A CALIBRATION PROCEDURE FOR NUCLEAR DEPTH PROBES WITH OKLAHOMA SOILS

By

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CHAPTER I

INTRODUCTION

"Highway pavement is only as good as its subgrade" is a statement often cited by civil engineers when discussing highway design and construction. This old cliche is quite true. Subgrade conditions are most important, and any factor which may alter the engineering properties of this foundation soil is of critical concern. Moisture variations can greatly weaken the subgrade by causing changes in soil volume and strength.

The School of Civil Engineering at Oklahoma State University, in cooperation with the Oklahoma Department of Highways and Bureau of Public Roads, initiated, in June, 1964, a six year study of subgrade moisture variations under highway pavements (Ref 1). Fifty research sites were selected and prepared for data collection with nuclear depth density and moisture probes (Ref 2). This equipment enables repeatable in-situ soil density and moisture content measurements.

Statement of the Problem

The utilization of depth density and depth moisture probes for non-destructive soil testing (density and moisture content determinations) has been investigated since 1950. A primary obstacle hindering their acceptance as a standard test procedure is calibration of the equipment. Calibration of an instrument of this type usually refers to the development of an empirical, graphical relationship between apparatus response and soil conditions. Reliability of field measurements using nuclear depth probes is dependent on calibration accuracy.

Much of the difficulty can be traced to the character of soil itself. Soil type may vary widely in a given area. The physical, chemical, and mineralogical characteristics of soil are subject to variation. Field measurements with nuclear depth equipment must be valid for the entire spectrum of subsurface conditions. The feasibility of employing nuclear probes for quality control and inspection is dependent upon their reliability under all field conditions.

Therefore, a calibration procedure using actual soil "standards" would be desirable for nuclear probes. A standard is defined as a mass of soil which has been compacted to a known density at a known moisture content under controlled conditions. Construction of this standard requires an efficient method of soil preparation and placement.

Scope of the Investigation

A method of calibration standard construction using three Oklahoma soils as calibration media is described herein. Thirteen standards were constructed with various densities and moisture contents. The equipment response of nuclear depth density and depth moisture probes is evaluated in terms of empirical calibration curves obtained from the soil standards. The effects of soil type, standard container geometry, and calibration procedure on equipment response is also investigated.

CHAPTER II

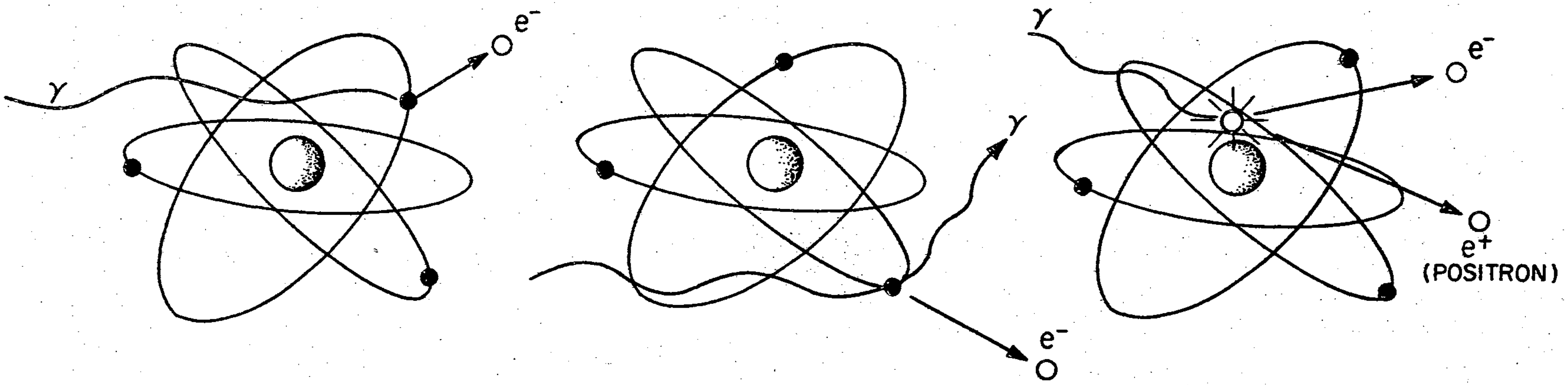
THEORY OF NUCLEAR MEASUREMENT

The basic operational theory behind nuclear testing procedures is of considerable importance to the calibration problem. The processes involved are, in general, quite complicated because of the radiation phenomenon, but the more important points will be summarized below.

Density Measurements

The density measurement technique utilizes a probe with a radioactive source, usually radium-226, which emits gamma radiation at a constant average rate. Gamma particles are capable of penetrating dense materials but are slowed with an accompanying energy loss as they pass through matter. Some gamma particles may be scattered through the material in a series of collisions, but others may be absorbed.

Gamma particles have three distinct reactions as they strike other atoms. From Fig 2.1, these reactions are the photoelectric effect, the Compton effect, and pair production. If a gamma particle collides with an orbital electron of another atom, it may transfer all of its energy to the electron. The "excited" electron is ejected from the parent atom with less energy than the original gamma ray. Materials have different rates of particle absorption by the photoelectric effect. The probability of this phenomenon occurring in a given material is dependent on the density of the material, atomic number and mass of the



(a) PHOTOELECTRIC EFFECT (b) COMPTON EFFECT (c) PAIR PRODUCTION

Figure 2.1. Gamma Particle Interaction (After Ref 4)

element, the percentage composition, and the total number of elements involved.

The Compton effect occurs when gamma particles collide with an orbital electron and are scattered with energy loss in the collision. The gamma particle continues in an altered direction with a lower kinetic energy. Usually, the electron struck is placed in an "excited" state and is ejected from the parent atom as with the photoelectric effect.

The third case is not significant in the mechanics of depth density measurements. Pair production occurs near the nucleus of an atom with a positron and electron being produced.

Photoelectric effects are predominant in soil materials when gamma particles have energies less than one Mev (million electron volts). One electron volt is the energy gained by an electron in falling through a potential difference of one volt. The Compton effect occurs as the principle mode of gamma particle interaction in the range of energies between one-half Mev to four Mev.

Two methods of gamma particle detection are used in conjunction with density testing. The backscatter process counts the number of gamma particles which are deflected by the soil back toward the source. The direct transmission technique utilizes gamma particles which pass through the soil with very little deflection. The source is at one location and the detection tube at another. A depth density probe employs the backscatter technique.

The density probe, seen in Fig 2.2, has two principle components, a radium-226 source at the bottom and a Geiger-Mueller tube at the top. The G-M tube consists of a thin cylindrical shell (the cathode) and a

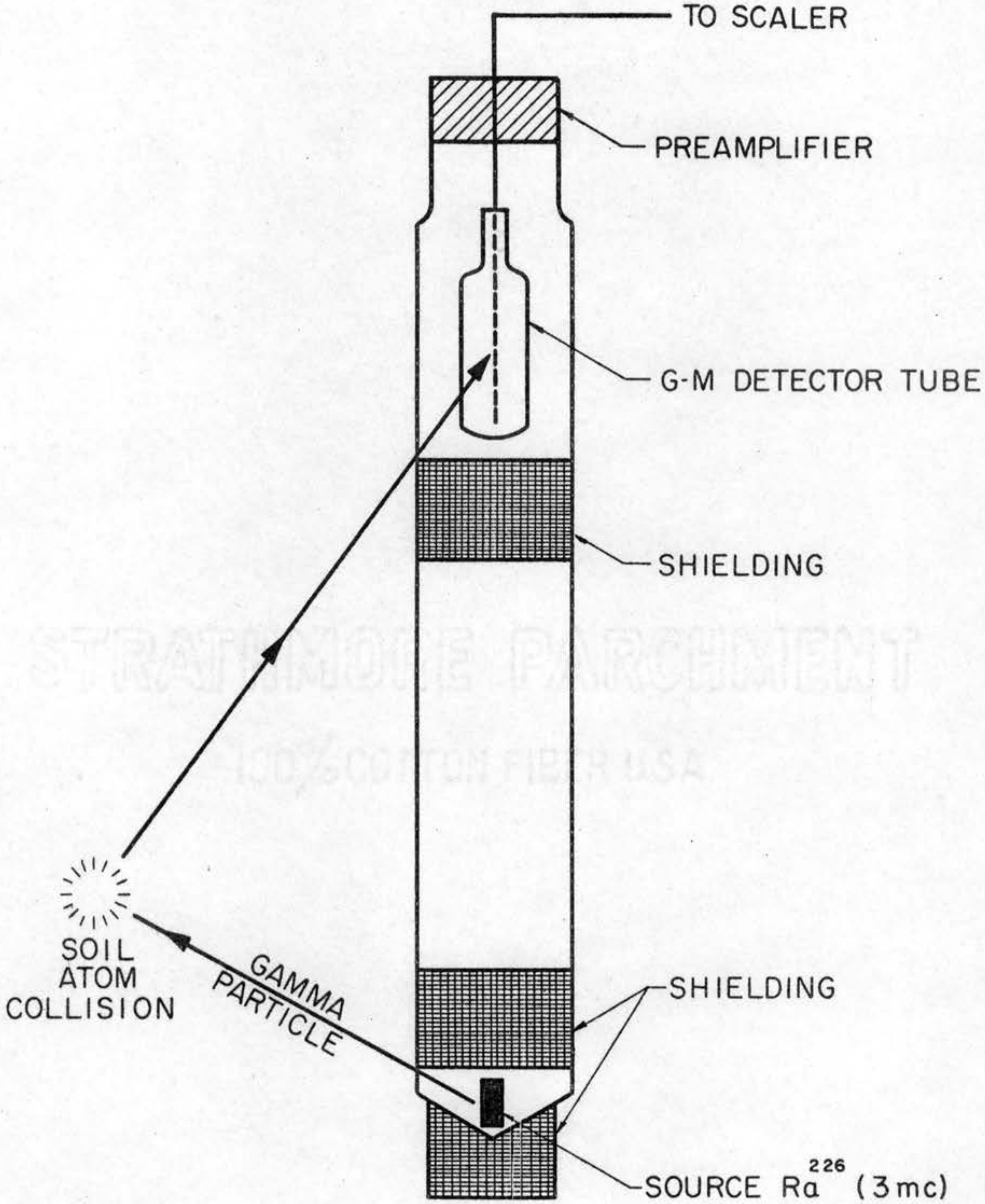


Figure 2.2. Depth Density Probe

fine wire anode suspended in an inert gas. A voltage of slightly less than that required to produce a discharge in the gas is applied between the anode and cathode. When a gamma particle enters the tube its energy ionizes a gas molecule. The electrons produced by ionization are accelerated toward the anode by the voltage gradient and cause additional ions to be produced. This is an extremely rapid reaction and produces an electrical discharge in the gas, resulting in an electrical impulse to the external circuit. The detection system counts all gamma particles regardless of their energy level. Shielding is placed between the detector and the source to prevent direct transmission. The distance between source and detector tube is most important because the probability of backscattered gamma radiation reaching the G-M tube increases with distance. The sensitivity of this instrument is quite poor, but it is believed that reliable results can be obtained when used with accurate calibration data (Ref 3).

As a soil increases in density, its ability to absorb gamma particles also increases. Fewer gamma particles will be backscattered to the detector tube. This means that a soil of 150 pcf wet density will have an instrument response or pulse count lower than a soil with a 120 pcf wet density. This relationship is valid for densities encountered in highway subgrades. The density measured is the total mass or wet density of the material.

Moisture Measurements

The nuclear method of determining moisture content employs fast neutrons. The moisture probe used in this study, shown in Fig 2.3, depends on a radium-beryllium reaction to produce fast neutrons. The

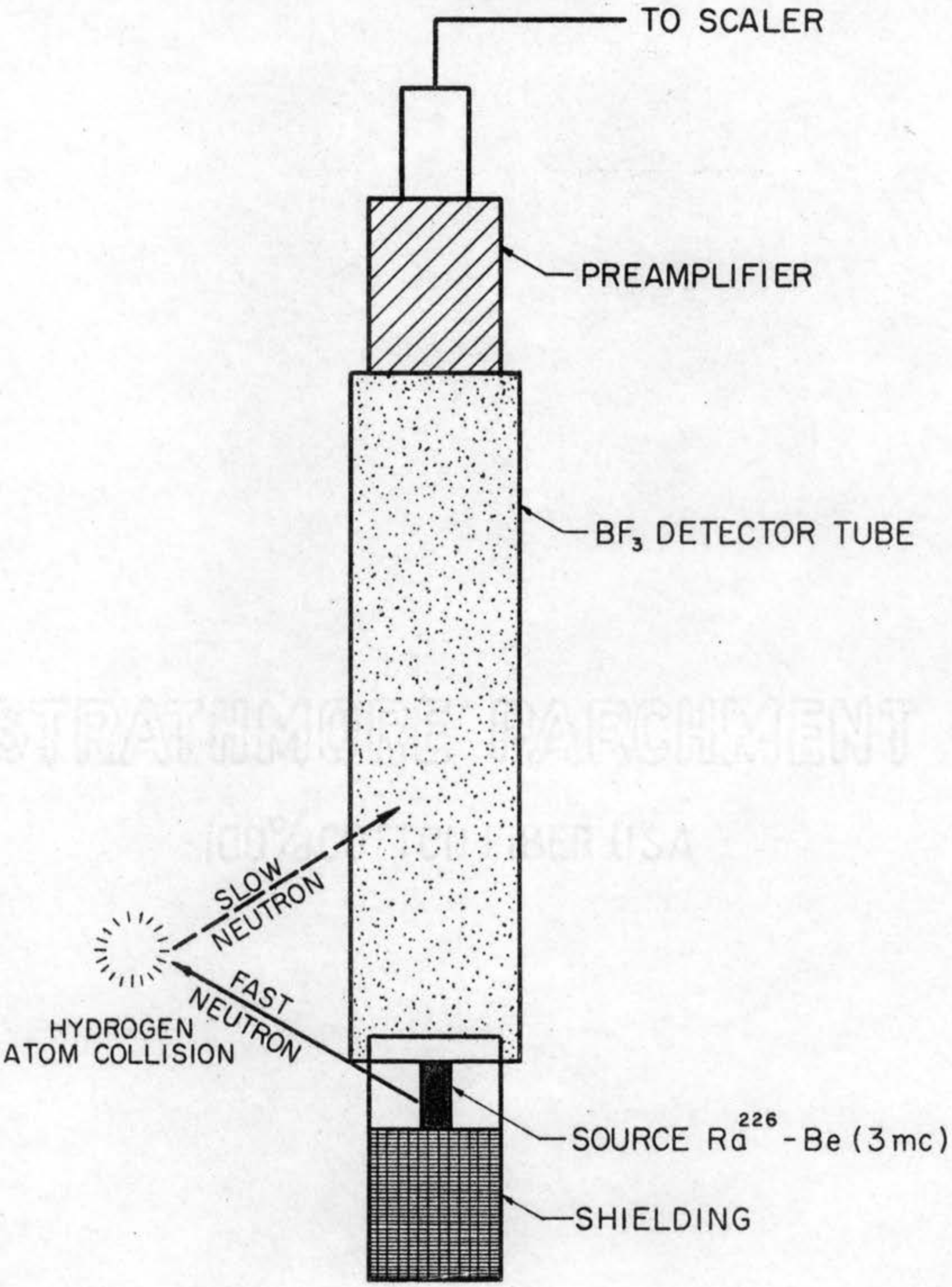


Figure 2.3. Depth Moisture Probe

radium-226 emits alpha particles which collide with the beryllium. This collision releases fast neutrons to be transmitted into the soil.

The neutron interaction with matter is quite similar to that discussed for gamma radiation. The neutron may engage in an elastic collision with other nuclei in the soil, or it may be absorbed by a nucleus. This results in the annilation of the neutron and, subsequently, an "excited" atom.

In any elastic collision the colliding particles must not lose their total kinetic energy. However, particle velocity may be altered. If an impinging neutron strikes a particle of the same mass in an elastic collision, the neutron could impart some or all of its energy to the struck particle. However, if the neutron were to strike an atom of much larger mass, it would merely be deflected with little loss in kinetic energy. The probability of an elastic collision is dependent on the size and mass of the atomic nuclei involved.

The only particle which has a mass of similar size as the neutron is the hydrogen ion. Therefore, hydrogen would have a greater probability of slowing the fast neutron. As can be seen in Table 2.1, it takes only about eighteen collisions with hydrogen atoms to slow or "thermalize" a fast neutron. Lithium is next, but it required approximately four times the number of collisions to thermalize a neutron. Furthermore, lithium is not found abundantly in most soils. Hydrogen exists as water and also in the ionic state. It can be said that the moisture probe is a hydrogen sensitive device, and by this means gives an indication of the amount of water in the soil.

The other important interaction mode is absorption. The ability of an atom's nucleus to capture a neutron is a function of the element

Element	Average number of collisions required for thermalization	Element	Average number of collisions required for thermalization
Hydrogen	18.2	Silicon	262
Lithium	69.3	Phosphorus	288
Beryllium	88.1	Sulfur	298
Boron	104.5	Chlorine	329
Carbon	115.4	Potassium	362
Nitrogen	133.5	Calcium	371
Oxygen	152	Titanium	442
Sodium	215	Manganese	514
Magnesium	227	Cadmium	1028

TABLE 2.1. RELATIVE EFFECTIVENESS OF ELEMENTS IN SLOWING DOWN FAST NEUTRONS

Some strong absorbers	Area Barns	Elements commonly encountered	Area Barns
Rare Earths	to 46,000	Iron	2.53
Cadmium	2,450	Potassium	2.07
Boron	755	Nitrogen	1.88
Indium	196	Sodium	0.505
Gold	98.8	Calcium	0.44
Lithium	71.0	Hydrogen	0.332
Silver	63.0	Aluminum	0.230
Chlorine	33.6	Magnesium	0.063
		Carbon	0.0034
		Sulfur	0.00052
		Oxygen	0.0002
		Phosphorus	0.0002
		Silicon	0.00016

TABLE 2.2. RELATIVE ABSORPTION CAPACITY OF SOME ELEMENTS FOR THERMAL NEUTRONS (0.025 ev)

and neutron energy. A list of strong absorbers or "poisons" is included in Table 2.2. The best absorbers have absorption cross-sections which are quite high. The unit of measurement for this property is the Barn; it is equal to an area of 10^{-24} cm^2. Although these neutron cross-sections have area units, they are not the physical cross-sections of the nuclei. In fact, nuclides have several cross-sections, and these variable cross-sections are often complicated functions of neutron energy (Ref 4).

Slow neutrons are counted by a tube filled with boron-trifloride gas enriched with boron-10. The slow neutrons combine with the nucleus of the boron-10 to form boron-11. This element is quite unstable, and it emits alpha particles when it disintegrates. These alpha particles ionize the gas in the detector tube to produce electrical pulses which are counted.

Certain of the neutron poisons (boron, cadmium, chlorine, and iron) will produce undesirable effects since the slow neutrons counted do not give a valid indication of soil hydrogen content. For instance, boron in concentrations of 2 to 100 ppm will give errors in equipment response up to ten per cent. A similar deviation will occur in the slope of the calibration curve with a chlorine content change of .0122 gram/gram of dry soil. An iron content change of five per cent will alter the slope of a moisture calibration curve by 3.3 per cent (Ref 5). Also, soils with high organic content (for example, peat) will give inaccurate results because of the presence of organic compounds. The hydrogen in these sources will thermalize neutrons just as efficiently as hydrogen in water.

CHAPTER III

REVIEW OF PREVIOUS CALIBRATION PROCEDURES

Nuclear equipment employed in this study was manufactured by Troxler Laboratories, Inc. of Raleigh, North Carolina. They rely on non-soil materials to produce factory calibration curves for nuclear depth density and moisture probes. Portland cement concrete was chosen as the density calibration media. Modified fifty-five gallon oil drums were filled with concrete, vibrated to uniform densities and moist cured. Aluminum access tubing was placed in the barrel prior to the pouring of the concrete. The surface of the cured material was coated with epoxy to prohibit moisture fluctuations (Ref 6).

The moisture equipment was calibrated with cadmium chloride-water solution standards. Cadmium is a very efficient absorber of fast neutrons; a high cadmium content will produce a low return of thermalized neutrons to the detector tube. The water acts as a thermalizer of fast neutrons. Various cadmium chloride-water solutions were correlated to soil response at a known moisture content; soils native to the North Carolina region were employed in this correlation (Ref 6).

Some early studies theorized that calibration curves between moisture content and counting rate would be independent of soil type. Belcher, Cuykendall and Sack first arrived at this conclusion (Refs 7, 8). The U.S. Army Corps of Engineers calibrated nuclear depth probes with laboratory standards (Ref 9). This calibration was checked in the field by comparing gravimetric moisture contents of soil samples obtained at test sites to the moisture content indicated by equipment response. It was observed that separate curves could be fitted through the data more closely than a single curve. However, the study chose to develop a single curve from the data obtained for density and moisture respectively. The Corps of Engineers concluded that this procedure had too much experimental error. They also state the density probe was not accurate enough for airfield measurements because of the calibration problem, but the moisture probe might be.

Later research by Belcher, Cuykendall, Sack, and Carlton (Ref 10) invalidated earlier work and recommended separate curves be developed for density gages. They found that composition or soil type effects were as much as nine per cent, indicating the need for separate calibration curves.

Holmes and Jenkinson (Ref 11) have stated that the character of the absorption cross-section of soil solids and the possibility of hydrogen existing within a soil in compounds other than water complicate the curve theory.

LeFevre and Manke (Ref 3) though acknowledging that separate curves for individual soils did exist, attempted to develop a median calibration curve for moisture and density gages. They state that for practical purposes the single curve approach is warranted so this method may be considered feasible for engineering use.

Richards (Ref 5) attempted density calibration using drums filled with uniform sand. Moisture studies utilized the same material with various amounts of water added. The sand was compacted in six inch lifts with a hand tamp. Pulse count in the various standards was correlated to moisture content as determined by samples taken from the standards. Cohesive soils were not employed due to problems of mixing and placement. The curve produced in the laboratory was compared to field data. Moisture readings were collected from several sites and in various soil types. The counts were used in conjunction with the laboratory curve to predict in-situ moisture content. Soil samples were removed from each soil test site, and gravimetric moisture determinations were completed as a check on the validity of the calibration curve. Richards concluded that this approach to the problem proved unsuccessful as unsatisfactory results were obtained.

Since preparing soil standards involved much time, labor, and large volumes of soil, Van Bavel, Nielson, and Davidson (Ref 12) chose neutron absorbers as calibration media. Their research concluded that neutron counts similar to those in soils could be obtained. Furthermore, they state that any field calibration will give results of indecisive validity.

McHenry (Ref 13) and Ballard and Gardner (Ref 14) recommend that standards must conform to certain basic criteria. They mention such factors as cheap construction, use of readily available materials, and use of materials which will not undergo extensive changes over a long period of time. The standard should provide a wide range of readings for the nuclear equipment. Other investigators have done work with paraffin, sand and ammonia alum mixtures, and polyethylene materials as calibration media (Ref 13). Ballard and Gardner (Ref 14) suggest possible standards of aqueous solutions of salts, slurries, non-compactible powders, solids, or sized solid particles. They also present some work with a mathematical analysis of the gamma scattering process and neutron interaction in a mass model.

LeFevre and Manke (Ref 3) used limestone aggregate, river gravel, expanded shale, and Permian red clay as soil standards. The aggregate, gravel, and shale were tested in dry, saturated, and drained states. Both density and moisture calibration curves were developed from this set of standards. The cohesive soil, Permian red clay, was mixed to various water contents and compacted in modified barrels by a power tamp.

Relatively small confined masses of material used as calibration standards are limitations in themselves. The moisture probe has a sphere of influence which varies with the moisture content. Van Bavel (Ref 15) has determined that this zone around the probe can be expressed by the following equation:

$$R \text{ (inches)} = 5.9 \left(\frac{100}{\text{Vol \% Water}}\right)^{1/3} \qquad (3.1)$$

where R is the radius of a sphere of neutron influence with its center at the source. This relationship is invalid below three per cent of water by volume. Richards (Ref 5) states that the minimum radius is from about four inches in water to over eighteen inches in dry soil. Other investigators have stated that the sphere of influence may vary from twelve to sixteen inches in ordinary soils. Troxler laboratories references Van Bavel's equation in its literature on moisture probe operation (Ref 16).

It is necessary to keep the sphere of influence of the moisture probe within the container to get valid results. The container's dimensions directly limit the moisture content to above a critical minimum value. A small container will, therefore, have a high minimum moisture content. In this connection Van Bavel (Ref 12) recommends that for calibration purposes at low moisture content, a homogeneous soil mass of at least four-foot dimensions is required.

The sphere of influence for the Troxler density probe is approximately five inches (Ref 17). This dimension is not of critical concern in the study as it is well within the boundaries of the calibration standard container.

The previous calibration experiments obviously illustrate the obstacles of equipment calibration. Although non-soil materials give excellent results in some cases, the problems involved with soil still exist. The authors chose to calibrate using soil standards with controlled moisture contents. Artificial standards were not considered. Furthermore, the authors chose cohesive soils as calibration media for two sets of standards. Cohesive soils have been neglected because of mixing and placement problems, but generally they are of critical concern in highway subgrades. Soils with high clay content are quite sensitive to moisture changes. In Oklahoma expansive clays are abundant and are thought to cause many subgrade failures.

It should be noted that this study required the calibration of both moisture and density probes. Subgrade moisture investigation requires engineering moisture content (weight ratio of water to soil solids in a soil mass). Water quantity, in pounds per cubic foot, cannot be related to general soil properties, liquid and plastic limit, shrinkage limit, optimum moisture content, etc. To obtain engineering moisture content, one must subtract the amount of water (pounds per cubic foot) from the wet density to obtain the dry density. The moisture content in pounds per cubic foot is divided by the dry density to determine the engineering moisture content in percent. Therefore, civil engineering requirements put an additional burden on calibration. Both probes must be accurate to produce acceptable data.

CHAPTER IV

CALIBRATION PROCEDURE

Materials

One sand and two cohesive soils were selected for the calibration standards. All materials are found in Oklahoma and represent a general cross-section of soils encountered in the state.

A fine yellow sand was located four miles west of Sapulpa, Oklahoma on highway US 66. As is seen in Fig 4.1, the sand is quite uniform. This material was weathered from a limonitic yellow sandstone formation which outcrops in the immediate area. It was selected because its grain size was between the coarse aggregates used by LeFevre and Manke (Ref 3) and cohesive soils.

Permian red clay (PRC) was selected as the second material because of its abundance in the Stillwater area. Also, LeFevre and Manke used the soil in their preliminary calibration study, and the authors desired to extend their work with the material. This clay was obtained from the excavation of the mathematics and statistics building on the Oklahoma State University campus at a depth of ten feet. Its physical properties can be seen in Fig 4.1 and Table 4.1. Grain size distribution data was obtained by hydrometer analysis.

The third soil was a brown silty clay found four miles north of Stillwater, Oklahoma on highway US 177. This material was selected

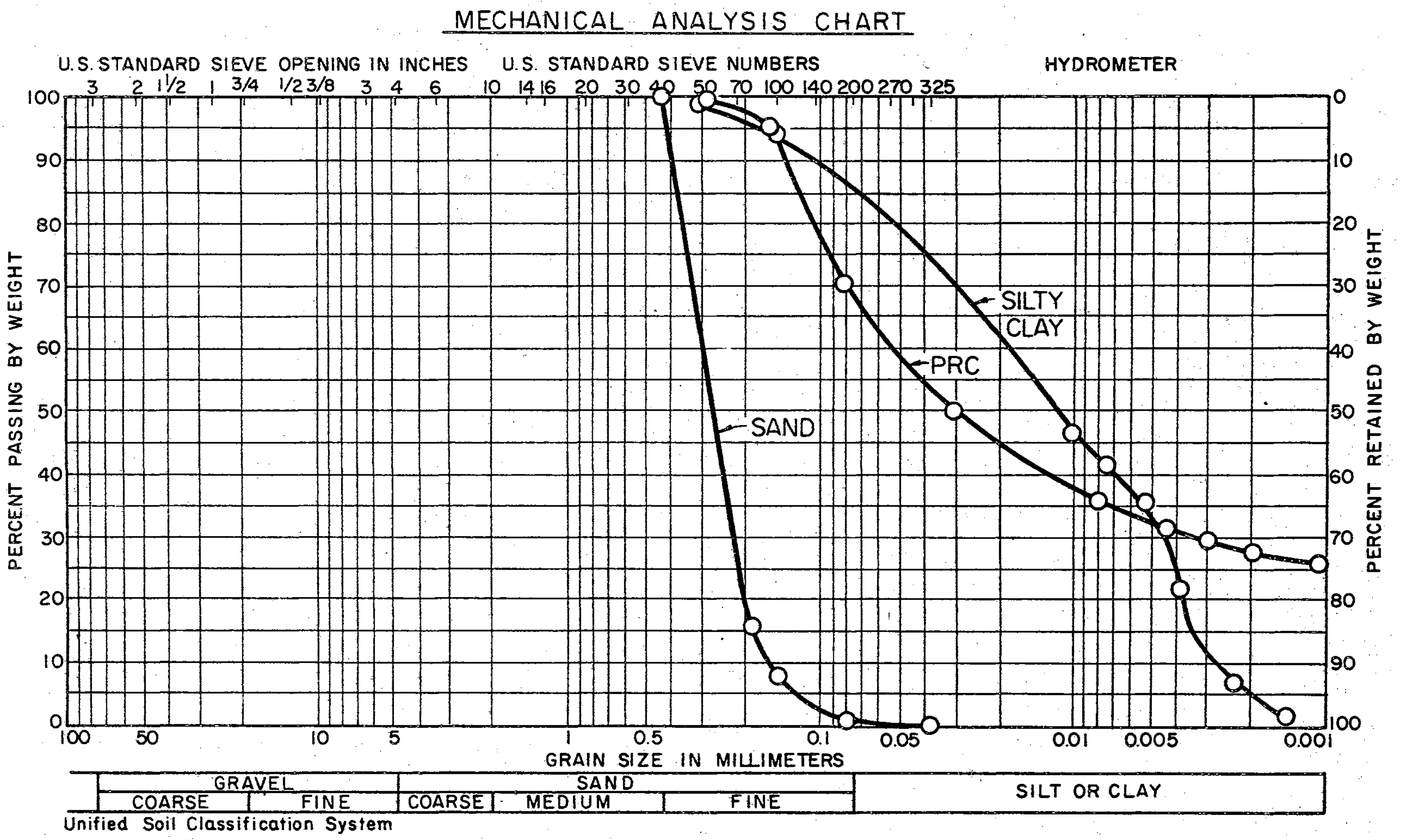


Figure 4.1. Grain Size Distribution of Calibration Soils

Soil	Physical Properties				Lineal* Shrinkage	Classification AASHO--UNIFIED	
	w_L	w_p	I_p	G_s			
Fine Uniform Sand	NP	NP	NP	2.66	NP	A3	SP
Permian Red Clay	41.3	22.0	19.4	2.72	11.8	A7	CL
Brown Silty Clay	23.1	18.0	5.1	2.66	5.3	A4	ML

*Texas Highway Department Bar Method

TABLE 4.1. PHYSICAL PROPERTIES OF CALIBRATION SOILS

because it had different characteristics than the Permian clay or sand, i.e., another distinct soil type. Its physical properties, see Table 4.1, indicated it was a good subgrade material; this is unusual in north central Oklahoma because Permian red clay predominates. The soil deposit was near a small stream and therefore it is believed the silty clay was deposited by water action. A trace of sand was also found in the soil. The grain size data was obtained from hydrometer analysis.

Thus, the selected soils for nuclear probe calibration provided two good and one very poor subgrade material. A grain size range was obtained that included fine sand, silt, and colloidal size clay. With the inclusion of LeFevre and Manke's data on nuclear probe calibration, the entire spectrum of Oklahoma soils would be, to some extent, investigated.

Soil Preparation

The sand was wet sieved through a U.S. Bureau of Standards No. 16 sieve into a thirty gallon waste can. Most of the organic matter (roots, weeds, etc.) was retained on the sieve screen and all of the sand passed into the can. The majority of clay particles found with the sand remained in wash water suspension and were removed as the water overflowed the container. The sieved sand was then oven-dried and placed in G.I. cans for storage.

The cohesive soils were processed differently. The soil was oven-dried for twenty-four hours, see Fig 4.2, and allowed to cool. The dry soil was placed in a Los Angeles Abrasion test machine, shown in Fig 4.3, and pulverized until the soil passed the U.S. No. 40 sieve in a Gilson Mechanical Testing screen, shown in Fig 4.4. The PRC has

Figure 4.2. Oven and Dry Soil

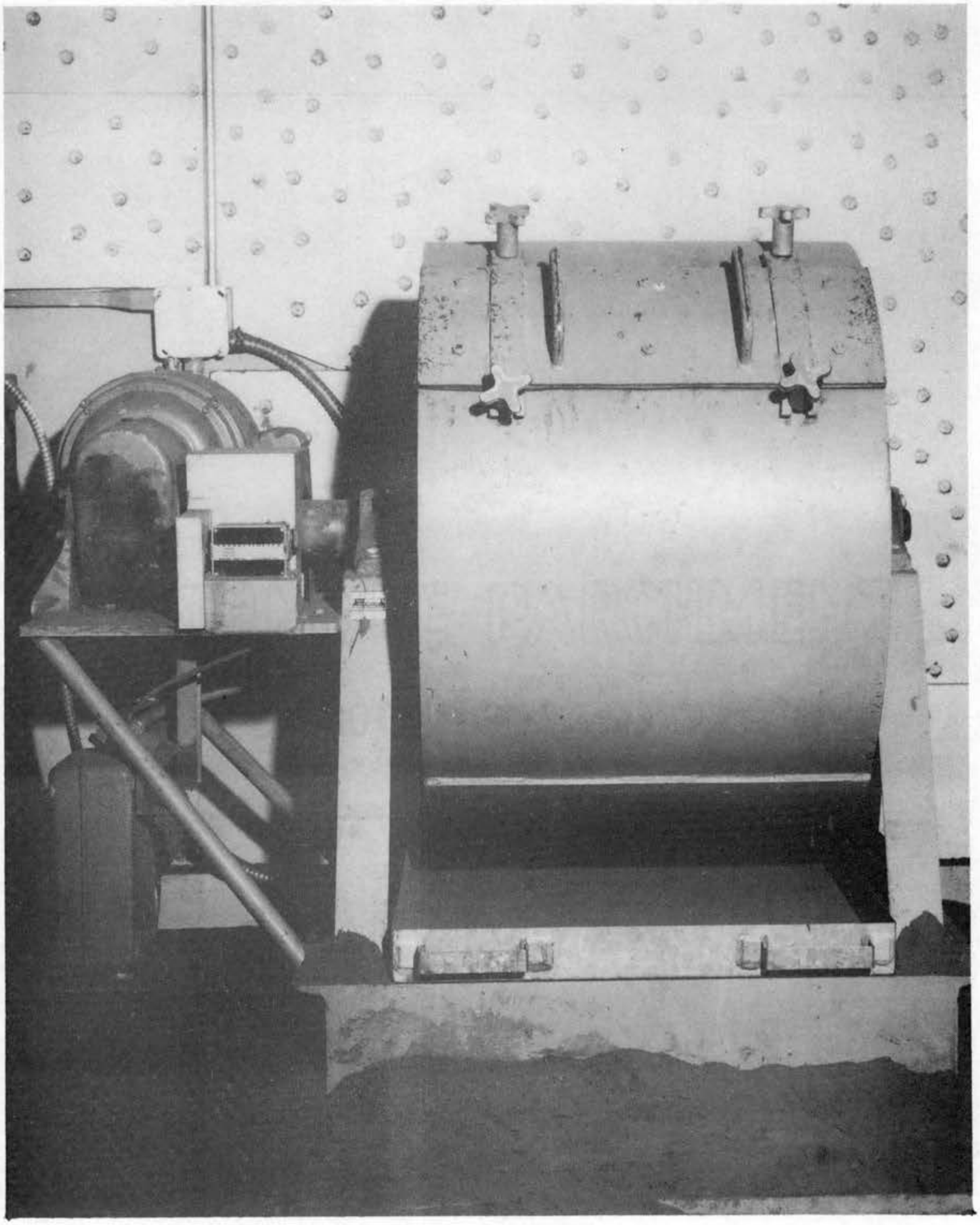


Figure 4.3. Los Angeles Abrasion Test Machine

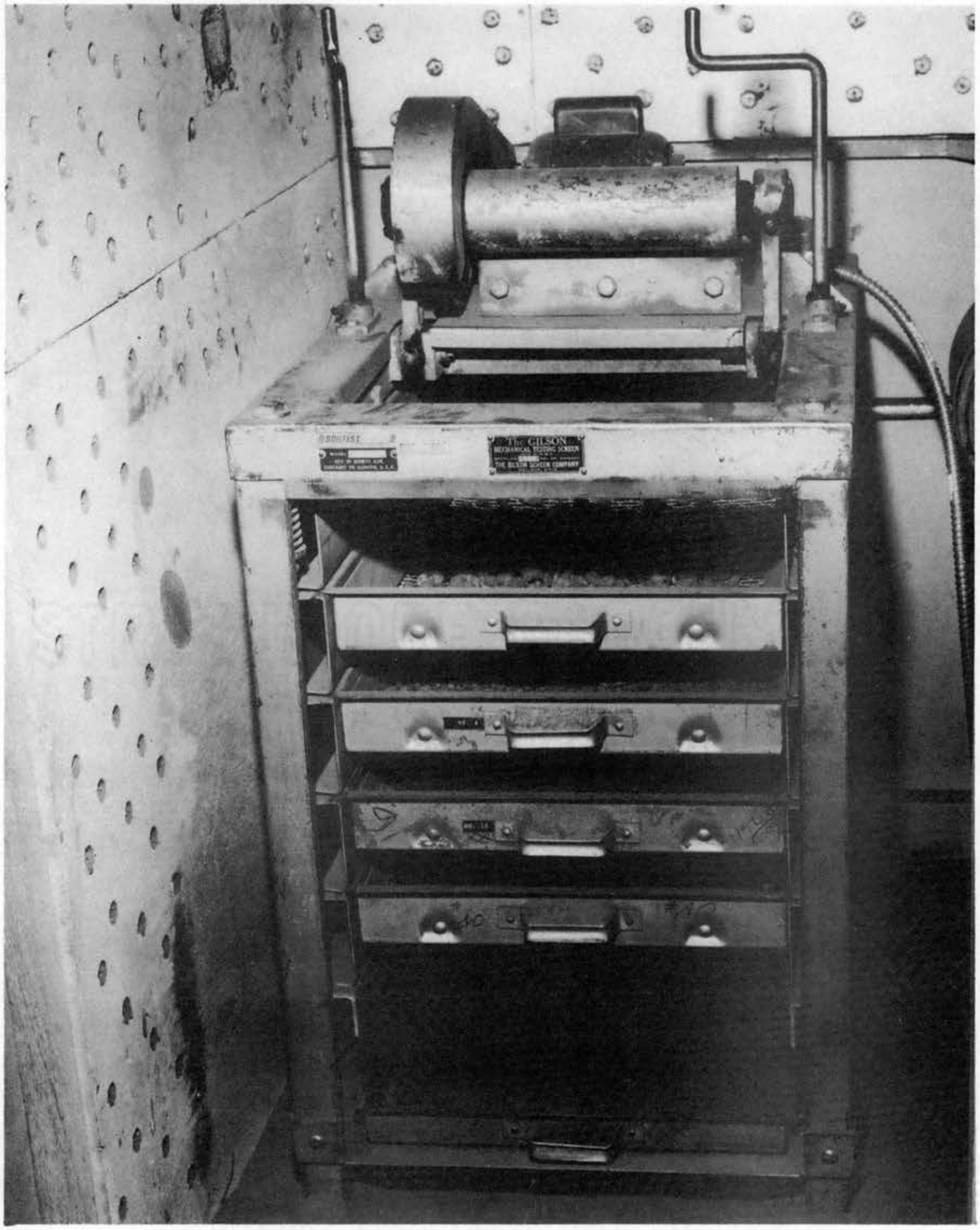


Figure 4.4 Gilson Mechanical Testing Screen

a very high dry strength and it required about one hour for the abrasion machine to break up thirty pounds of soil. Only one third of this material would pass the No. 40 sieve. Therefore, it is recommended that a grinder be employed, as shown in Fig 4.5, to break down all soil retained on the No. 40 sieve. Otherwise, the time required for processing highly plastic clays is extremely long. The silty clay broke down quite easily in less than thirty minutes per thirty pound load. Its yield (passing the No. 40 sieve) was above sixty per cent and any material retained was pulverized in the grinder. The soil before and after processing is shown in Fig 4.6 in the G. I. storage cans.

It should be noted that if highly plastic clays are being processed by the above procedure, all personnel in the immediate working area should wear face masks to prevent inhaling the dust. The clay size particles remain in suspension indefinitely if proper ventilation of the area is not available. This creates a definite health hazard for the personnel involved.

Container Preparation

A fifty-five gallon drum was cut down to a height of approximately twenty-four inches, as seen in Fig 4.7. The inside was painted with red lead and then with a coat of epoxy to prevent corrosion. A drainage outlet of galvanized pipe fittings was placed on each barrel. A plywood template was cut to fit under the barrel to keep deformation of the bottom at a minimum during compaction. Each barrel rested on a channel frame. The drainage outlet, template, and steel frame are shown in Fig 4.8. A steel frame and hoist, shown in Fig 4.9, was used to move the compacted standards, which weighed over six hundred pounds

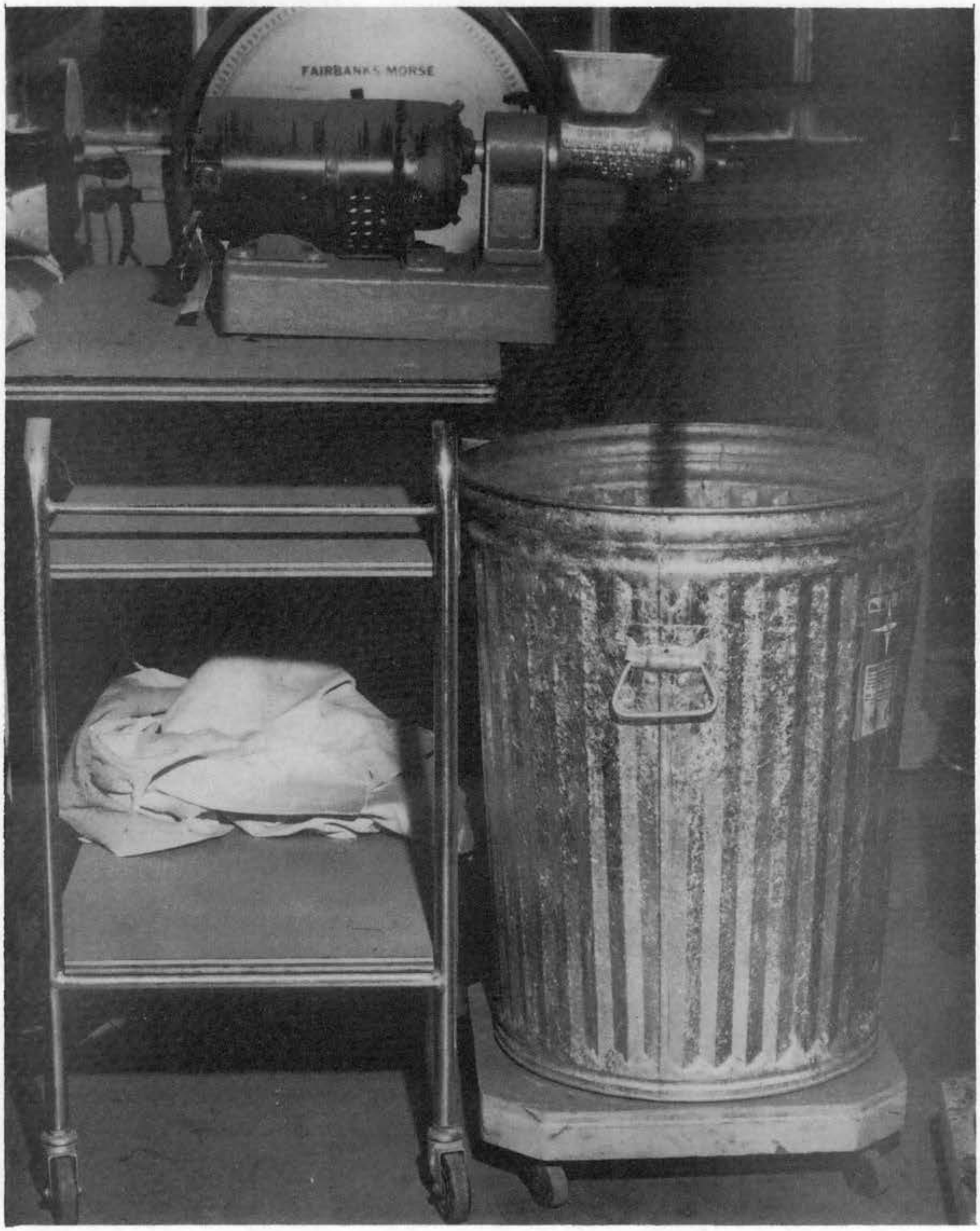


Figure 4.5. Soil Grinder

Figure 4.6. Soil Before and After Processing

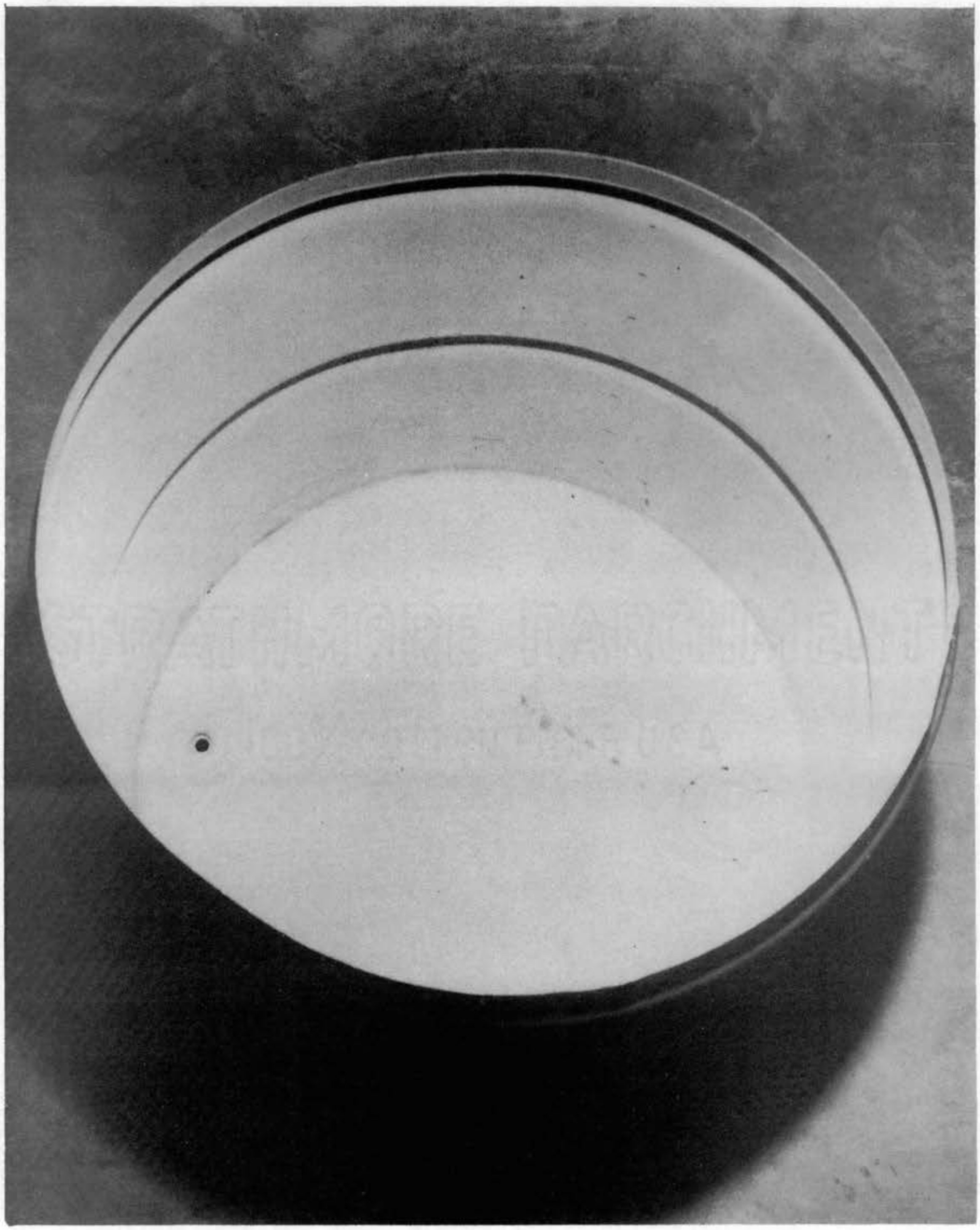


Figure 4.7. Empty Standard Container

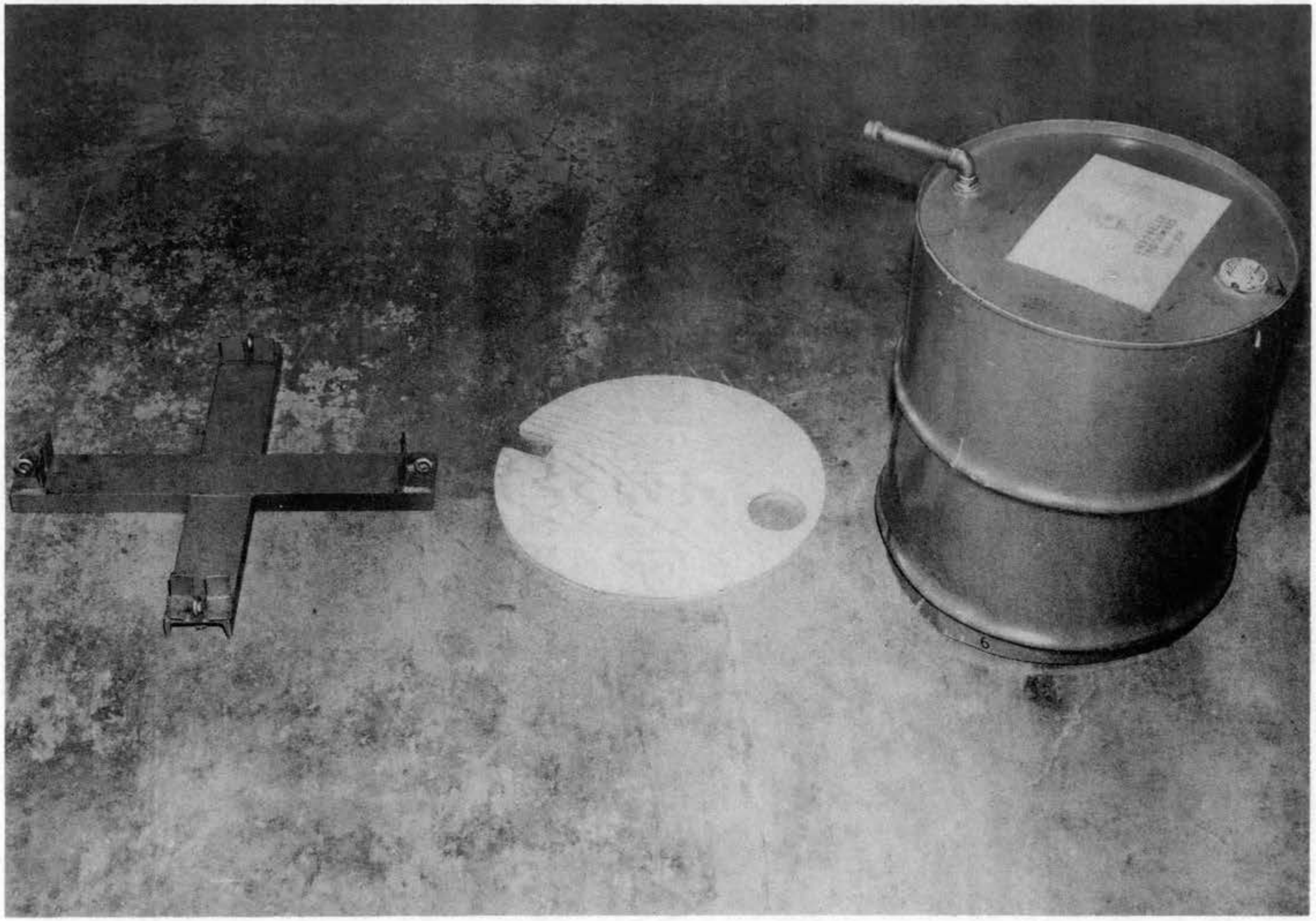


Figure 4.3. Steel Frame, Plywood Template, and Standard Container with Drainage Facility

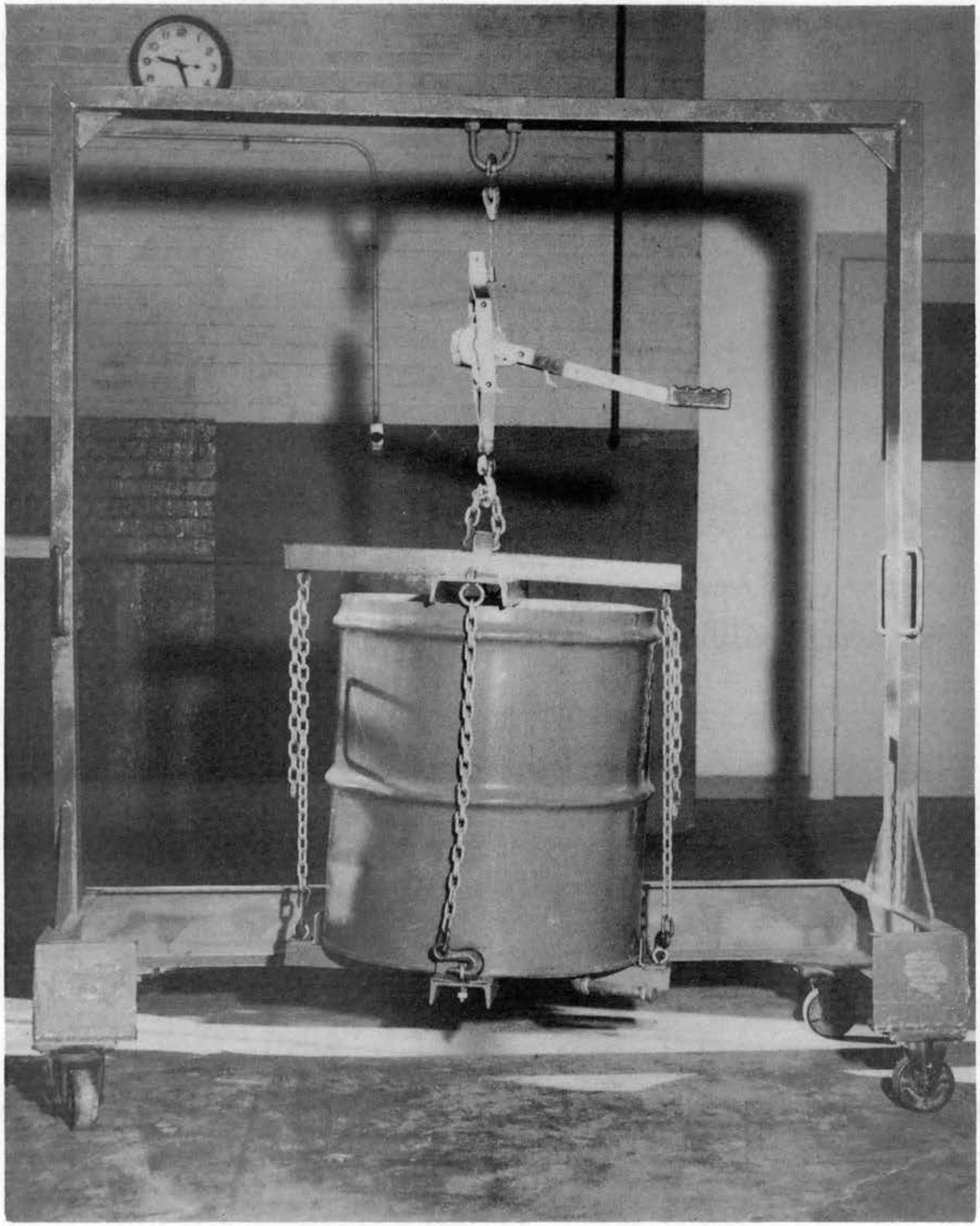


Figure 4.9. Steel Frame and Hoist

when completed.

After painting, each barrel was filled with water to a predetermined mark (twenty-one inches in this study). This mark represented the proposed top of the soil mass after compaction. The barrel was weighed and the volume of the container to this specified mark was determined from weight of water calculations.

Construction of Standards

All materials were mixed and compacted in three inch lifts. The uniform sand had a very narrow range of obtainable void ratios. Therefore, its minimum void ratio ($e = 0.67$) was selected as the design value. The minimum moisture content for the standard (based in Eq 3.1) was calculated in lb per cu ft and converted to engineering moisture content. Five moisture contents were chosen for the set of standards. Therefore, with void ratio and moisture content selected as control values, the amount of water and sand for a compacted three inch lift was calculated.

The two cohesive soils were prepared in similar manner. Using Harvard Miniature apparatus, Standard AASHO and Modified AASHO compaction curves were developed and the minimum allowable moisture content for an eleven inch sphere of influence was calculated from Eq 3.1. These data are shown in Fig 4.10 and Fig 4.11 for the PRC and silty clay. Moisture contents for the PRC standards were selected between 16% and 25%. Note that standards No. 8 and No. 9 are at or above the plastic limit. The silty clay standards were chosen between 9% and 18%. Standard No. 10 was mixed to obtain a moisture content which theoretically would allow the sphere of influence outside the

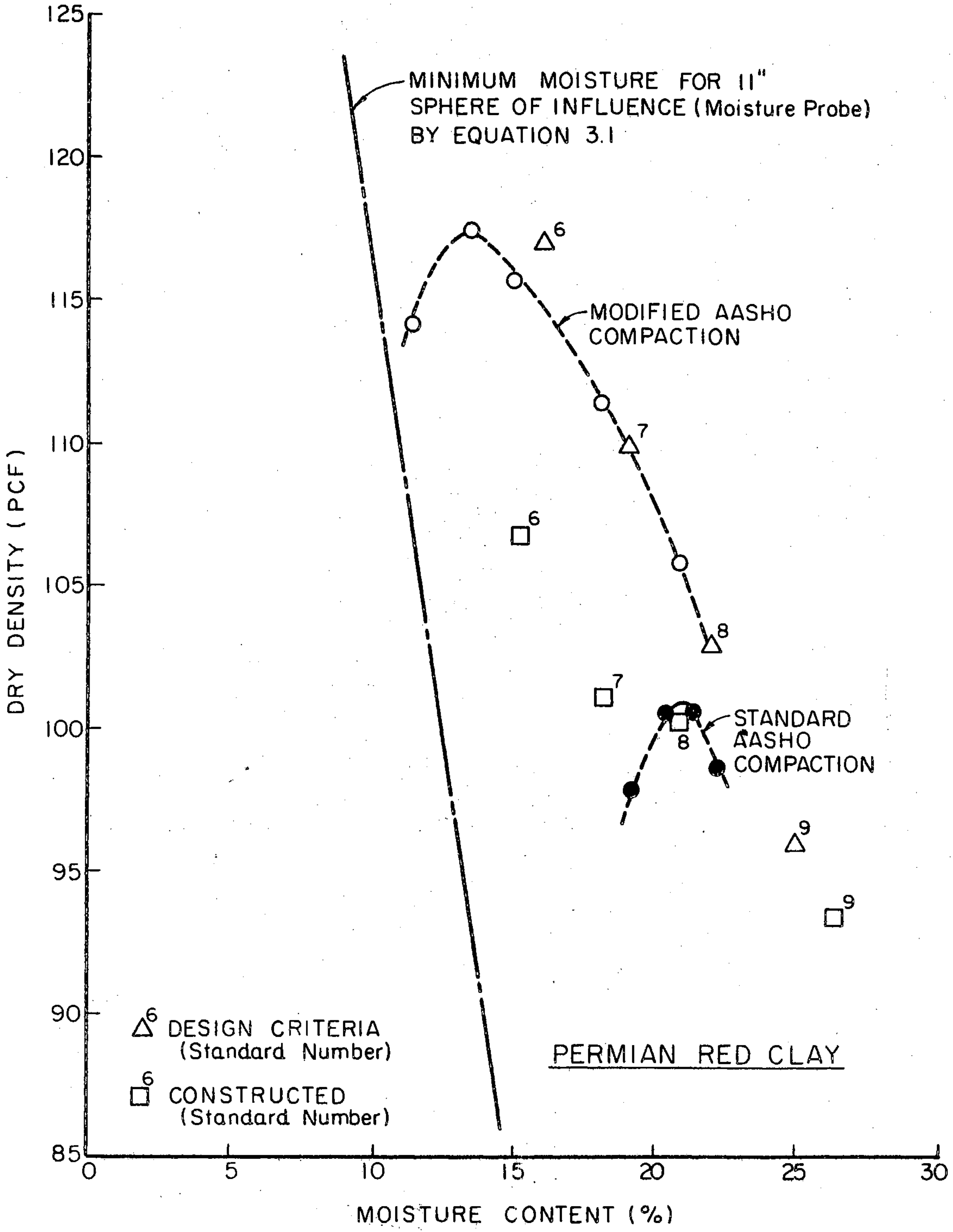


Figure 4.10. Compaction Curve, Permian Red Clay

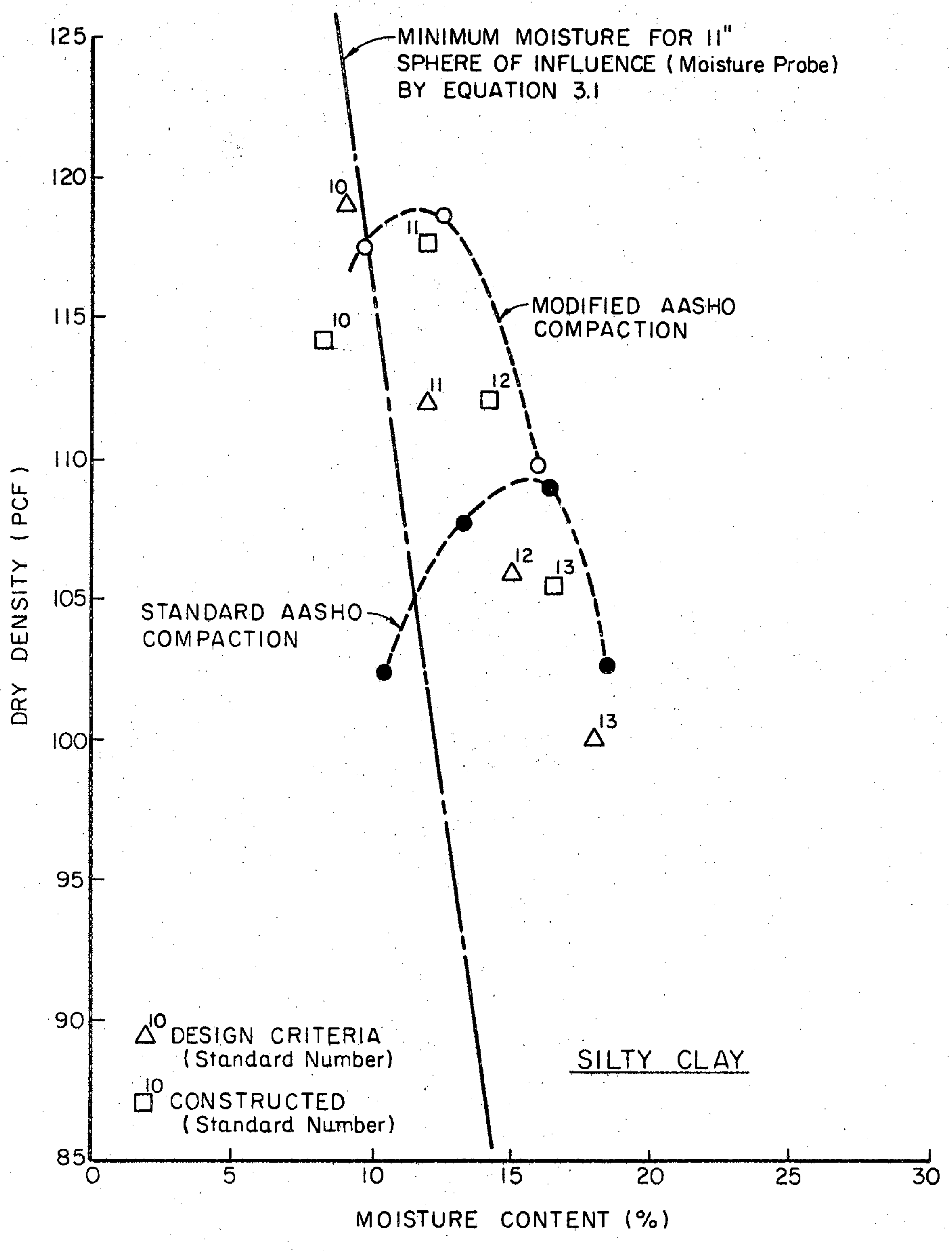


Figure 4.11. Compaction Curve, Silty Clay

container. Standard No. 13 was at the plastic limit of the soil.

Also, it was desired to obtain a large range of wet density values since the standards would be used for density probe calibration research. The design void ratios of the cohesive materials were selected based on the compaction curves obtained. Points above the Modified AASHO curve and below the Standard AASHO curve were included to determine the most efficient region of compaction with the equipment available.

The correct amounts of soil and water were weighed and mixed until well blended. Hand mixing was employed in all cases as shown in Fig 4.12. The processed cohesive soils were assumed to have an initial moisture content of two per cent. Hand mixing was selected because it is the only quick method of working with clays in dry powder form. Water was added and the mixture troweled thoroughly. No spraying was used because of evaporation problems.

Once the soil and water were mixed, the "mud" was shoveled into the standard, as is shown in Fig 4.13, and compacted immediately with a power tamp as shown in Fig 4.14. Three inch gradation marks were placed on the inside wall of the drum prior to compacting, and the soil was compacted until each mix of soil and water filled the three inch lift. Seven lifts were placed in each standard. The barrel was covered with polyethylene sheeting to prevent evaporation. The compacted standard and evaporation cover are shown in Fig 4.15.

It should be noted that this approach to the compaction phase eliminated any problems with timing or otherwise controlling the compactive effort applied to each lift. The lift was compacted until it fit the design requirements and the three-inch lift thickness was

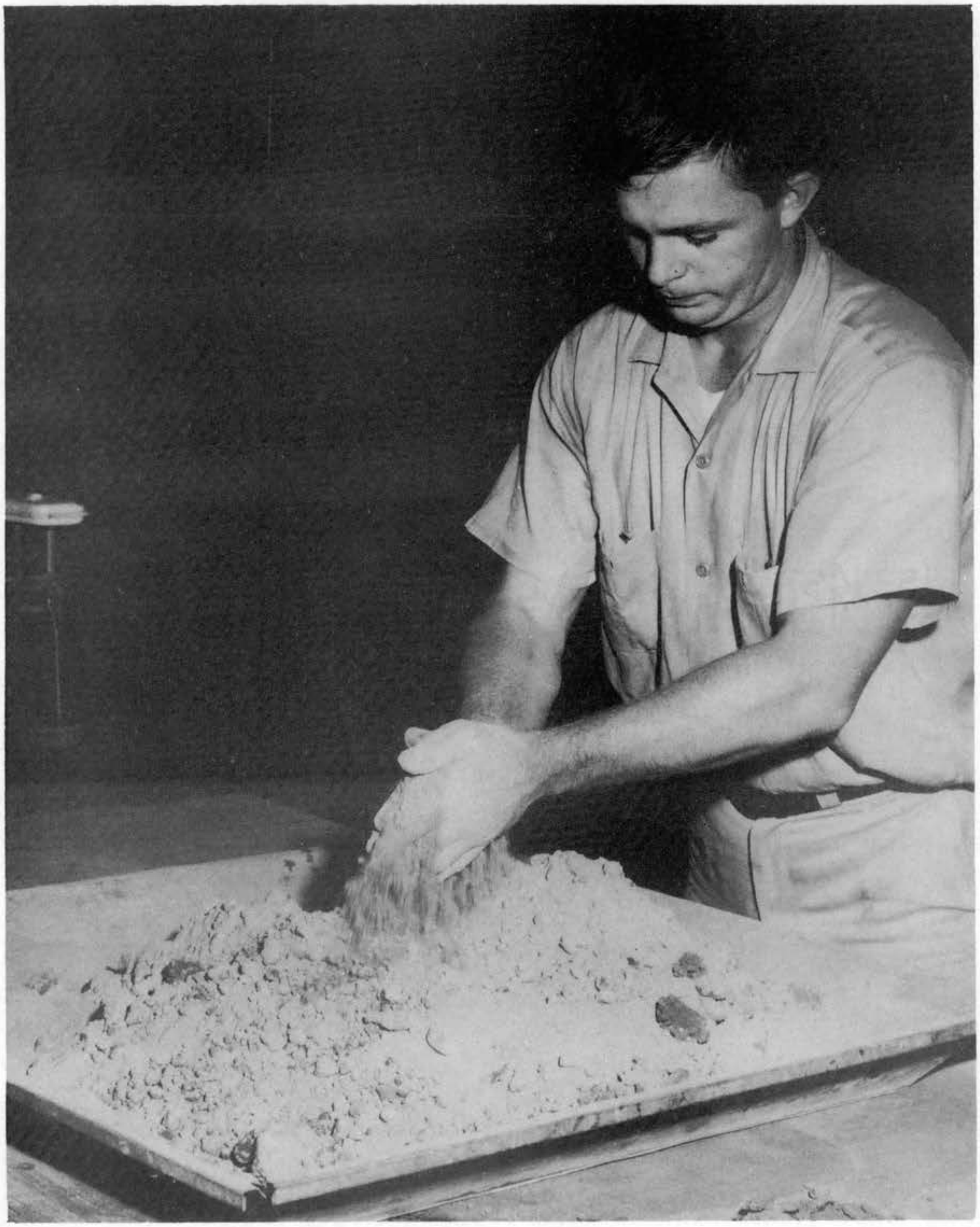


Figure 4.12. Hand Mixing Procedure

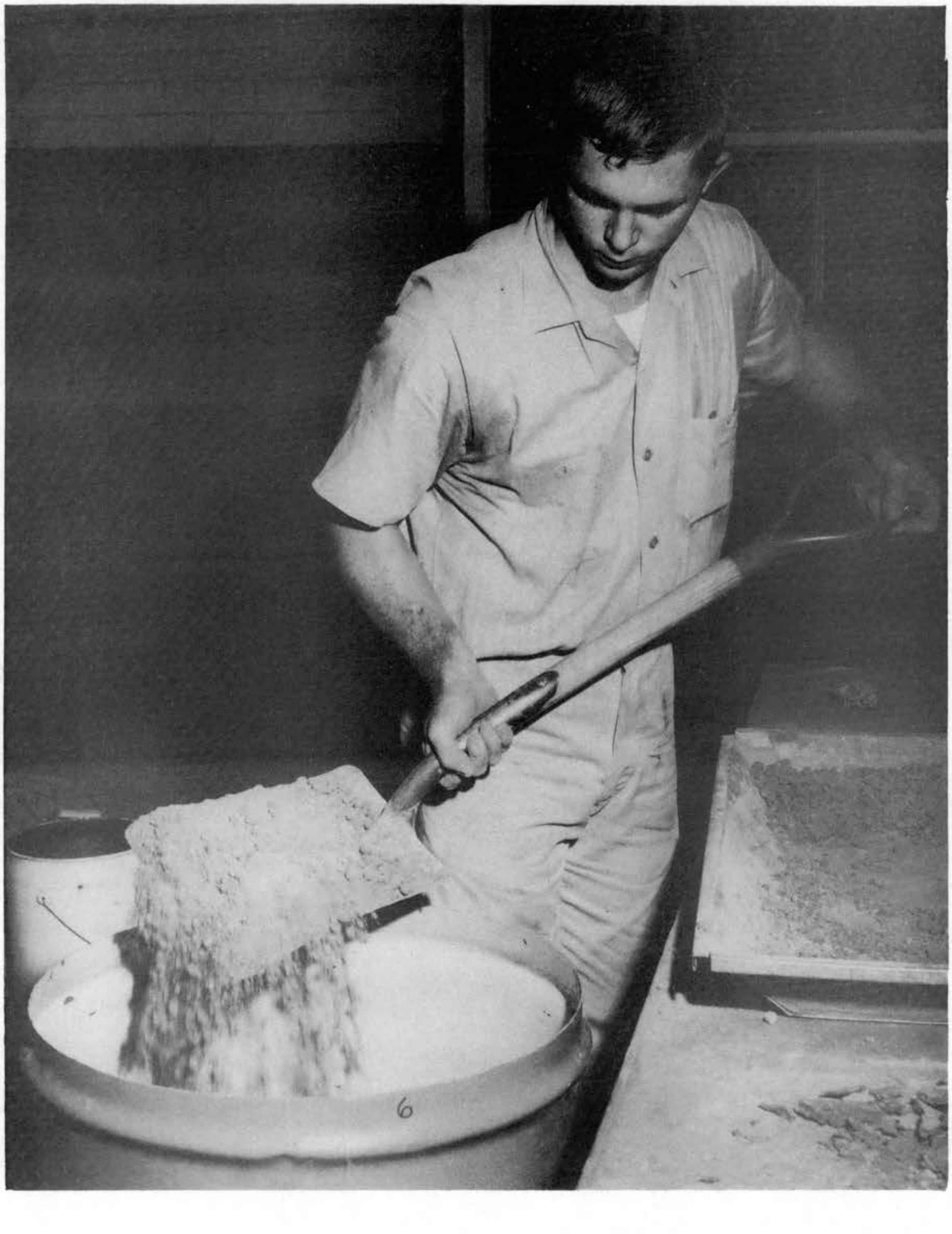


Figure 4.13. Lift Placement

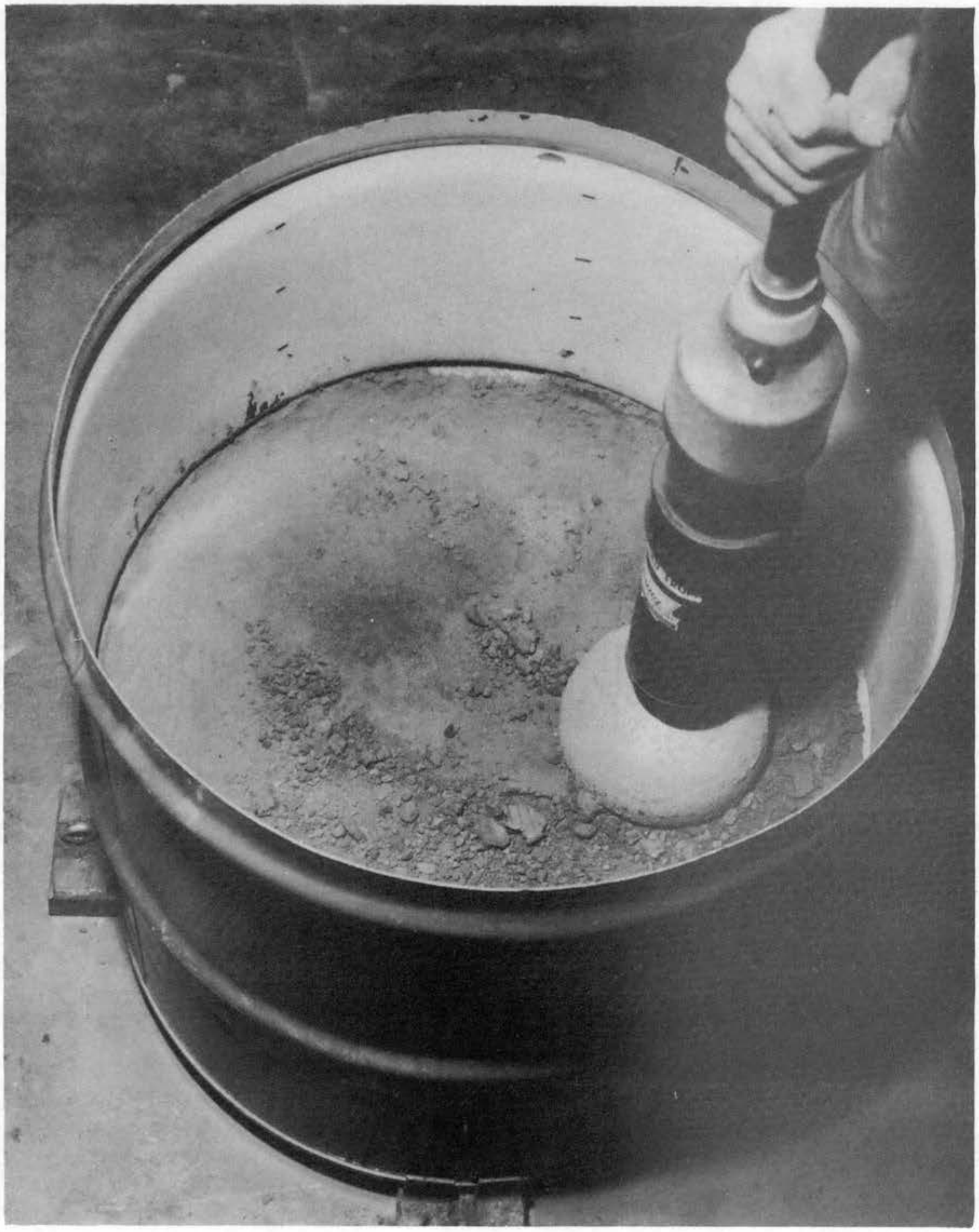


Figure 4.14. Compaction Procedure

Figure 4.15. Standards without Access Tubing

achieved.

Later, the standard was weighed to determine the weight of the soil mass. Using the volumes previously determined, the average mass density of the soil was determined.

The sand standards were allowed to cure for ninety days, the PRC, forty-five days, and the silty clay, fifteen days. Standard construction schedule created the variation in curing times as access tubing for the entire group of standards was placed during one continuous operation. Furthermore, the volume of soil required for four or five standards made it impractical to store all soils and compact the standards at one time. Therefore, the three types were compacted at intervals, requiring the storage of only one soil at a time.

To install the aluminum access tubing, a two inch OD thin wall stainless steel tube was pushed slowly into the center of the compacted soil. This tube had a 1/16 inch wall thickness and was honed at one end to minimize soil disturbance as it was forced into the clay. The equipment and procedure are illustrated in Figs 4.16 and 4.17. A wooden guide template was placed on the soil surface to guide the tube into the standard. The cutting tube was pushed by a Tinius Olsen 200,000 pound universal testing machine. The steel tube was removed with an overhead crane and moisture samples were taken from the cored soil. An aluminum access tube (2.000 inch OD, 1.900 inch ID, 24 inch length) sealed at the bottom was inserted into the cored hole. The standard was recovered with polyethelene sheeting to prevent evaporation. The completed standard with access tubing is shown in Fig 4.18 and a section of a typical standard is illustrated in Fig 4.19.

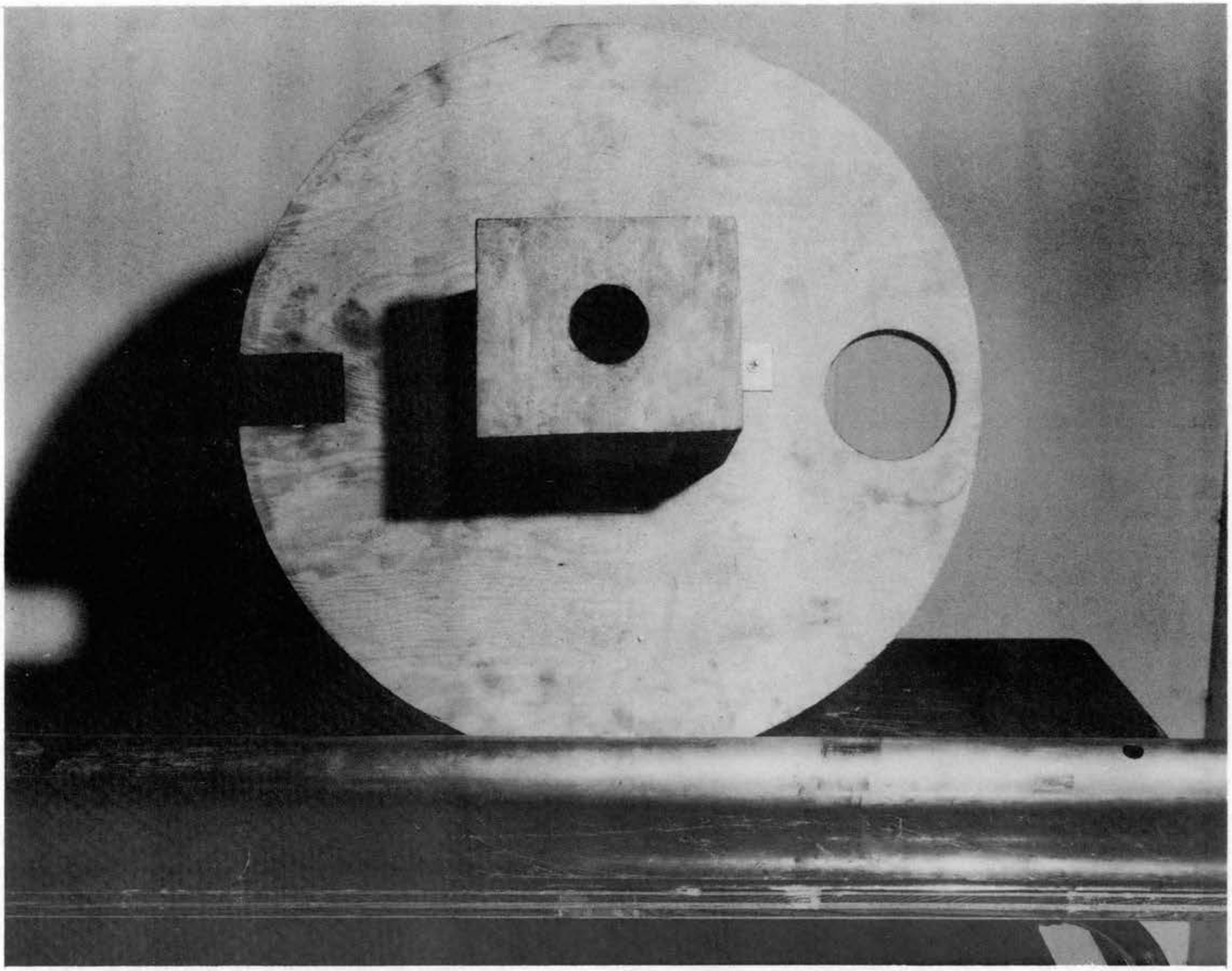


Figure 4.16. Template Guide and Steel Cutting Tube

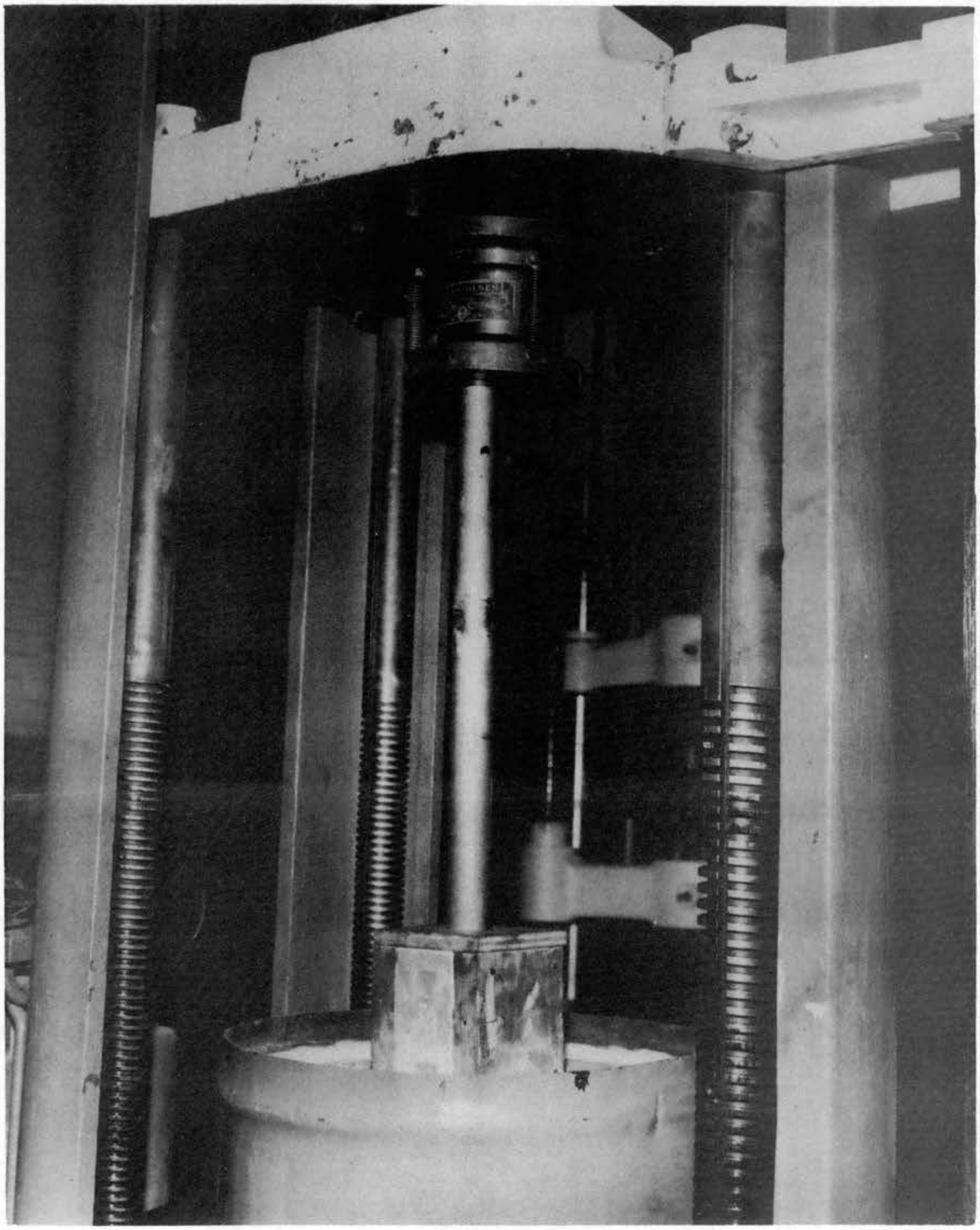
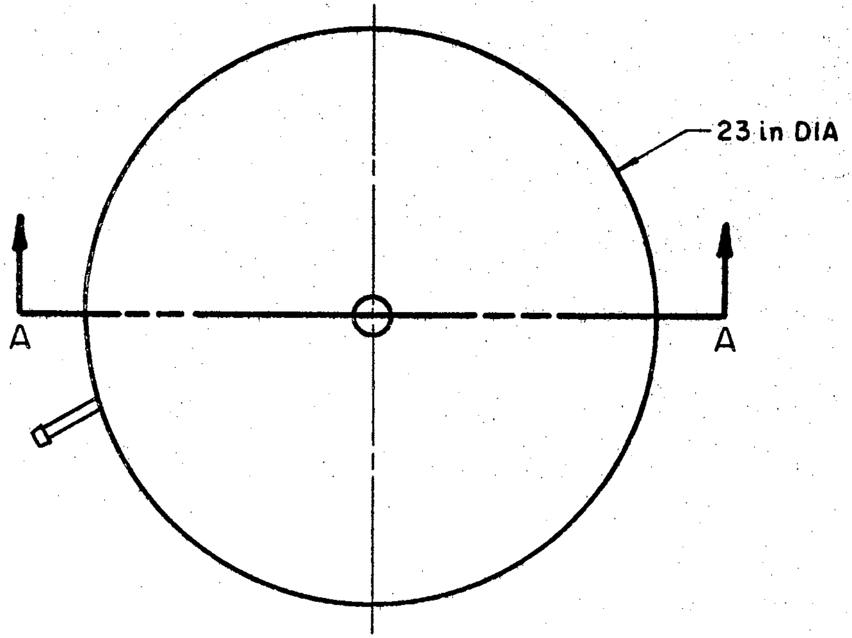
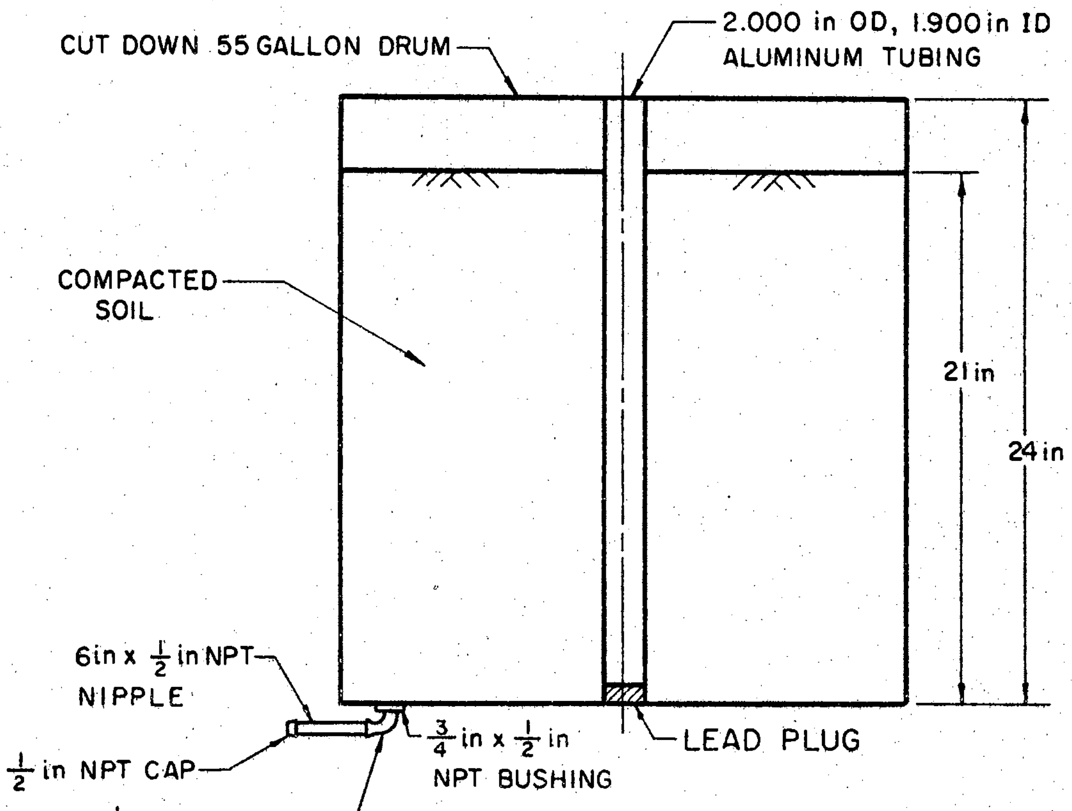


Figure 4.17. Coring Procedure

Figure 4.18. Completed Calibration Standards



TOP VIEW



SECTION A-A

Figure 4.19. Typical Calibration Standard Section

Discussion of Construction Procedure

The mixing and placement procedures as previously described worked well. Hand mixing of the clay was tedious but it is the best method. The clay near or above the plastic limit is almost impossible to mix with a mechanical mixer as the powdered clay "balls up" in small spheres which are saturated in the center and dry on the surface. The sand mixed well with very little difficulty. Mechanical mixing is a possibility with fine granular soils especially if thicker lifts are required. The length of mixing time varied with the soil and the moisture content required. However, an average mixing time of fifteen minutes ~~per lift~~ for the silty clay and clay, and ten minutes for the sand is reasonable.

Evaluation of the constructed sand standards is somewhat difficult. The moisture contents were all very near to the total saturation value for a void ratio of 0.67. Furthermore, some moisture migrated to the bottom in all the sand standards. Sand above the "water table" was from two to four per cent drier in moisture content as the surface was approached. This provided a moisture profile in the sand. This would not effect the bulk or mass density and average moisture content calculations but the moisture and wet density fluctuations would affect the nuclear equipment responce.

Access tube installation in the sand standards created an additional problem. The sand core did not remain in the core tube and was difficult to remove from the standard. The core separated near the saturation line in the standard because of the difference in unit weights of the material. One attempt to remove the remaining core at the bottom of the hole (Standard No. 2) caused a large disturbance of the sand near the hole and sluffing of soil into the hole. Therefore, the access tubing was not inserted to the bottom of the sand standards, but it did penetrate the saturated zone.

Sand Standard No. 2 had a design moisture content at the calculated total saturation for the minimum void ratio. The lifts in this standard did not compact well and the sand began to bulk which indicated total saturation. This behavior verified the void ratio calculations for the sand. Attempts to vary the sand void ratio failed because of the uniformity of the material.

The main problem encountered with the cohesive soil was in mixing, Placement was done efficiently with the power tamp in most cases. However, standards No. 6 and No. 10 were designed above the Modified AASHO curves for the respective materials. As can be seen in Figs 4.9 and 4.10, the constructed standards had good moisture contents when compared to the design criteria, but the average bulk densities were not fulfilled. It is believed that the Modified AASHO curve represents the limit of compaction capabilities for this procedure and equipment. Standard No. 10 is designed to have a moisture content less than that required by Eq 3.1 for the standard container. The significance of this point will be discussed in Chapter V.

Generally, the moisture contents of the cohesive standards were close to design specifications, but the density values were somewhat less. The dry density figures were computed on the basis of the average wet density data.

A possible source of human error in this procedure could be in the weighing. Volumes for the standard containers varied between 4.66 cubic feet and 4.92 cubic feet. An average value was 4.8 cubic feet for the volume of a compacted soil mass. The platform balance used was accurate to one half pound. It should be noted that a large error of five pounds made in weighing the water filled barrel would alter the average bulk density by three pcf. The same error when determining the soil weight would alter the average bulk density about 2 pcf.

Another possible source of error was the estimate of initial water content of the soil. However, as stated previously, the constructed standards had moisture contents quite close to the design criteria. It is believed that this factor was not in error. It should be standard procedure to store the clay soils in sealed containers to keep the soil from absorbing large amounts of water from the atmosphere.

The core moisture samples from the cohesive soil standards were quite uniform with a variance of less than one per cent moisture content. This is considerably less than one pcf in any of these standards.

The access tubing fit tightly in the cohesive soils with an absolute minimum of air gap between soil and tube wall. The outside of the steel cutting tube was lubricated with silicon grease to prevent as much soil disturbance as possible. It worked well for the clay soils. In contrast, the access tubing in the sand standards did not fit as tightly. Perhaps the best procedure for sands is to compact the soil around the access tube (Ref 3).

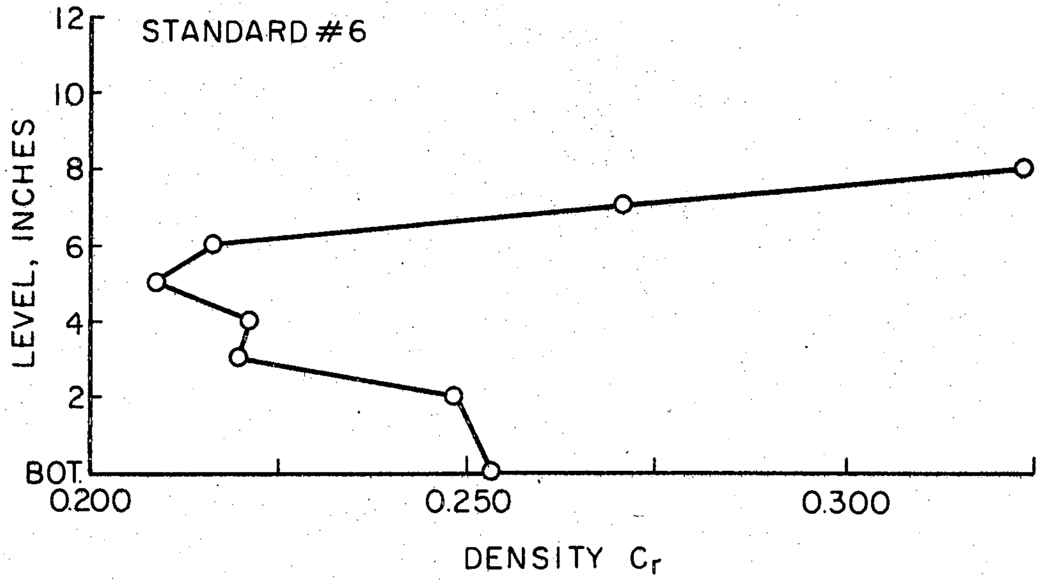
Data Collection Procedure

Probe readings were taken at two inch intervals starting at the bottom of the access tube and progressing upward. The data taken at each level consisted of 10 one minute counts which placed the pulse response at the ninty-nine per cent confidence level with a statistical deviation of less than one per cent (Ref 18). The probe was moved toward the top of the standard until a large deviation in equipment response was noted.

Ten standard counts, each of one minute duration, were taken before and after each series of data readings. The standard count is the probe response in its protective shield, which acts as a secondary reference standard.

Typical probe responses are shown in Fig 4.20 for the density probe and Fig 4.21 for the moisture probe. The term C_r is the count ratio, or the ratio between probe response in the calibration standard and the average count in the reference standard.

The density response is of particular interest. The small decrease in probe response is understandable as the probe is raised from the bottom of the access tubing. The probe is no longer influenced by the bottom of the barrel or the lead plug in the access tubing. However, as the probe reached the five or six inch level a sharp decrease in C_r was noted. This was characteristic of all the cohesive soil standards. It is thought that the compaction procedure employed produced this effect. The standard barrel was most stable in the steel frame during compaction of the third, fourth, and fifth lifts. The most efficient compaction was developed in this region and



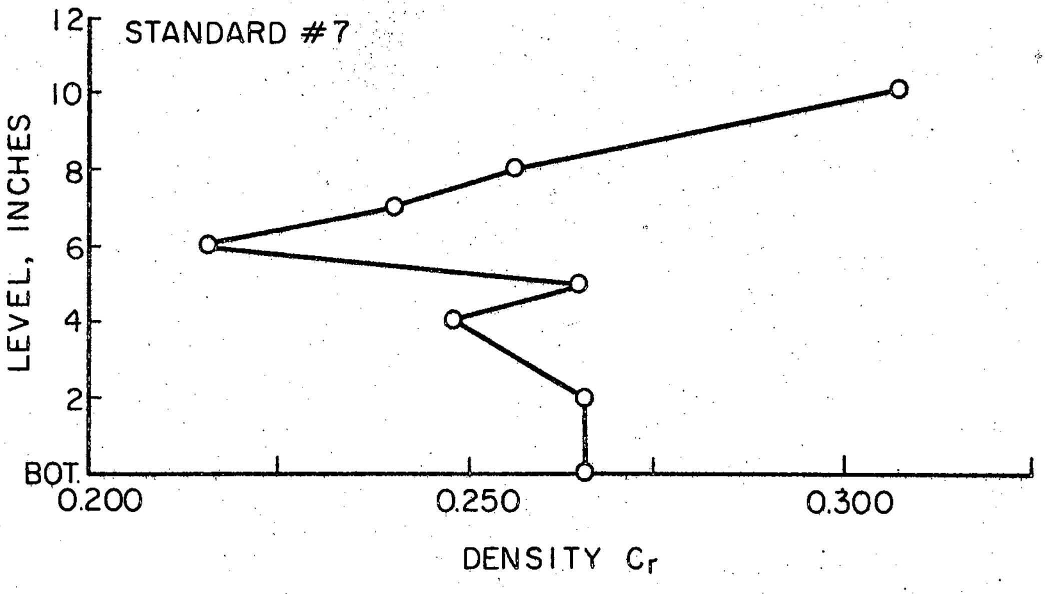


Figure 4.20. Typical Density Probe Response

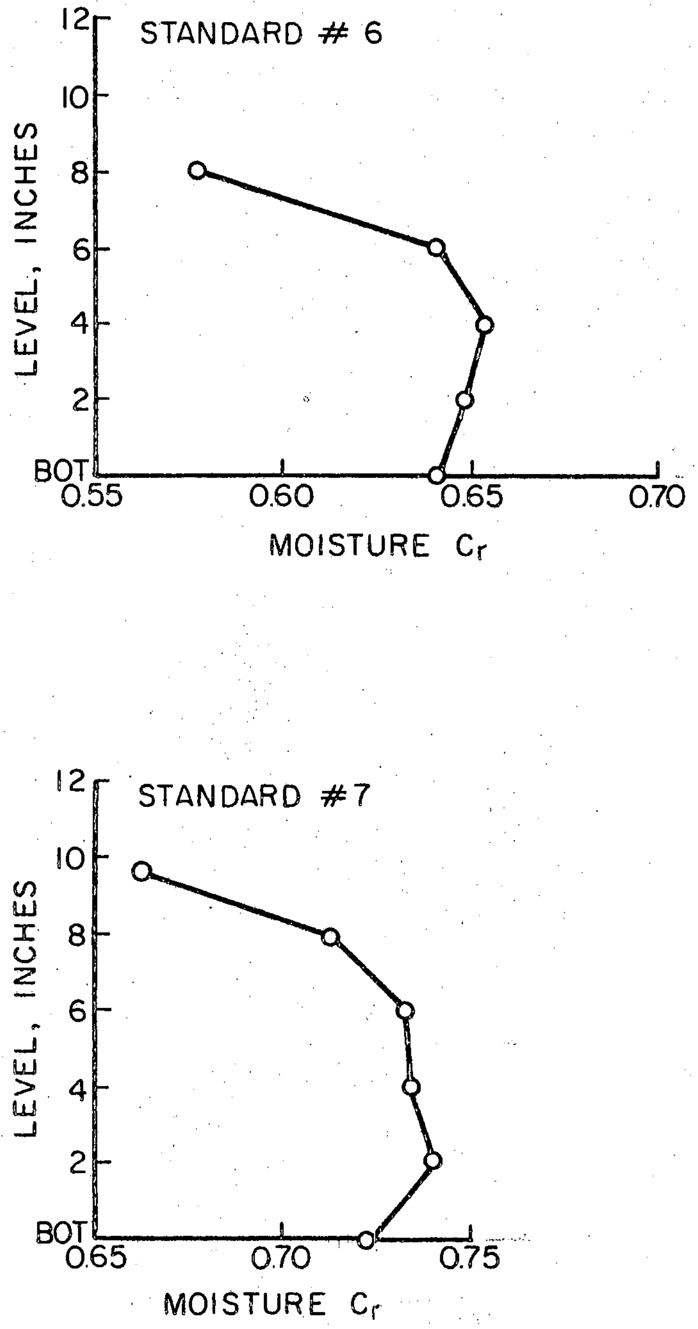


Figure 4.21. Typical Moisture Probe Response

in the soil immediately below it. Part of the compaction effort in the upper lifts was transmitted to the lower levels causing an area of higher densification. As the probe approached the surface of the standard, the C_r was influenced greatly as is illustrated by the sharp increase in pulse count. This response does not give valid indication of density at this level.

The moisture response indicated a uniform distribution of moisture in the clay standards with a gradual decrease in count ratio as the probe was raised. This decrease is due to container geometry as the detector tube was at or near the soil surface.

In both cases, the physical dimensions of the standards limit the region of valid probe response to about six inches. The data obtained with probe at the bottom of the access tube was utilized because it differed only slightly from the probe response in levels immediately above it. Data obtained in the regions which are discussed previously as giving erroneous pulse counts was not considered. The moisture probe data was judged valid until the response began to decrease characteristically.

Therefore, it is recommended that future soil standards be designed with twenty-four to thirty inch soil thicknesses to increase the region of valid probe response. The nuclear equipment used in the study is illustrated in Figs 4.22 and 4.23.

Figure 4.22. Nuclear Equipment

Figure 4.23. Data Collection Procedure

CHAPTER V

DATA PRESENTATION AND DISCUSSION

The following chapter correlates the nuclear equipment response to the moisture contents and bulk densities of the calibration standards. Calibration curves for the density and moisture probes are recommended on the basis of data obtained in this study and in previous work at Oklahoma State University.

Density Probe Data

According to theory, the relationship between mass density and count ratio C_r is approximately linear on a semi-logrithmic plot for values in the range of normal soil mass densities. Density data obtained in this study have been plotted with average count ratio as the ordinate on the log scale and wet density in pounds per cubic foot as the abscissa on an arithmetic scale. A least squares regression analysis was utilized to determine the equation of the linear curve.

Figure 5.1 illustrates the curves obtained for the three soils and the Troxler calibration curve for Portland cement concrete. The data indicate four separate relationships exist, with the Troxler calibration curve near the middle of the data.

The sand data were interesting because of their close fit to a linear curve. As previously discussed, the standards had definite wet density profiles due to moisture migration. The instrument responses

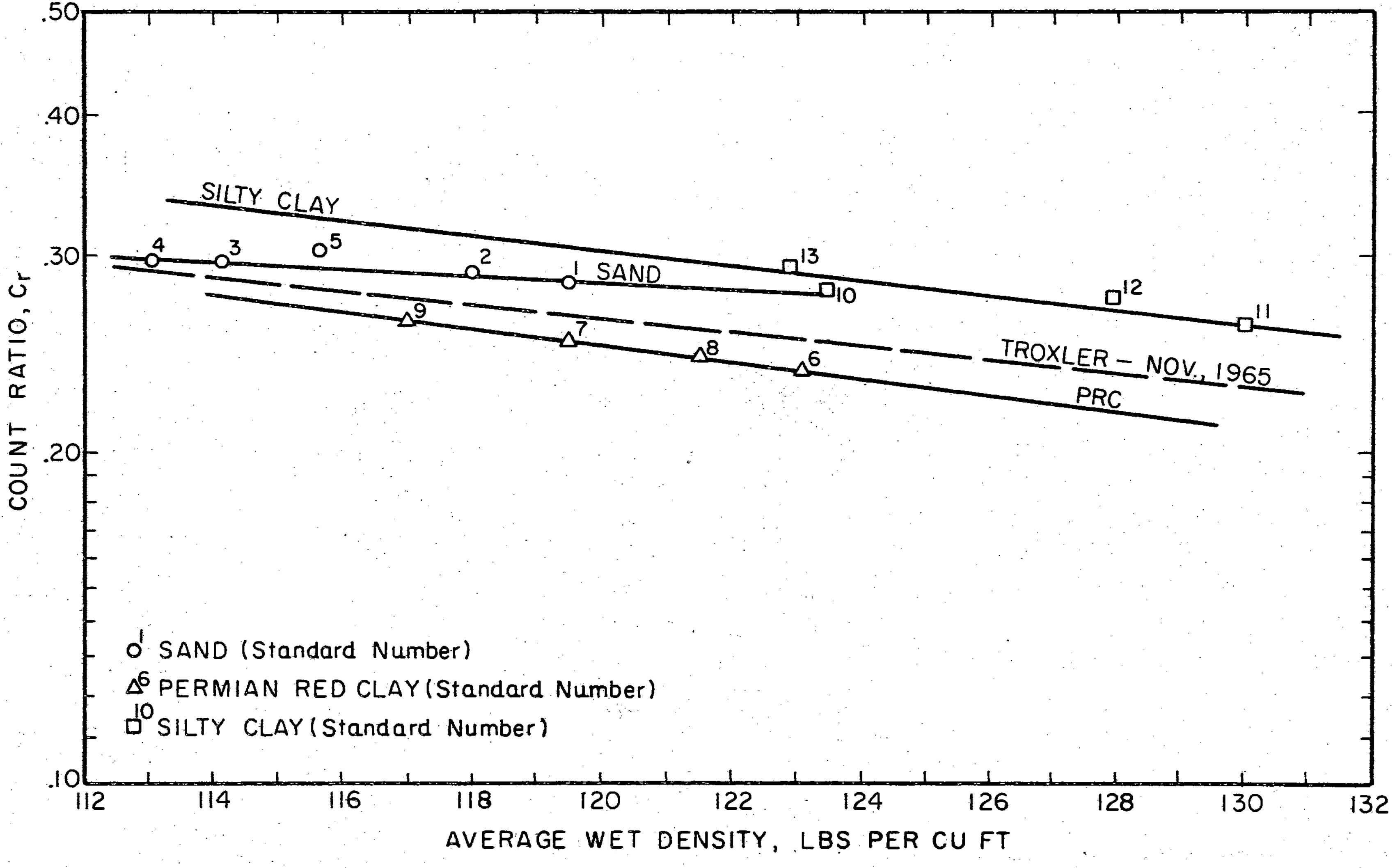


Figure 5.1. Density Calibration Curves

for all valid levels were averaged to determine the count ratio for the average bulk density of the standards. Even though the density response was not constant at all levels, the average count ratio still produced the linear curve with minimal scatter.

This averaging approach was used for the PRC and silty clay to maintain a consistent procedure. The experimental data produced quite favorable results. The PRC standards fit the linear curve theory excellently for all four points. The silty clay has more data scatter but still produced good results. Table 5.1 lists the equations for the four density calibration curves.

Figure 5.2 incorporates the previous experimental data of LeFevre and Manke with the data obtained in this study. LeFevre and Manke proposed a band width type calibration, as shown by the two heavy black lines. They theorized the band width would be narrowed by additional research. Subsequent data for the PRC falls within this range. In fact, it correlates quite well with previous PRC standards constructed at Oklahoma State University. Therefore, it is believed that the PRC density curve is valid for this particular density probe. However, the silty clay and sand are outside the band. This indicates that perhaps the band approach is not satisfactory with new experimental results diverging from a median calibration curve. Separate density curves for individual soil types are recommended on this basis.

Moisture Probe Data

Figure 5.3 shows the moisture probe response for the series of standards as well as the Troxler calibration curve. These curves are arithmetic plots with count ratio as the ordinate and volumetric water

DENSITY:	
Sand	Log Percent Standard = 1.79890 - .00286D*
PRC	Log Percent Standard = 2.22390 - .00689D
Silty Clay	Log Percent Standard = 2.04927 - .00485D
Troxler-1965	Log Percent Standard = 2.17815 - .00650D
Manke-LeFevre-1966	Log Percent Standard = 2.08490 - .00570D
MOISTURE:	
Sand	Percent Standard = 26.04 + 2.96W**
PRC	Percent Standard = 20.16 + 2.80W
Silty Clay	Percent Standard = 9.37 + 3.21W
Troxler-1965	Percent Standard = 6.69 + 3.43W
Manke-LeFevre-1966	Percent Standard = 2.20 + 4.11W
PRC-Silty Clay	Percent Standard = 8.20 + 3.35W

* D = wet density in lb per cu ft

** W = volumetric water content in lb per cu ft

TABLE 5.1. CALIBRATION CURVE EQUATIONS

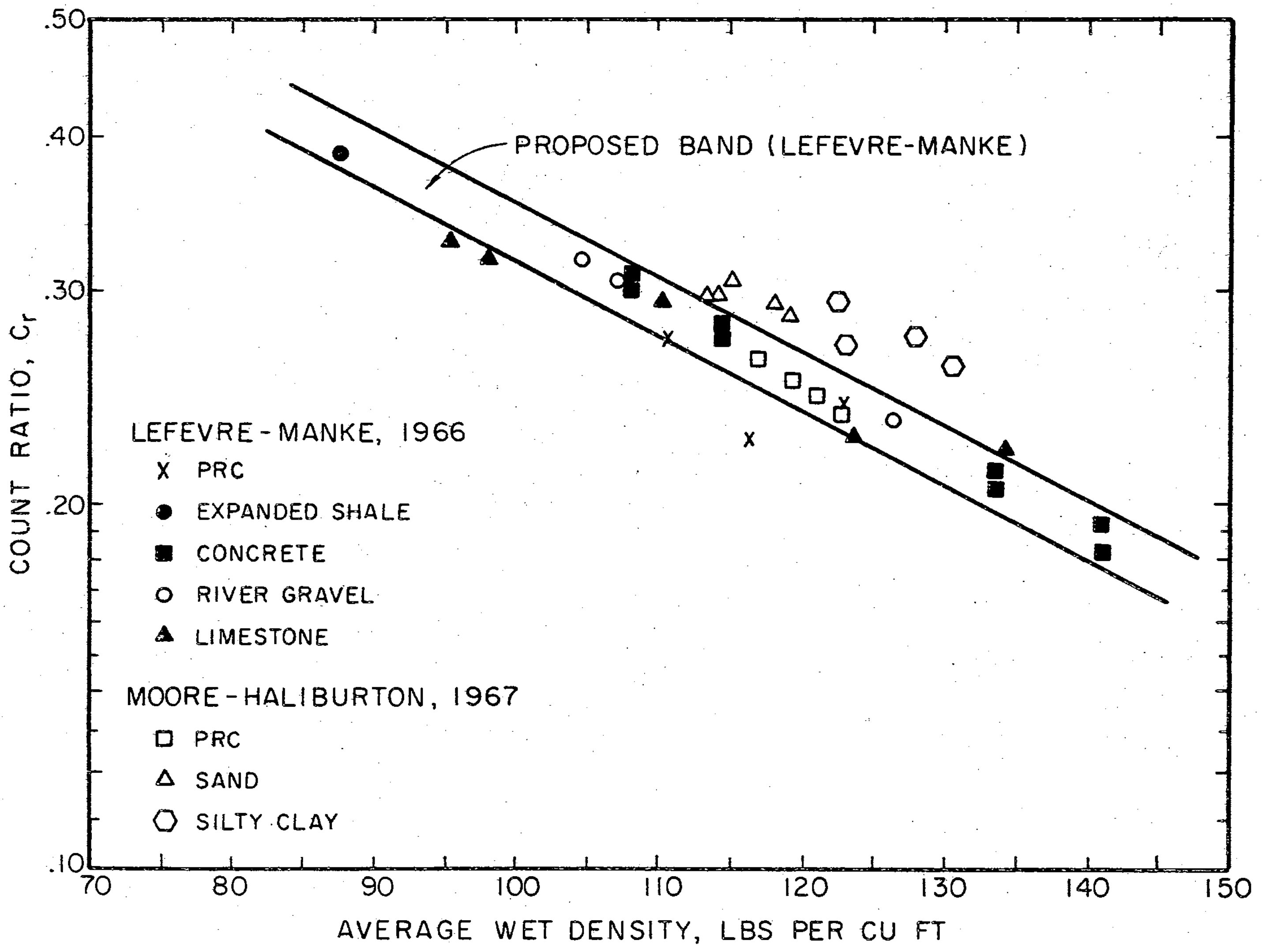


Figure 5.2. Combined Density Calibration Data

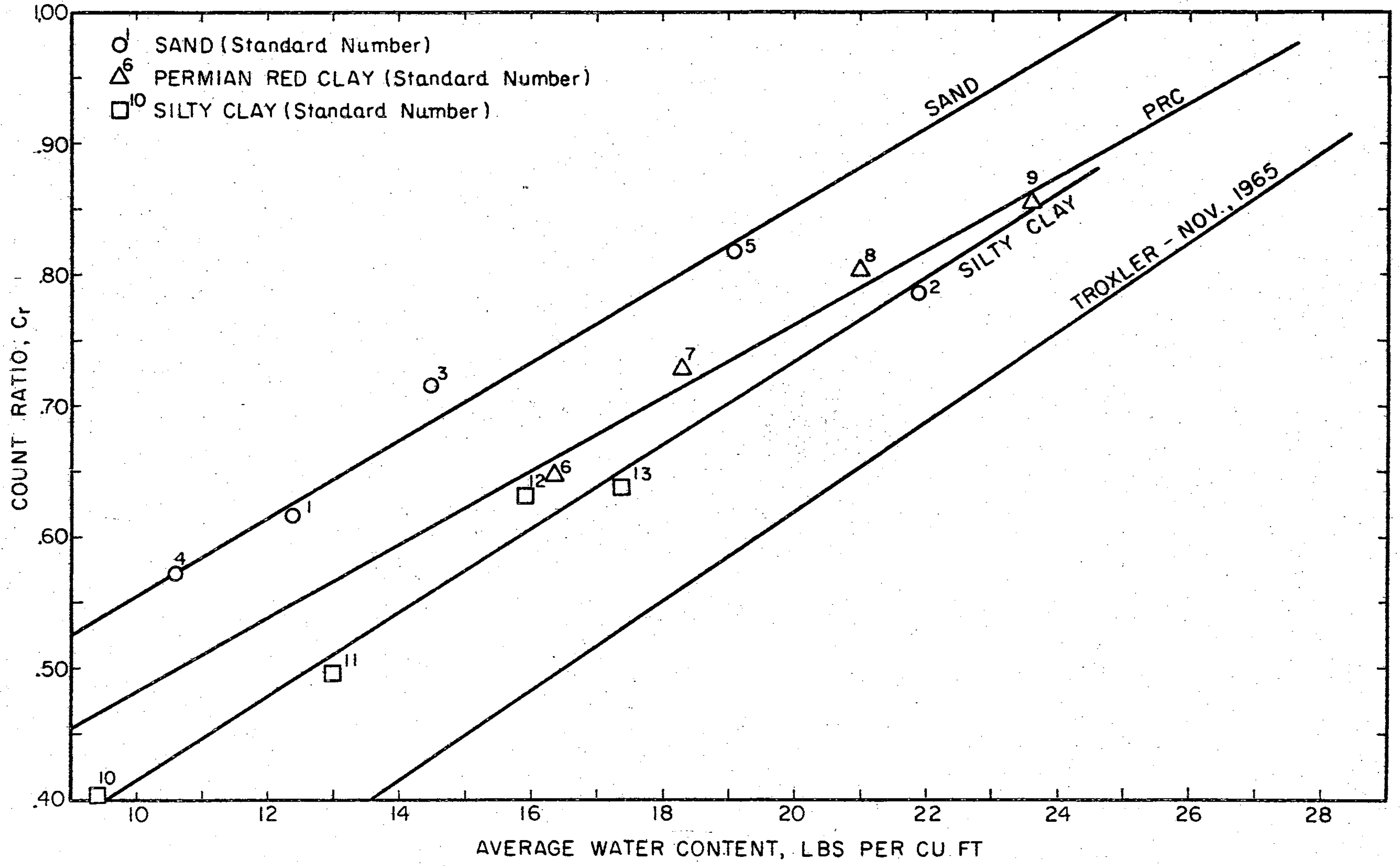


Figure 5.3. Moisture Calibration Curves

content in lb per cu ft as the abscissa. In the range of normal soil moisture content the curves should be linear.

The moisture probe data for all standards was computed on the basis of average probe response as previously discussed. The sand presented considerable difficulty in this respect. The average moisture content from the soil samples was calculated and plotted against average moisture probe response. This average moisture content was always less than the design value. The sand standards, with the elimination of Standard No. 2, produced a linear curve with a small amount of scatter. Standard No. 2 had considerable air gap between the access tube and the soil mass. This was caused by an attempt to remove sand from the cored hole with an auger section. Consequently, the water migrated to air space around the access tube in the saturated zone. The concentrated moisture region produced an erroneous gage response.

The PRC and silty clay data also produced linear curves which indicate separate curves for the three soils. The slope of the calibration curves are somewhat similar to the Troxler curve, as seen in Table 5.1. A general observation of the data indicates it would be feasible to combine the data for the cohesive soils and develop a moisture curve suitable for use in clays and silty clays.

Figure 5.4 is a composite plot of all moisture data including that presented by LeFevre and Manke. The additional data indicate the LeFevre-Manke calibration curve should be reduced in slope. It is thought that one curve for the moisture probe is a distinct possibility.

It was mentioned previously that Standard No. 10 was compacted at a low moisture content which would permit the sphere of influence of

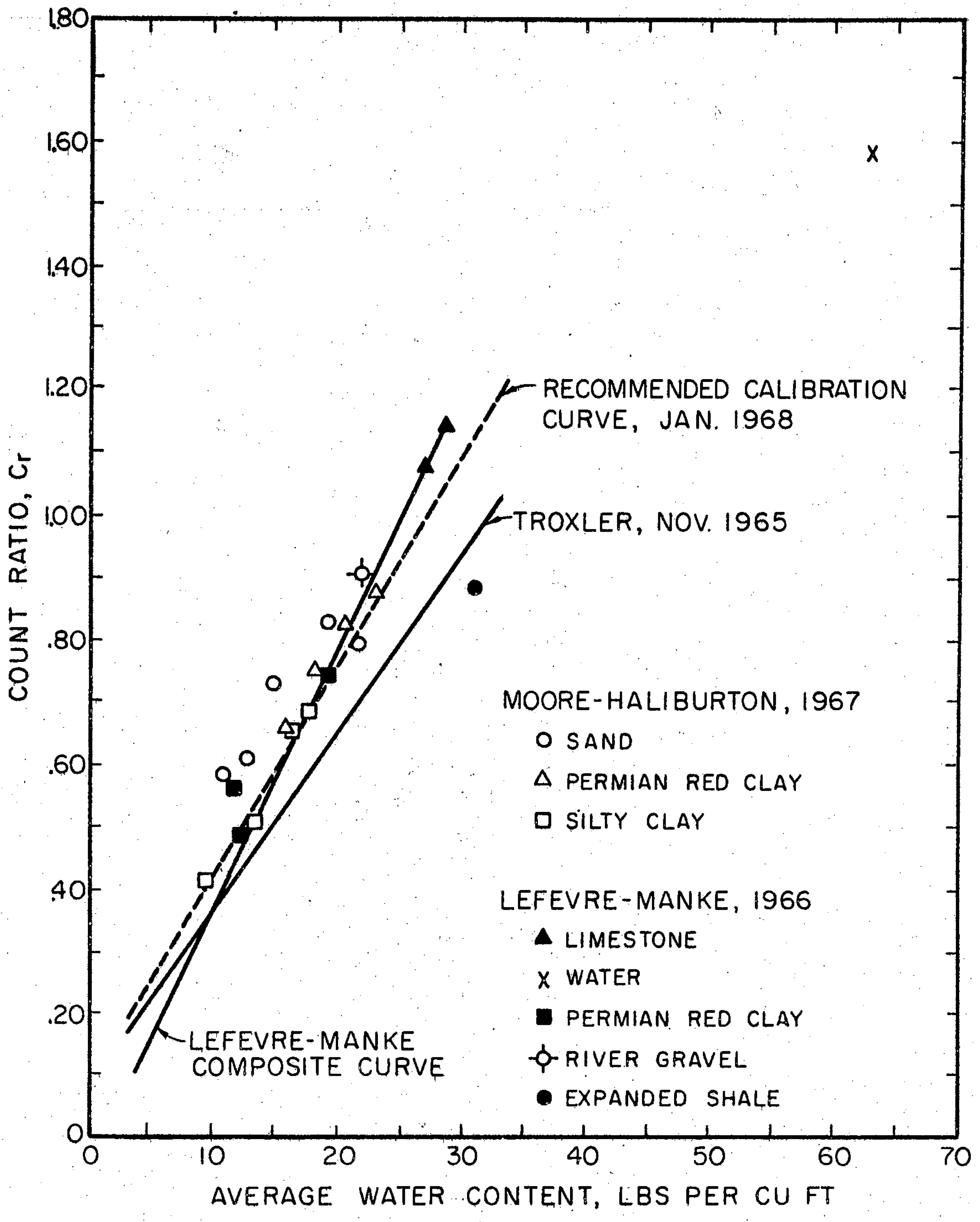


Figure 5.4. Combined Moisture Calibration Data

the moisture probe to extend outside the container. If this occurred it was believed a rapid decrease in C_r would be evident. However, the sharp decrease was not noted. In fact the point is considered quite valid for the calibration procedure. Evidently, the moisture content selected was not dry enough to produce the change anticipated in moisture probe response.

Evaluation of Data

This study has indicated possible solutions for the calibration problem. The density probe is quite "insensitive", but the calibration curve for the device is not. A small deviation of C_r will produce a large change in wet density. The scatter of density calibration curves will put a premium on accurate judgement in field data evaluation. In this respect, the PRC curve is valid for the reasons already discussed. It is quite similar to the median curve (middle of the proposed band) recommended by LeFevre and Manke. Therefore, for the present time, it is believed that the PRC curve should be used for reduction of all SMV data. This recommendation is subject to revision upon further experimentation with other soils.

A median moisture curve is a definite recommendation. The silty clay and PRC data from this study should be combined and one curve used for all soils encountered. As stated previously this curve will not differ from LeFevre and Manke's median curve in the range of normal subgrade moisture contents. The flatter slope will influence the data evaluation only in extremely wet or dry soils. This curve is also shown in Fig 5.4. It is believed that for Oklahoma soils this curve is more nearly accurate than the factory calibration curve.

Standard No.	Material	Design Criteria		wet (pcf)	dry (pcf)	$\hat{w}$ (%)	Volumetric Moisture Content (pcf)	C_r Moisture	C_r Density
1	Sand	e=.67	w=20%	119.50	107.10	11.58	12.40	.614	.284
2	"	e=.67	w=25%	118.09	96.20	22.75	21.89	.786	.289
3	"	e=.67	w=22%	114.21	99.73	14.52	14.48	.715	.295
4	"	e=.67	w=18%	113.14	102.53	10.35	10.61	.571	.295
5	"	e=.67	w=24%	115.73	96.62	19.78	19.11	.817	.302
6	PRC	γ_d=117	w=16%	123.15	106.83	15.28	16.32	.646	.237
7	"	γ_d=110	w=19%	119.54	101.15	18.18	18.39	.729	.251
8	"	γ_d=103	w=22%	121.44	100.41	20.94	21.03	.801	.244
9	"	γ_d=96	w=25%	117.09	93.47	25.27	23.62	.853	.262
10	Silty Clay	γ_d=119	w=9%	123.54	114.10	8.27	9.44	.401	.270
11	"	γ_d=112	w=12%	130.72	117.71	11.05	13.00	.495	.258
12	"	γ_d=106	w=15%	128.08	112.11	14.24	15.96	.631	.276
13	"	γ_d=100	w=18%	123.00	105.59	16.49	17.41	.636	.290

TABLE 5.2. DATA SUMMARY SHEET

A data summary for all standards is shown in Table 5.2. Results of chemical analyses on the three calibration soils are given in Appendix B. No significant amounts of either gamma or neutron absorbers were found in any of the three soils. Some other explanation must exist for the different calibration curves produced by each soil type.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

From the data presented in Chapters IV and V, the following conclusions may be drawn:

1. A suitable method for preparing cohesive soil standards has been determined. The equipment required is found in most materials laboratories or is readily available. Furthermore, the soil standards are dual purpose. They can be utilized to calibrate both nuclear density and moisture depth probes.

2. For present SMV research, a median calibration curve for density is satisfactory. However, the magnitude of separation in calibration curves with respect to soil type is such that this approach is not the final solution. Indications are given that separate calibration curves will be required for proper field data evaluation. The Band approach does not appear to be valid because of diverging data responses.

3. A median calibration curve for the moisture probe is recommended for field data evaluation. Although the different soils indicated that each had a separate calibration curve, the variance was not large. For moisture probe use to be practical, one curve is possibly the only solution. However, the curve must be determined from actual soil-water systems.

With respect to further research, the following recommendations are made:

1. A wider range of soils should be studied with regard to soil calibration, particularly as they effect density gage response.

2. Attention should be given to correlation of physical, mineral, and chemical properties with gage response, especially for density gages. Particular emphasis should be given to correlating the results from standard engineering tests. It is felt that "mathematical model" correlation, even if all variables are ever correctly considered, will not be accepted by field engineers. Chemical property correlation will probably be too expensive and time-consuming.

3. The processing phase for cohesive soils is the most time-consuming part of the calibration process. This procedure should be made more efficient, if possible.

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APPENDIX A

NUCLEAR EQUIPMENT

APPENDIX A

Nuclear Equipment

Scaler: Troxler Model 200-B
Serial Number 256

Density Equipment: ST-DD-2 Depth Density Gage, Shield and Standard Model S-7, Serial Number 77 with Depth Density Probe Model 504, Serial Number 23, using a 3 millicurie radium-226 source, Serial Number R-3-15

Moisture Equipment: SY-SM-1 Depth Moisture Gage, Shield and Standard Model S-5, Serial Number 407, with a Depth Moisture Probe Model 104, Serial Number G-19953, using a 3 millicurie radium 226-beryllium source, Serial Number N-3-149.

APPENDIX B

CHEMICAL ANALYSIS

APPENDIX B

Chemical Analysis

Chemical Analysis by Andrew S. McCreath and Son, Inc. Analytical and Consulting Chemists, Harrisburg, Pennsylvania.

	Sand	PRC	Silty Clay
Calcium	0.086%	0.73%	0.29%
Iron	0.28%	2.54%	1.37%
Cadmium	*	*	*
Sodium	0.085%	0.58%	0.55%
Potassium	0.20%	1.01%	0.87%
Lithium	0.005%	0.002%	*
Boron	0.005-2.05%	**	**
Beryllium	*	*	*

* less than 0.001%

** less than 0.005%

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Master of Science

Thesis: A CALIBRATION PROCEDURE FOR NUCLEAR DEPTH PROBES WITH OKLAHOMA SOILS

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