix. The barrel to contain the 4 weight percent moisture mixture is large than the other barrels, with a diameter of about 95 cm and a height of about 65 cm. Each standard will be identified with a permanent marking, a unique number stamped or engraved upon the side of the barrel.

Six full standards with uniform moisture concentration distributions of 4, 8, 13, 19, 25, and 35 weight percent (wt%) water will be made. Figures 1 through 12 give a sketch and brief description of each of the standards. Four additional partially-filled standards containing uniformly distributed 19 wt% moisture will be constructed. These four partially-filled standards will be filled to heights of 4, 6, 8, and 10 cm. Four standards will be created with step gradients in the moisture concentration as a function of depth from the matrix surface. The matrix layers of these moisture gradient standards will be separated by thin sheets of aluminum foil to maintain the integrity of each layer as it is prepared. The first gradient standard will contain 8 wt% moisture matrix for the top 3 cm and 13 wt% moisture matrix for the remaining standard fill volume. The second standard will contain 8 wt% moisture in the top 3 cm, 13 wt% moisture in the next 3 cm, and 19 wt% moisture in the remainder of the standard. The third standard will contain 13 wt% moisture in the top 3 cm and 19 wt% moisture in the remainder of the standard. The final gradient standard will contain 13 wt% moisture in the top 3 cm, 19 wt% moisture in the next 3 cm, and 25 wt% moisture in the remainder of the standard. Two standards will be constructed that will provide an indication of the probe sensitivity to the average thermal neutron absorption cross-section of the matrix. These two standards will contain uniformly distributed boron carbide powder added to 19 wt% moisture matrix in amounts that make the total boron concentration equal to 0.004 and 0.008 wt%.

2.4 PROCEDURE FOR PREPARATION

The following section will discuss the considerations that went into the design of the standards and the details for the construction of these standards. The primary potential safety hazards during the construction of the moisture standards are: temperature hazards associated with any heating equipment, inhalation hazards associated with the mixing of the particulate moisture matrix, and mechanical hazards associated with the lifting of heavy objects and with the use of a mixer. Westinghouse Hanford Company (WHC) Industrial Safety will be consulted prior to the start of any of this work and a WHC Job Hazards Analyses Worksheet will be completed.

2.4.1 Material Surrounding Standards for Infinite Detector Response

Calculations indicate that, especially for the low moisture content barrels, structures outside of the barrel may influence the probe responses. These influences are expected to be relatively small for most of the standards, but would need to be accounted for in computer models of the standards. Because the nearby structures include objects which are not well characterized, such as the concrete floor, it would be difficult to accurately include them in the model. To make the standards effectively infinite, (that is no structure outside of the standard will influence the results of the measurements) the standards will be surrounded by known materials. Polyethylene sheet, an excellent neutron moderator and reflector, will be placed under most of the standards to ensure that they are completely isolated from the floor beneath them. The thickness of these polyethylene sheets will vary for each standard and the minimum thickness will be confirmed with calculations. Table 2-1 shows the expected thicknesses of polyethylene sheet to be used under each standard. Another way to isolate the standard from surrounding materials is to place the standard a large enough distance from materials that would have an effect so that the effect is negligible. Because the thin single-layer standards are used to give an indication of the depth investigated by each detector, using polyethylene under them to isolate them would produce an undesirable large background signal. Instead, the thin single-layer standards will be placed upon thin aluminum platforms that are located 122 cm above the floor. Except for the thin layer standards, all standards will be constructed on palettes so that they may be more easily moved for weighing.

Table 2-1. The thickness of polyethylene planned to be used under each standard.

<u>ID#</u>	<u>Standard Description (Moisture)</u>	<u>Polyethylene Thickness (cm)</u>
1	4 wt% uniform	10.2
2	8 wt% uniform	10.2
3	13 wt% uniform	5.1
4	19 wt% uniform	5.1
5	25 wt% uniform	2.6
6	35 wt% uniform	2.6
7	8 wt% / 13 wt% layered	5.1
8	8 wt% / 13 wt% / 19 wt% layered	5.1
9	13 wt% / 19 wt% layered	5.1
10	13 wt% / 19 wt% / 25 wt% layered	2.6
11	19 wt% with low boron content	2.6
12	19 wt% with high boron content	2.6
13	19 wt%, 4 cm thick	None
14	19 wt%, 6 cm thick	None
15	19 wt%, 8 cm thick	None
16	19 wt%, 10 cm thick	None

The standards will be horizontally spaced from one another and from other structures above the floor, including the walls. The edge of each standard will be placed a minimum distance of 100 cm from the edge of any adjacent standards. The edge of every standard will also be placed a minimum distance of 150 cm from a wall or other vertical building structure. During experimental measurement data acquisition, no person or object will be allowed within 150 cm of the standard being used. The roof is more than 6 m above the floor and will therefore not affect the measurements.

2.4.2 Calibration of Measurement Tools and Instruments

The tools that will be used in these tests that will need calibration are the following:

- Small scale for weighing individual samples
(100 g \pm 0.1 g)
- Medium scale for weighing individual samples
(up to 100 kg \pm 0.03%)
- Large scale for weighing the total containers
(up to 340 kg \pm 0.04%)
- Rulers used for measuring the heights of the various fills
(60 cm \pm 0.04 cm)
- Solid state sensors used for temperature measurements.
(0 - 200°C \pm 0.1°C)
- Thermocouples used for temperature measurements.
(0 - 100°C \pm 1°C)
- Neutron and gamma-ray radiation survey instruments.
(0 - 300 mRem/h \pm 10 mRem/h neutron and 0 - 100 mR/h \pm 1 mR/h gamma)

The model name and serial number of each calibrated tool used in the preparation or tests will be recorded in an appropriate logbook. A copy of the current calibration sheet, when applicable, will also be obtained and placed in a logbook.

2.4.3 Standard Volume and Mixture Density Determinations

The determination of the volume as a function of fill depth of each standard will be one of the primary measurements in the determination of the density of the mixture. First, the volume of each barrel as a function of depth will be determined by weighing the barrel when filled with water for two different fill depths near the expected mixture fill depths. The density of water will be used to correlate these weights and fill depths with the fill volumes.

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The barrels will be on thick polyethylene sheets which will prevent the bottom from bowing and producing inaccurate volume measurements. All "filled" barrels will be filled with a sand and hydrated alumina mixture to a compacted height of 38 ± 2 cm, except the 4 wt% moisture barrel will be filled to a compacted height of 60 ± 3 cm.

The barrel mixtures will then be leveled. When the barrel is filled to the proper depth, the top will be leveled and the distance to the top of the barrel measured in several locations (at least four). The average of these measurements will be denoted H_{air} .

The procedure for determining the density of the mixed and compacted material will be as is described below.

[Note: All weights must be converted or measured in kilograms and all distances converted or measured in centimeters before use in the following equations.]

Measure the weight of the empty barrel, W_{barrel} .
Zero the scale with the empty barrel on it.

Fill the barrel to a height of below the desired matrix fill height (about 33 cm for most barrels) with water and measure both the distance from the top of the barrel to the surface of the fill substance (water), H_{f1} , and the weight of the fill substance (water), W_{f1} . The distance measurement is the average of four measurements taken at 90 degree increments around the barrel perimeter:

$$H_{f1} = (H_{f11} + H_{f12} + H_{f13} + H_{f14}) / 4 .$$

The uncertainty in the fill depth, H_{f1} , is given by

$$\sigma_{H_{f1}} = \frac{1}{4} \sqrt{\sigma_{H_{f11}}^2 + \sigma_{H_{f12}}^2 + \sigma_{H_{f13}}^2 + \sigma_{H_{f14}}^2}$$

where $\sigma_{H_{f1i}}$ are the estimated uncertainties in each measured depth, H_{f1i} .

Fill the barrel to a height above the expected matrix fill height (about 41 cm for most barrels) with water and measure both the distance from the top of the barrel to the surface of the fill substance (water), H_{f2} , and the weight of the fill substance (water), W_{f2} . The distance measurement is the average of four measurements taken at 90 degree increments around the barrel perimeter:

$$H_{f2} = (H_{f21} + H_{f22} + H_{f23} + H_{f24}) / 4 .$$

The uncertainty in this fill depth is calculated following the example for fill depth H_{f1} .

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For barrel fill depths near or between H_{f1} and H_{f2} , the weight of the water will be assumed to follow a linear relationship with the fill depth:

$$\begin{aligned} W_{f1} &= M * H_{f1} + B, \text{ and} \\ W_{f2} &= M * H_{f2} + B. \end{aligned}$$

Solving these equations for the constants M and B for each barrel will produce a relationship between the barrel fill depth and the fill volume. This technique for determining the fill volume should greatly reduce any uncertainties in the volume because possible changes in the cross-sectional area of the barrel as a function of height.

given that $W_{f1} = \rho_{water} * V_{f1}$, then

$$V(H) = \frac{M * H + B}{\rho_{water}}$$

Over the range of expected water temperatures (5 to 15 °C), the density of water (negligible uncertainty assumed) may be expressed as

$$\rho_{water} = 0.999897 + 4.74E-5 * T - 6.71E-6 * T^2$$

where T (°C) is the measured water temperature. The density of water as a function of temperature was obtained by performing a second degree polynomial fit to data given in Table 2-2 (Weast, 1968).

Table 2-2. The absolute density of water.

Temperature (°C)	Density (g/cm ³)
5	0.999965
7	0.999941
9	0.999781
11	0.999605
13	0.999498
15	0.999099

The uncertainty in the constants M and B are given by

$$\sigma_M = \frac{W_{f2} - W_{f1}}{H_{f2} - H_{f1}} \cdot \sqrt{\frac{\sigma_{W_{f2}}^2 + \sigma_{W_{f1}}^2}{(W_{f2} - W_{f1})^2} + \frac{\sigma_{H_{f2}}^2 + \sigma_{H_{f1}}^2}{(H_{f2} - H_{f1})^2}}$$

and

$$\sigma_B = \sqrt{\sigma_{W_{f1}}^2 + (M * H_{f1})^2 * \left(\left[\frac{\sigma_M}{M} \right]^2 + \left[\frac{\sigma_{H_{f1}}}{H_{f1}} \right]^2 \right)}$$

Empty the barrel of water and thoroughly dry it. Fill the dried barrel with mixture to a height about 10 percent higher than the desired compacted fill height. Vibrate the barrel to compact the mixture and increase its density. Once the barrel has been filled with moisture matrix the filled volume of the barrel and its associated uncertainty are given by the equations

$$V_{fill}(H) = \frac{M * H + B}{\rho_{water}}$$

$$\sigma_{V_{fill}} = \frac{\sqrt{(\sigma_M * H)^2 + (\sigma_H * M)^2 + \sigma_B^2}}{\rho_{water}}$$

Measure the weight of the mixture in the filled barrel W_{fill} . Measure the height of the air above the mixture H . The density of the mixture is given by

$$\rho_{mix} = \frac{W_{fill}}{V_{fill}}$$

where V_{fill} is the volume of the mixture calculated from H_{air} .

The uncertainty in this density is given as

$$\sigma_{\rho_{mix}} = \frac{W_{fill}}{V_{fill}} \cdot \sqrt{\left(\frac{\sigma_{W_{fill}}}{W_{fill}} \right)^2 + \left(\frac{\sigma_{V_{fill}}}{V_{fill}} \right)^2}$$

Once the height of the region from the top of the mixture to the top of the barrel is determined, the thin Aluminum top can be placed on top of the mixture and sealed into place using a silicone caulking. Care must be taken while applying the sealant so that the entire lid is sealed, however, it is desirable to minimize the amount of excess sealant applied.

2.4.4 SMMS Probe Placement Jig

An aluminum jig will be constructed that will be placed on each of the standards. At the center of this jig will be a ring that allows the SMMS probe to pass through it. This jig will both position the probe in the center of the barrel, and stabilize it so that the cables are away from the top of the mixture and not likely to overturn the probe. The probe will be able to fit through the central portion freely so that it can reach the aluminum lid over the mixture.

2.4.5 Confirmation of Material Dryness

The material to be mixed must be dry and contain negligible concentrations of unbound water. To determine if the sand, hydrated alumina, and boron carbide are dry, they will be tested by taking small (about 15 g) random samples and subjecting them to a thermogravimetric test. The thermogravimetric test will involve the use of a mass spectrometer (Netzsch, model STA409-QMS) that analyzes the vapors released from the sample during heating. The spectrometer allows any weight losses from the sample to be differentiated by the species mass, often providing identification of the element or compound released from the sample. Six samples will be taken of both the sand and the hydrated alumina (from different bags) and three samples will be acquired of the boron carbide. The sand and hydrated alumina samples will be taken from bags in the top, middle, and bottom of the shipping palette. If the samples show less than 0.1% change in weight from water loss after heating them to between 110 and 130 °C, then they will be considered dry and one can proceed directly to the process of mixing matrix constituents for standard preparation.

2.4.5.1 Drying the Material

If either the sand, hydrated alumina, or boron carbide contain water that is not chemically bound, then the material must be dried before the mixing process is started. The material to be dried can be placed in unsealed containers (up to 100 kg at a time) and heated in an oven to remove the moisture. The drying will occur at a temperature of between 130 and 160°C. Drying time duration will be sufficient to allow the bulk material to reach a minimum temperature of 105°C. Calibrated thermocouples will be used to verify oven and material temperatures during drying. One sample must be acquired from near the volumetric center of each batch for thermogravimetric testing to confirm that the material is dry. Once the material is dried it must be cooled, capped and stored indoors until the mixing process begins.

2.4.6 Mass of Materials

Once the materials are known to be dry, they can be mixed. Mixing the standards by volume would require a knowledge of the porosity of the sand and hydrated alumina. Since this parameter is not well known and can change, a more accurate method will be to mix the components by weight. Table 2-3 lists the relative weights of the components needed for each mix.

Table 2-3. Relative mass of each constituent for each matrix composition.

<u>Matrix Description</u>	<u>Sand</u>	<u>Hydrated Alumina</u>	<u>Boron Carbide</u>
4 wt% H ₂ O	1.000	0.1305	0.0
8 wt% H ₂ O	1.000	0.3003	0.0
13 wt% H ₂ O	1.000	0.6006	0.0
19 wt% H ₂ O	1.000	1.215	0.0
25 wt% H ₂ O	1.000	2.592	0.0
35 wt% H ₂ O	0.0	1.000	0.0
19 wt% H ₂ O, 4E-3 wt% B	1.000	1.215	0.00005111
19 wt% H ₂ O, 8E-3 wt% B	1.000	1.215	0.0001022

2.4.7 Mixing of Materials

The materials will be mixed in a steel drum cement mixer filled to about half capacity. The mixer will be cleaned and dried before mixing of a given moisture content mixture. The boron containing matrices will be mixed last. Each of the materials will be weighed and poured into the mixer. The mixer will be run for about 5 minutes or until the color of the mixture is uniform. The mixed contents will be pored into the proper barrel. Two samples (about 20 g each minimum) of each pour will be taken and weighed for later analysis. The sample containers will be marked with the moisture standard identification number, the pour number, the expected moisture concentration, and the letter A or B to differentiate the two samples (e.g. MS4,P2,19%,A). The mixer need not be cleaned during the mixing of a given moisture content mixture, but must be cleaned and dried between batches. The process of mixing and pouring will be video taped. The room temperature and relative humidity will be recorded on the day of mixing.

This process will continue until the compacted barrel is filled to the desired height. It is anticipated that each completely filled uniform moisture standard, for example, will require two batches of matrix to be filled. Detailed records of all of the pours will be recorded for each barrel. The errors in the process will be calculated.

2.4.8 Sampling of Materials and tests to be performed

Samples of each pour will be taken using a dry sampling tool and placed in plastic containers which have close fitting lids. These samples must be capped, labeled and logged into the notebook as to the pour number. Only one sample from each pour will initially be subjected to analyses. The second sample will be retained for possible additional analyses to be performed if the results from the first sample vary significantly from expected values.

These samples will be subjected to elemental analysis. The concentration of the elements, listed below, will be measured. All elements,

except chlorine, will be measured using an inductively coupled plasma - atomic emission (ICP-AE) technique. Chlorine will be measured by using an ion chromatography technique. The purpose of these analyses is both to confirm that the matrix has the correct proportions of constituents and to determine the quantities of any trace significant thermal neutron absorbers that may be present in the mix constituents.

·	Ag	·	Li
·	Al	·	Mn
·	B	·	Nd
·	Cd	·	Ni
·	Cl	·	Sb
·	Co	·	Se
·	Cu	·	Si
·	Fe	·	Sm

Thermogravimetric analysis will be performed upon each sample to determine its total (bound and unbound) water concentration. These measured water concentrations will be used as the definitive moisture concentrations of a given moisture matrix.

2.4.9 Pouring of Materials into Barrels

The mixed material will be slowly poured into the barrel directly from the elevated cement mixer. The operator doing the pouring will have a face mask to prevent ingestion of any dust. The operator will ensure that, in general, all of the material mixed in a given batch is poured into the standard.

2.4.10 Compaction of Material in Barrels

After all material has been transferred into a barrel, the material will be compacted. Moisture standards, similar to those planned, have been constructed for other purposes (Engelman et al. 1995). Engelman used pneumatic vibrators to compact his standards until a desired density was achieved. The mixtures of hydrated alumina and sand decreased in volume about 15% after the initial pour. Although there is not an exact target bulk density for our standards, uniform density (in the range between about 1.5 and 1.8 g/cm³) is desired. Compaction will reduce the likelihood of material settling in the standards and will also reduce the need for and the effect of the external shielding. Vibrators will be attached to the barrel and activated. The duration, frequency, and magnitude of the vibration will need to be determined from tests. The vibration must be sufficient to compact the matrix to the desired density but must not cause significant segregation of the matrix constituents. Additional material will need to be added to the standard and vibration repeated if compaction causes the height of material in the standard to drop below 36 cm.

2.4.11 Leveling of the top of the Standard

Once the standard has been compacted, the top must be leveled. This will be done using a large round-edged trowel or straight edge. The tool will be used to carefully level the surface while disturbing as little material as possible.

2.4.12 Sealing of Standards

After the compaction of the standard, the top needs to be sealed. The aluminum lid will be placed on the top of the compacted material. A level will be used to ensure that the lid is level and is properly seated on top of the mixture. The silicon sealant will then be applied to the entire joint between the lid and the barrel.

2.4.13 Determination of Standard Bulk Density

The bulk density of the verification standard will be the ratio of the measured weight to the measured volume. A more complete discussion of the measurement of these two quantities is given in sections 2.4.3.

2.4.14 Calibration Data Collection and Logbook Records

The data collected will consist of several items. The first of these will be the data on the making of the samples, including the following:

- volumes of the containers and mixtures,
- weights of the containers and lids,
- results of the tests made on the samples to determine if the sand, hydrated alumina, or boron carbide require drying,
- weights of all mixtures and their constituents,
- weights of all samples taken from the mixtures,
- data on the compaction time interval,
- results of the sample thermogravimetric analyses.
- results of the sample elemental analyses.

These data, for each barrel, will be recorded in a logbook. The accuracy associated for each measurement will be calculated and recorded. The final determination of the moisture concentration and its associated uncertainty will be recorded in the logbook. Each standard (barrel) will be labeled with its moisture concentration(s).

2.4.15 Core Samples of a Test Standard

A test standard containing uniform 19 wt% moisture will be prepared and destructively tested by taking three core samples. These core samples will be analyzed to demonstrate that the mixing, poring, and compaction result in a uniform distribution of materials (i.e. moisture) throughout the mixture. The core samples will be obtained by driving three open ended pipes into the matrix. The matrix can then be removed from around each pipe and the bottom end of the pipe sealed for removal. The cores will then be extruded so that a sample can be obtain from the bottom fourth, middle bottom fourth, middle top fourth, and top fourth of the matrix. After the coring process is complete, the barrel can be emptied, cleaned, and reused.

If the analysis of the four samples from each core indicates that the process does not produce a uniform hydrogen concentration, there are two possible reasons. It might indicate that the mixing is not complete, or that the two components have shifted positions during vibration. If the changes are monotonic it would indicate that a settling of the components is occurring which might be remedied by reducing the amount of compaction. If the results are randomly scattered about some mean value it would indicate that more mixing is required. If the results are scattered around a sloping curve it would indicate that both processes may be occurring. In any of these cases the process will be corrected and a second standard constructed. Test standards will be constructed and destructively tested until uniformity of the constituents is verified.

This process will continue until the results are uniform to within ± 1 wt%. All of the remaining standards will be made based on the procedure developed from these tests.

3.0 SURFACE MOISTURE MEASUREMENT SYSTEM VERIFICATION

3.1 DESCRIPTION OF PROBE AND DATA ACQUISITION SYSTEM

The probe consists of a nearly cylindrical steel housing (8.9 cm OD) containing three ^{10}B -lined neutron detectors, neutron shielding, two solid state temperature sensors (SSTS), and associated signal amplification/processing electronics. The analog signals from each detector are sent via coaxial cable to a data acquisition system in the SMMS van. This system consists of a PC-based computer with user interface, a NIM bin containing signal amplifiers and a quad-input multichannel analyzer, and voltage sensors for reading the probe SSTS outputs.

3.2 EXPERIMENTAL PROCEDURE

3.2.1 Safe Operation and Handling

The primary safety concern during the testing with the SMMS probe is radiological hazards associated with the neutron source. Complete radiological safety procedures for the handling and storage of the neutron source may be found in the *Safe Operating and Emergency Procedure for the In-Tank Neutron Moisture Measurement Probe* (Watson 1995). A brief description of some of the more important aspects of radiological safety is included below.

3.2.1.1 Radioactive Source Storage

When measurements are not being made, the Cf-252 radioactive source will be stored in a lockable cask in a Health Physics designated radioactive material storage area in the Hanford Site 306E building. Storage and work with the source in this location will be governed by a radiation work permit. Calibrated dosimetry measurement devices will be needed to establish boundaries for radioactive material and radiation areas.

3.2.1.2 Insertion and Removal of the Source From the Probe

The source is stored in a lockable cask. The cask needs to be opened and the source unscrewed for the source to be moved. The source handling tool (95 cm long) is to be used for all source manipulations. Once the tool is used to remove the source from storage, the source is moved to the probe and inserted. The source is screwed into the probe housing using the tool.

3.2.1.3 Probe Handling Procedures

The probe will be handled by the support/signal cable or by the end of the housing opposite the source when the source is installed.

3.2.1.4 Radiation Work Permit

A radiation work permit (RWP) is required for both the storage and use of the neutron source. The RWP will specify required training and dosimetry for all individuals entering the radiation area set up during source use. RWP number R-135 is being established for this work.

3.2.1.5 Control of Radiation Area During Measurements

The operator is required to maintain surveillance of the radiation area while the source is out of storage. Unauthorized personnel are restricted from entering the radiation area and authorized personnel not needed to assist test set up should remain outside the radiation area.

3.2.2 Setup and Turn On

The SMMS probe must be connected, via the signal/support cable, to the SMMS van data acquisition system. The probehead is semi-permanently attached to the signal cable. The signal cable will be attached to an electronics box containing the probe related circuitry (intrinsic safety, demultiplexor, etc.) that, in the field, is located in the deployment masthead. A separate signal cable connects this electronics box to the van data acquisition system. The data acquisition system is used to obtain all detector signal and SSTS signal information from the SMMS probe. The system also provides power to the probe, through a system of intrinsically safe electrical barriers. The data acquisition system power must be established and the system data acquisition software must be executed. The neutron source must be loaded into the probe. The probe may require a few minutes of time with power established before the detectors and electronics are operating in a stable manner. No measurements should begin for a minimum of two minutes after initial total system power is on.

3.2.3 Temperature Measurements and Equilibrium

The temperature of the detectors and electronics may have an effect upon the electrical gain and or count rates produced by the probe in response to a given surrounding media. It is therefore important to know the temperature of the probe during the calibration tests so that field measurements may be adjusted, if needed, to correspond to measurements during the calibration tests. The temperature within the probe is measured by two solid state temperature sensors (SSTS) located within the probe housing. Before a measurement, readings provided by these SSTS must indicate that the probe has reached temperature equilibrium. Temperature equilibrium will be defined as all probe SSTSs measuring less than a 1°C change in temperature over a time interval of 5 minutes. The SSTSs in the probe must be calibrated prior to initial use in the probe.

The SSTSs selected for use have an output of 10 mV per degree Celsius. The temperature sensors will be characterized in a temperature bath or similar apparatus at four points over the temperature range of -20 to 100 °C. The apparatus will have a temperature calibration that is traceable to National Institute of Standards and Technology (NIST) temperature standards. The four temperature calibration points will be selected to provide greatest accuracy at temperatures above 30 °C. At the time of calibration, each SSTS will be

serialized to provide traceability to its four temperature calibration points. At the four temperature points the output voltage of the temperature sensor will be measured. A calibration curve will be prepared for each SSTS. With the calibration curve, the electronics in the probe will be calibrated to a best-fit straight line curve using voltage standards whose calibration is traceable to NIST voltages standards. If the accuracy of the best-fit straight line curve does not provide sufficient accuracy (about 5 to 10%), then a temperature conversion table or a curve fitting algorithm will be implemented in the software.

3.2.4 Use of In-Field Calibration Check-Standards

The calibration check-standards will consist of 5 wt% borated polyethylene cylinders (25.4 cm diameter, 34.3 cm long). Both will have a carbon steel lined cylindrical hole (about 22 cm deep) along its axis. The steel lining will be about 0.3 cm thick for one and about 1.3 cm thick for the other. These standards are intended to provide an in-field indication of whether or not the probe is functioning correctly and is returning the same responses, adjusted for temperature and source decay, obtained during the calibration.

Before and after each day of calibration tests measurements will be made in these check-standards. The probe will be inserted fully into each of these standards and the total counts will be recorded from each detector over a 3 minute data collection interval. The count rate for each probe will be recorded.

3.2.5 Probe Placement On Standard

The probe will be placed on the top of a standard in the placement jig discussed in 2.4.4. This jig will position the probe in the center of the of the barrel, keep it vertical and stable, and keep the cables from interfering with the measurements.

3.2.6 Calibration Data Collection

Detector response data will be collected on each standard. Collection time will be a minimum of five minutes on each standard. Test personnel will determine if additional data collection time is required for each standards based upon detector count rates. A collection time will be chosen that allows each detector to accumulate a minimum of 10,000 counts when placed upon a standard. A logbook will be maintained that provides a record of measured detector responses for each standard. This information will include the data collection time interval, the total number of counts (above discrimination) recorded by each detector, and the temperature of each SSTS. The raw data collected by the data acquisition system multichannel analyzer will be stored

electronically in files on the system disk. The settings of all adjustable electrical components, such as amplifier gain, will be recorded. Each detector high voltage setting will be measured and recorded prior to assembly of the sensor package into the probe housing.

In addition to acquiring probe responses with the probe placed on the standards, count rates will be measured with the probe isolated. The probe will be suspended in air at least 2 meters above the floor and 3 meters from any walls. Other than the probe support cable, no hydrogenous materials will be located within 2 meters of the probe. With the probe essentially isolated from outside materials that could provide signal to the probe, a measurement of the detector responses will be made for a minimum of 15 minutes.

3.2.7 Data Storage and Archival

Data storage will initially be in a Data Logbook for most data. Data collected electronically using the SMMS data acquisition system will be collected on the system hard drive and then transferred by removable media (e.g. floppy disk) to a computer with access to the WHC common file storage (CFS) for archival.

3.2.8 Data Comparison with Calculations

The primary purpose of the measurements is to verify the computer model predictions, which are a function of the model inputs. The primary model input parameters are the geometry and material properties of the probe and the standards. Physical dimensions, within mechanical tolerances, may need to be adjusted in each part of the geometry to obtain best agreement with measured results. Many other inputs, such as material compositions and densities, should be well known but may be adjusted within known uncertainties. Exact neutron source strength and placement may need to be adjusted in the model. Modeling of neutron interactions in the ^{10}B detector lining may need to be adjusted to account for physical detector characteristics, such as efficiency.

Model calculations will be performed that provide response predictions with a stochastic precision of 2.5% or better. The probe model will be adjusted in an attempt to obtain agreement between all measured detector responses and calculated responses of 5% or better.

4.0 TEMPERATURE AND GAMMA-RAY EXPOSURE RATE TESTS

The operational sensitivity of each of the detectors at high temperatures and in high gamma-ray fluxes need to be measured separately and simultaneously. One needs to determine the influence on the electronic gain of the signal and how it will affect the count rate above a given electronic

threshold. An ideal neutron detector should show little or no change in the count rate as the temperature and gamma-ray flux are increased to the expected limits. The experiments described here will demonstrate the separate and combined effect of these parameters and should provide information that will enable corrections to be applied to in-tank data based upon temperature data.

4.1 EXPECTED CONDITIONS FOR THE SMMS

The SMMS array of detectors is expected to operate in a field of up to 2 grey/h and temperatures of up to 79°C. The tests described here will first measure the response of the detectors, and electronics in a various gamma-ray fields, then measure the response of the detectors and electronics in a temperature field. The final set of measurements will measure the response in combined temperature and radiation fields.

The SMMS masthead contains both intrinsic safety and demultiplexing electronics for the probe. These electronics, enclosed in a metal box, will be subjected to the outdoor temperature extremes of the Hanford Site. Typical Hanford Site temperatures range from about 0 to 35 °C for most of the year.

4.2 DETECTOR GAMMA-RAY EXPOSURE RATE TESTS

The gamma-ray fields are produced by NIST traceable calibrated gamma-ray sources. Different fields can be produced by changing the distance of the sources from the detector. First, connect a multichannel analyzer (MCA) to the analog output of the detector amplifier electronics. Place a Cf-252 neutron source (any available source) next to the detectors and place the detectors in position for the gamma-ray field measurements. The detector axes, placed perpendicular to the irradiation beam, will be positioned at the proper distance from the source for the desired exposure rate. Measure the detector responses before the gamma-ray sources are exposed. Expose the gamma-ray sources and remeasure the detector responses. Record the pulse height spectra for both measurements for each detector onto disk. Re-shield the gamma-ray sources and reposition the detectors for the next field intensity. Repeat the procedure of measuring a "background" spectrum and a spectrum with the gamma-ray sources exposed. Repeat the process for all of the gamma-ray source to detector spacings (all exposure rates). A test will be performed with the detectors subjected to a 2 grey/h gamma-ray field.

Data will be reported as detector count rate, above a given threshold, as a function of exposure rate. Also a graph showing the MCA spectrum for several exposure rates will provide qualitative information.

4.3 DETECTOR TEMPERATURE DEPENDANCE TESTS

The first set of temperature measurements will be carried out in an oven. The final set of combined temperature and gamma-ray measurements will be carried out with a heater, thermocouple and the gamma-ray sources discussed above.

Connect the electronics so the analog output of the detector is connected to the MCA. Place a Cf-252 source near the detector and close the oven door. Record a pulse height spectrum for each of the detectors at ambient temperature. Record the ambient temperature.

Increase the oven temperature by about 10°C and let the detector come to thermal equilibrium. Record pulse height spectra for the three detectors. Repeat this last procedure until a temperature of 80°C is reached.

4.4 COMBINED DETECTOR GAMMA-RAY AND TEMPERATURE TESTS

A thermal blanket is to be wrapped around the each detector in the gamma-ray measurement configuration. Before the sources are exposed in each position, a background neutron spectrum (no gamma-ray field) must be measured.

The temperature will then be increased to 80°C and the gamma-ray source exposed. Pulse height spectra will be recorded for each detector. The unit will then be moved to the second source position. The measurements will be repeated at 80°C. The unit will be allowed to cool down and a final measurement made with the gamma-ray sources not exposed. Only two temperatures (ambient and 80°C) will be measured in the combined tests. Only two positions for different gamma-ray exposures will be measured. Spectra will be obtained for all three detectors at each position.

4.5 TEMPERATURE DEPENDANCE TEST OF THE INTEGRATED SMMS PROBE

In order to assess the total temperature dependance of the probe's response to changes in temperature, the complete SMMS probe will be tested in temperatures ranging from -10°C to 80°C. The results from these integrated temperature tests will be used to apply any needed corrections to the probe response from in-tank data.

These tests will be performed within an environment chamber located at the 305 building, Hanford Site 300 Area. The SMMS probe will be placed in the chamber within a polyethylene cylinder. A Cf-252 neutron source will be placed within the cylinder. The position of the source is not critical since the source is only used to supply a steady but uncalibrated neutron signal for the detectors. Two calibrated solid state temperature sensors within the environment chamber to monitor the ambient temperature. The temperature of the chamber will be stepped in increments of approximately 10°C from -10°C to

80°C. At each temperature step, the temperature of the chamber will be maintained until the SSTS within the SMMS both indicate a temperature within 1°C of the chamber temperature. The SMMS SSTS must have current calibration. Once the probe has reached temperature equilibrium with the chamber, analog spectral count rate data will be obtained from each detector using the SMMS van data acquisition system for a time interval of 15 minutes. Data will be collected for each temperature step and the actual temperature measured by the SMMS SSTS will be recorded.

4.6 COMBINED SMMS PROBE GAMMA-RAY AND TEMPERATURE TEST

A thermal blanket is to be wrapped around the probe in the gamma-ray measurement configuration. Before the sources are exposed, a background neutron spectra must be measured for each detector. The temperature will then be increased to about 80°C and the gamma-ray source exposed. A gamma-ray field of 2.0 grey/h will be produced at the location of the center of the SMMS probe by positioning the probe a calibrated distance from the source. A gamma-ray dose rate meter will be used to measure the field at this location both with and without the thermal blanket to ensure that the blanket is not providing significant attenuation (>2%) of the photon field. Pulse height spectra will be recorded for each detector. The unit will be allowed to cool down and a final measurement made with the gamma-ray sources not exposed. Only two temperatures (ambient and maximum) will be measured in the combined tests. This test is intended to demonstrate that the completed probe is capable of functioning in the most extreme ambient conditions specified in the design requirements document.

4.7 TEMPERATURE DEPENDANCE TEST OF THE SMMS MASTHEAD ELECTRONICS

In order to assess the dependance of the masthead electronics, an assembly of the electronics used for probe signal processing will be tested in temperatures ranging from -5 °C to 45 °C. The results from these integrated temperature tests will be used to determine if the signals from these electronics require corrections as a function of temperature.

These tests will be performed within an environmental chamber located at the 305 building, Hanford Site 300 Area. Two calibrated solid state temperature sensors will be placed within the environmental chamber to monitor the ambient temperature. The temperature of the chamber will be stepped in increments of approximately 10°C from -5°C to 45°C. At each temperature step, the temperature of the chamber will be maintained for a minimum of 30 minutes prior to measurements being made to assure temperature equilibrium. Test pulses of the approximate size and shape of those expected from the probe detectors will be sent to the masthead electronics using multiple pulsers. The amplitudes of the input pulses will be measured and the amplitudes of the output pulses will be measured for each detector channel using the data acquisition system MCA.

5.0 REFERENCES

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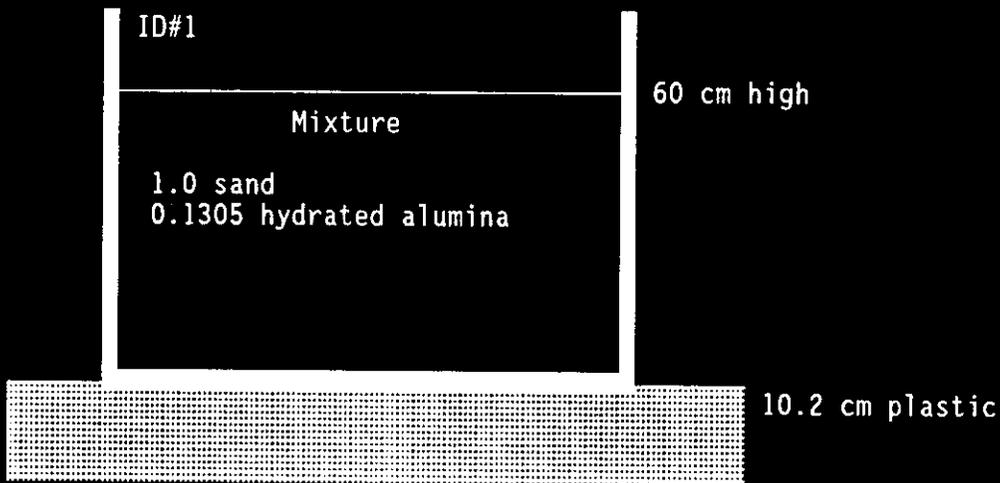


Figure 1. Uniform 4 wt% Standard

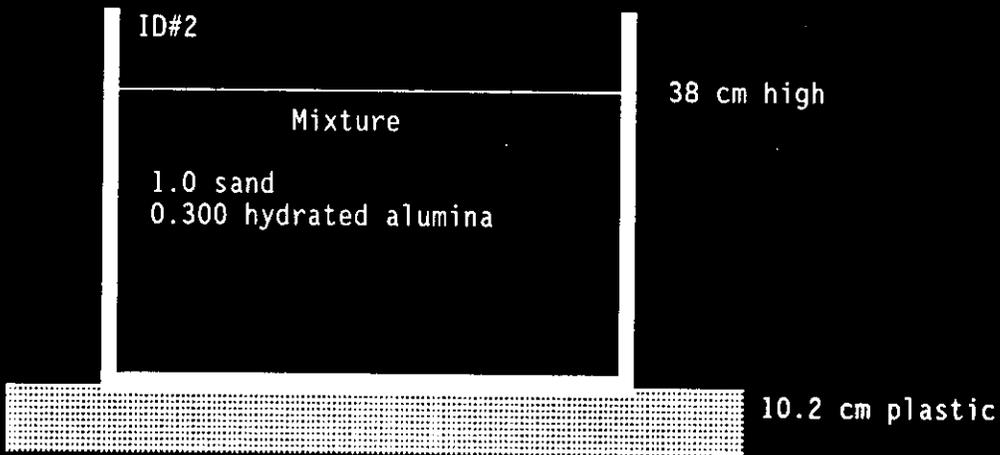


Figure 2. Uniform 8 wt% Standard

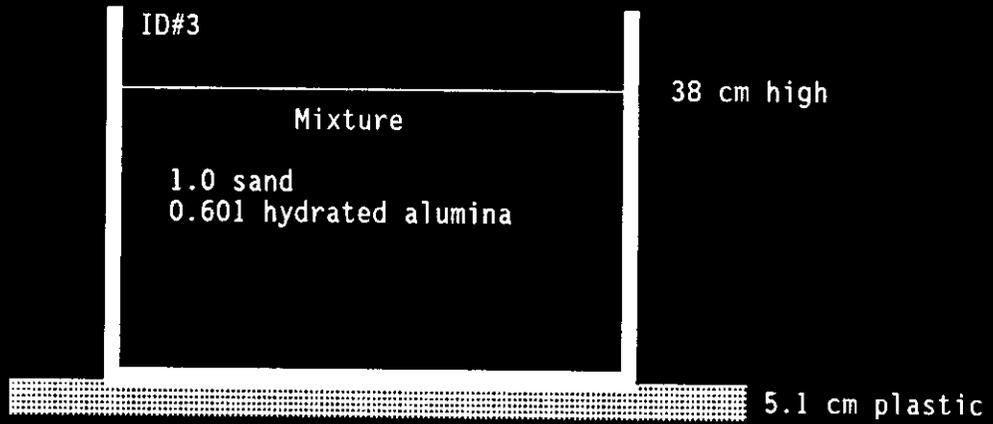


Figure 3. Uniform 13 wt% Standard

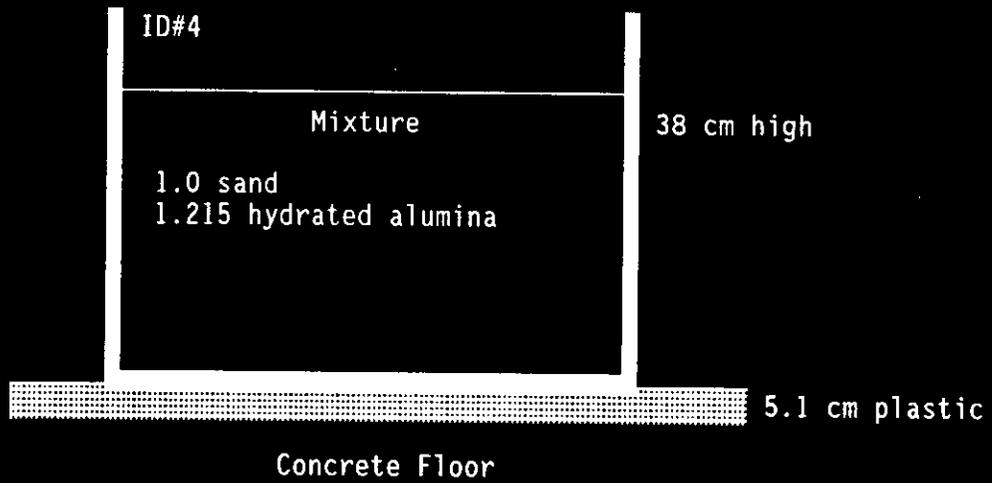


Figure 4. Uniform 19 wt% Standard

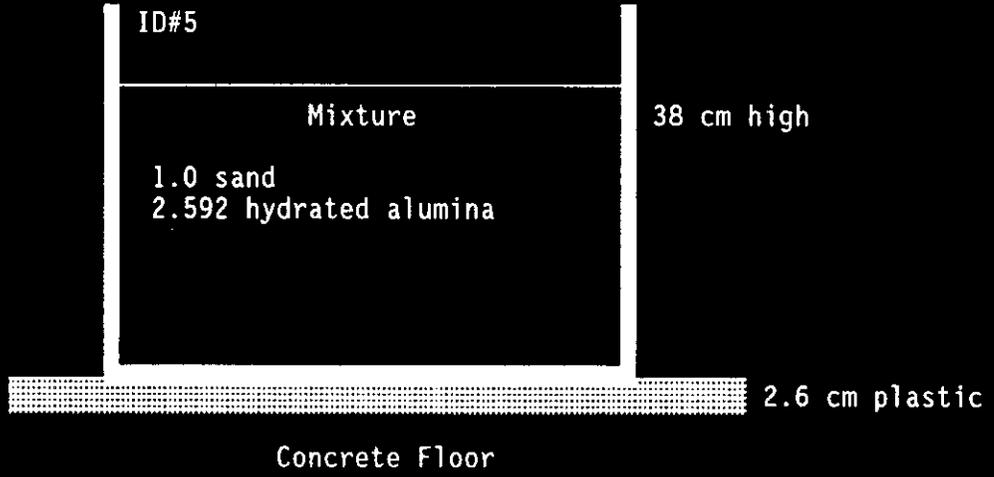


Figure 5. Uniform 25 wt% Standard



Figure 6. Uniform 35 wt% Standard

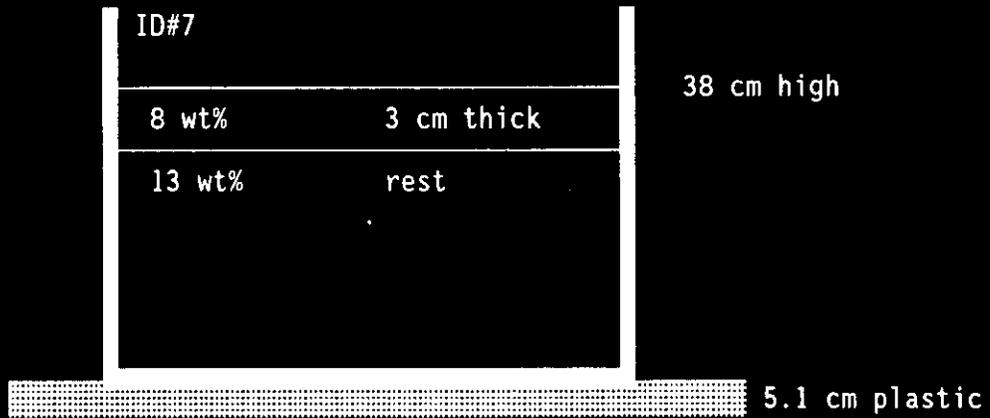


Figure 7. Layered 8 and 13 wt% Standard

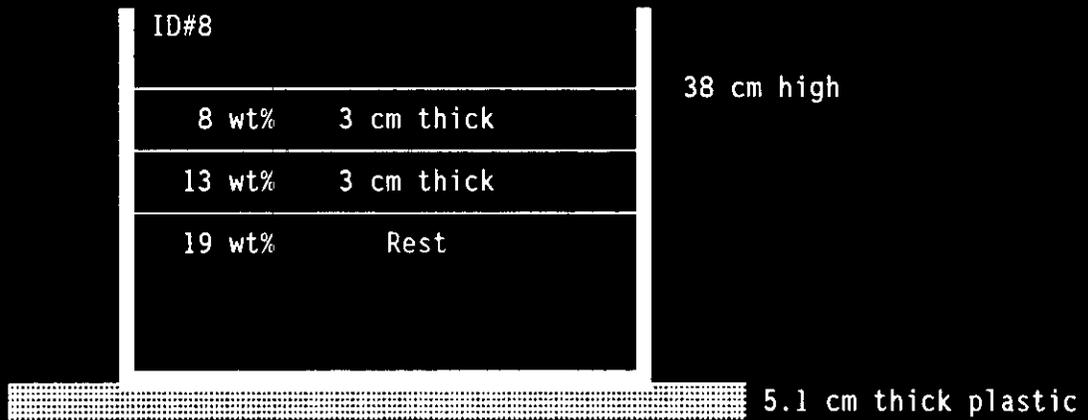


Figure 8. Layered 8, 13, and 19 wt% Standard



Figure 9. Layered 13 and 19 wt% Standard



Figure 10. Layered 13, 19 and 25 wt% Standard

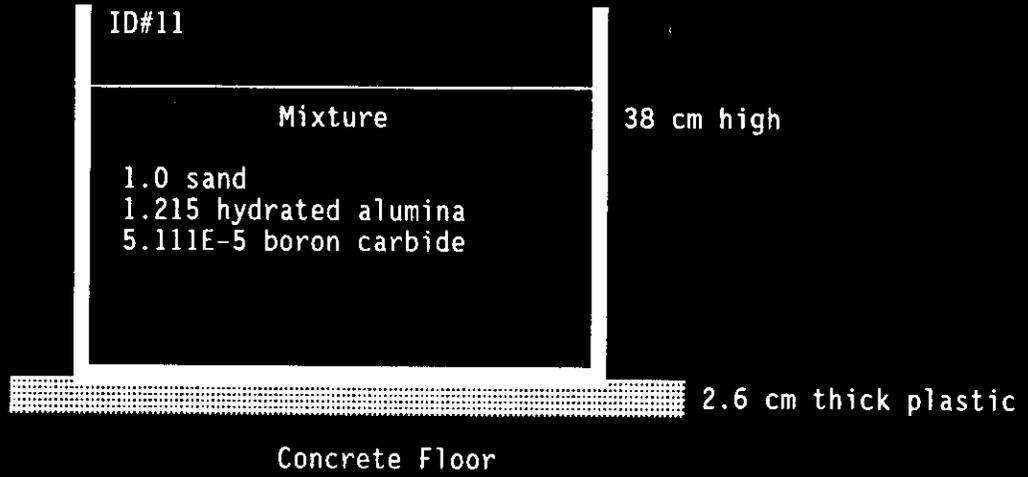


Figure 11. Uniform 19 wt% Standard with Low Boron

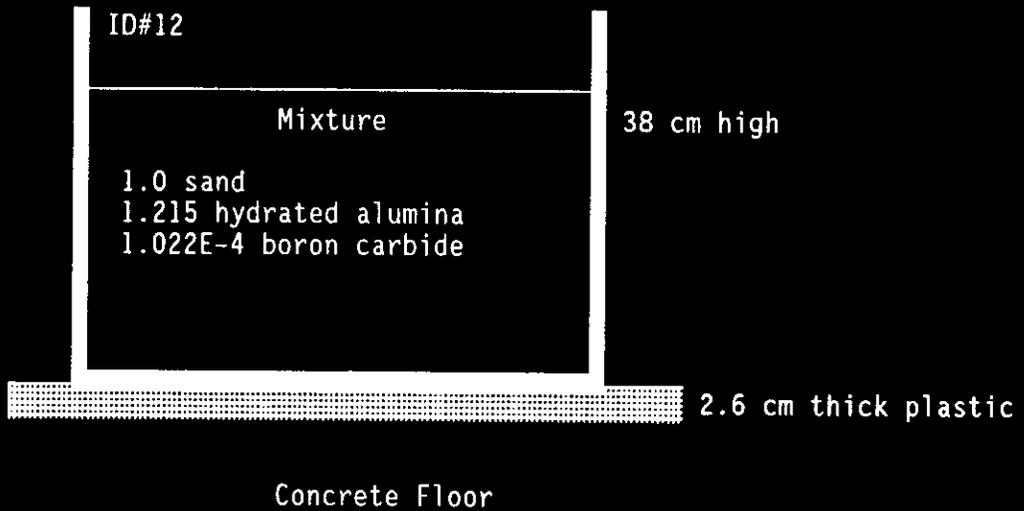


Figure 12. Uniform 19 wt% Standard with High Boron



Figure 13. Thin 19 wt% Standard (4 cm Thick)

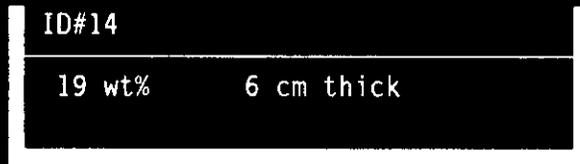
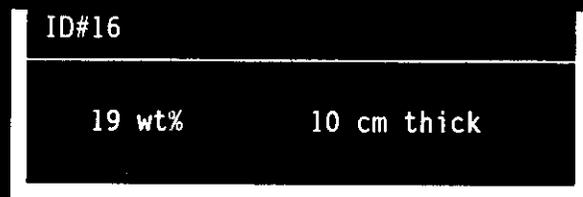


Figure 14. Thin 19 wt% Standard (6 cm Thick)



Figure 15. Thin 19 wt% Standard (8 cm Thick)



1.22 meter Stand

Figure 16. Thin 19 wt% Standard (10 cm Thick)

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