

CHANGE LETTER

MATERIALS TESTING MANUAL	CHANGE LETTER NO. 33
<p>SUBJECT:</p> <p>Title Page; Table of Contents; Series 100 Cover Sheet; Series 400 Cover Sheet; Series 700 Cover Sheet; Arizona Test Methods 104e, 105f, 415d, 424d, 733b, and 736b.</p>	<p>EFFECTIVE DATE:</p> <p style="text-align: center;">December 4, 2014</p>

SUMMARY:

NOTE: Unless otherwise specified, changes issued under this Change Letter are effective for projects with a bid opening date on or after December 4, 2014. Retain items removed from the Materials Testing Manual under this change letter for use as necessary on projects with a bid opening date prior to December 4, 2014.

1. **TITLE PAGE** - The Title Page has been revised to show the latest Change Letter number and revision date. Please replace the existing Title Page with the attached.
2. **TABLE OF CONTENTS** - The Table of Contents has been revised to reflect the changes made in this Change Letter. Please replace the existing Table of Contents with the attached.
3. The following items have been revised to reflect the changes made in this Change Letter. Please replace the existing items with the attached.

Series 100 Cover Sheet – “SAMPLING”

Series 400 Cover Sheet - “BITUMINOUS MIXTURES”

Series 700 Cover Sheet - “CHEMICAL AND SPECIALTY”

4. The following test methods have been revised. Please replace the existing test methods with the attached.

Arizona Test Method 104e- “SAMPLING BITUMINOUS MIXTURES”

Arizona Test Method 105f- “SAMPLING SOILS AND AGGREGATES”

MATERIALS TESTING MANUAL

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Arizona Test Method 415d- "BULK SPECIFIC GRAVITY AND BULK DENSITY OF COMPACTED BITUMINOUS MIXTURES"

Arizona Test Method 424d- "DETERMINATION OF AIR VOIDS IN COMPACTED BITUMINOUS MIXTURES"

Arizona Test Method 733b - "SULFATE IN SOILS"

Arizona Test Method 736b - "CHLORIDE IN SOILS"



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Attachments

MATERIALS
TESTING MANUAL
SAMPLING AND TESTING PROCEDURES



PREPARED BY:
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INTERMODAL TRANSPORTATION DIVISION
MATERIALS GROUP

REVISED TO CHANGE LETTER NO. 33
(December 4, 2014)

MATERIALS TESTING MANUAL

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** The above Arizona Test Methods, and also commonly used AASHTO and ASTM procedures and specifications are shown on Series 500 Cover Sheet (July 15, 2005).

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(January 17, 2014)

- Appendix A - Arizona Department of Transportation Local
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- Appendix B - Final Certification of Materials for Consultant
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- Appendix D - Code of Federal Regulations (23 CFR 637, Subpart B)
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APPENDIX A1	Rounding Procedure (July 15, 2005)
APPENDIX A2	Metric Guide (July 15, 2005)
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SERIES 100

SAMPLING

The following methods shall be performed in accordance with the respective designation:

ARIZONA TEST METHODS:

<u>TITLE</u>	<u>DESIGNATION</u>
Sampling Bituminous Materials.....	ARIZ 103a
Sampling Bituminous Mixtures.....	ARIZ 104e
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Note: Sampling of crumb rubber is performed in accordance with Arizona Test Method 714.

AASHTO TEST METHODS:

<u>TITLE</u>	<u>DESIGNATION</u>
Sampling Freshly Mixed Concrete	R 60
Sampling and Amount of Testing of Hydraulic Cement	T 127
Reducing Samples of Aggregate to Testing Size.....	T 248

NOTE: It shall be assured that the appropriate methods as given in the project requirements are being adhered to.

NOTE: Refer to Series 900, "Materials Quality Assurance Program", of the Materials Testing Manual for current guidelines on sampling of materials for acceptance, independent assurance, and correlation testing.

SERIES 400
BITUMINOUS MIXTURES

The following test methods shall be performed in accordance with the respective designation:

ARIZONA TEST METHODS:

<u>TITLE</u>	<u>DESIGNATION</u>
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ARIZONA TEST METHODS: (continued)

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AASHTO TEST METHODS:

<u>TITLE</u>	<u>DESIGNATION</u>
Quantitative Extraction of Asphalt Binder from Hot Mix Asphalt (HMA).....	T 164
Preparing and Determining the Density of Asphalt Mixture Specimens by Means of the Superpave Gyrotory Compactor.....	T 312

NOTE: It shall be assured that the appropriate test methods as given in the project requirements are being adhered to.

SERIES 700
CHEMICAL AND SPECIALTY

The following test methods shall be performed in accordance with the respective designation:

ARIZONA TEST METHODS:

<u>TITLE</u>	<u>DESIGNATION</u>
Testing of Paint, Varnish, Lacquer, and Related Material.....	ARIZ 702a
Sampling and Sieving of Crumb Rubber.....	ARIZ 714b
Heating and Drying Materials in Microwave Oven.....	ARIZ 719c
Tensile Proof Dowel Test.....	ARIZ 725a
Reflectance, Dry Opacity, and Yellowness Index of Traffic Paint.....	ARIZ 726a
Chloride in Hardened Concrete.....	ARIZ 727a
Exchangeable Sodium in Topsoil.....	ARIZ 729b
Calcium Carbonate in Topsoil (Neutralization Potential of Topsoil).....	ARIZ 732a
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ARIZONA TEST METHODS: (continued)

<u>TITLE</u>	<u>DESIGNATION</u>
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Rock Salt in Crash Barrel Sand.....	ARIZ 744

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Water Permeability of Geotextiles by Permittivity		D 4491
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NOTE: It shall be assured that the appropriate test methods as given in the project requirements are being adhered to.

SAMPLING BITUMINOUS MIXTURES

(An Arizona Method)

1. SCOPE

- 1.1 This procedure describes the methods which are to be used when sampling bituminous mixtures.
- 1.2 Sampling bituminous mixtures by this procedure may involve hazardous material, operations, or equipment. This procedure does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of any regulatory limitations prior to use.

2. SAMPLING PLANT-MIXED BITUMINOUS MIXTURES

- 2.1 Asphaltic Concrete and Asphaltic Concrete (Asphalt-Rubber) shall be sampled as described in Subsections 2.4 through 2.6.
- 2.2 Asphaltic Concrete Friction Course and Asphaltic Concrete Friction Course (Asphalt-Rubber) shall be sampled as described in Subsection 2.7.
- 2.3 Minimum sample sizes shall be as follows:
 - 2.3.1 For Asphaltic Concrete Friction Course mixtures or Asphaltic Concrete Friction Course (Asphalt-Rubber) mixtures, 50 pounds.
 - 2.3.2 For Asphaltic Concrete mixtures or Asphaltic Concrete (Asphalt-Rubber) mixtures designed with Marshall design procedures, 75 pounds.
 - 2.3.3 For Asphaltic Concrete mixtures designed with Gyrotory design procedures, 130 pounds.
- 2.4 A 4 foot x 1 foot x 1/16 inch steel plate, which has been prepared with a 1/8 inch hole at each corner of one end and a sufficient length of wire tied through each hole to form a loop approximately 4 feet in length, shall be placed on the roadway just ahead of the laydown machine. Except for wider mats when a sample is being taken from the middle of the mat, the

steel plate is placed so that the end with the wire is approximately one foot in from the right or left edge of the mat being laid. The sampling should be alternated between the right and left edges, and on wider mats also the middle when practical. The wire attached to the end of the plate shall be held to the ground to allow the laydown machine to pass over the plate and wire.

2.5 After the laydown machine has passed, locate the plate by raising the wire.

2.6 The sample shall be taken from the plate using a flat square point shovel. The sample shall consist of the full depth of material for one shovel width from the center portion of the plate over its entire length. Material covering the entire plate shall not be taken. A single pass of the shovel shall be made, moving along the surface of the plate until the shovel is full. Carefully deposit the bituminous mixture into a 5-gallon bucket, or other suitable container. Material which has sloughed into the resultant trench shall not be obtained. At the next undisturbed area of material on the plate, repeat shoveling and placing the material into the container. If necessary, additional material may be obtained by using an additional plate(s) in the immediate vicinity and combining all material. The use of an additional plate(s) cannot be used in lieu of splitting.

Note: As an alternate to obtaining the sample from the plate using a shovel as described above, a rectangular metal template ("cookie cutter") and metal plate of sufficient size may be used to sample the bituminous mixture. The metal template and plate shall be of sufficient size so that the desired amount of material is obtained by a single use of the template and plate at any one location. The metal plate shall be prepared with a wire(s) of sufficient length attached to each corner on one side of the metal plate (the short side when the plate is not square) so the metal plate may be located by raising the wire(s) after the laydown machine has passed. The metal plate shall be placed on the roadway at the location where the sample is to be taken, just ahead of the laydown machine. If the metal template is not square, it shall be placed on the roadway so that the longest side is in a transverse direction across the roadway. The wire(s) shall be held to the ground to allow the laydown machine to pass over the plate and wire(s). After the laydown machine has passed, locate the plate by raising the wire(s). The template is pressed through the bituminous mixture until it rests squarely upon the plate. The entire amount of

bituminous mixture is removed from the interior of the template and carefully placed into a 5-gallon bucket, or other suitable container. Obtaining multiple samples cannot be used in lieu of splitting.

- 2.7 When sampling Asphaltic Concrete Friction Course or Asphaltic Concrete Friction Course (Asphalt-Rubber), an adequate amount of material shall be taken from the truck at the mixing plant and placed into a 5-gallon bucket, or other suitable container. The sample shall be taken from at least 3 random locations, approximately 12" below the surface, within five minutes from the time the loading of the truck is completed.
- 2.8 Material that is to be tested immediately after it has been sampled shall be protected to avoid heat loss while it is being transported to the laboratory.

3. SAMPLING FINISHED BITUMINOUS PAVEMENT

- 3.1 Samples of bituminous mixture from finished pavement shall be taken through the complete thickness of the pavement or lift, in such a manner which causes minimum disturbance to the sample.
- 3.2 If coring apparatus is used, the coring bit shall be subjected to enough vertical pressure to penetrate the pavement without causing damage to equipment or disturbance of the sample. Minimum core diameter shall be 4 inches.
- 3.3 If coring equipment is not available, the sample may be taken with the use of a saw, pick, jackhammer, or other suitable means if a suitable specimen can be obtained for the intended testing.
- 3.4 All samples shall be handled carefully so that they maintain their briquette form. The samples shall be transported on a relatively flat surface, and adequately protected to preserve their shape and to prevent damage.
- 3.5 The use of ice may be found helpful in obtaining and/or preserving the condition of the specimen.

**4. SAMPLING MISCELLANEOUS PLACEMENT
OF BITUMINOUS MIXTURES**

- 4.1 The sampling of bituminous mixture used in paving slopes, median islands and other miscellaneous placement shall be accomplished by taking an adequate amount of material from the hauling vehicle by random shovelfuls.

5. SAMPLE IDENTIFICATION

- 5.1 Each sample shall be identified by an accompanying sample ticket. Sample tickets shall be filled out as required to provide necessary information. The remarks area of the sample ticket shall be used as necessary to provide additional information, including the phone number of an individual who can be contacted regarding the sample.
- 5.2 The source of the sample shall be the "original source" of the material, as indicated on the sample ticket.
- 5.3 An example of a completed sample ticket used by ADOT for construction projects is shown in Figure 1. Commonly used codes for filling out the sample ticket are shown on the back side of the sample ticket (see Figure 2).
- 5.4 The sample ticket consists of three copies. The center copy is kept by the person submitting the sample, the original copy is included inside the sample container, and the third copy is attached to the sample container. When filling out sample tickets, make certain information is clear and easily read on all three copies.

**PRESS FIRMLY USING A
BALL-POINT PEN WHILE FILLING OUT FORM**

ARIZONA DEPARTMENT OF TRANSPORTATION
SAMPLE TABULATION
SOIL, AGGREGATE, & BITUMINOUS MIXES

ADOT 44-9346 R07/14

USE CAPITAL LETTERS

LAB NUMBER			ORG NUMBER	MATL	TYPE	PUR-POSE	TEST LAB	SIZE	SIZE %
			9999	AC	34	A	P		
TEST NO.	LOT OR SUFFIX	SAMPLED BY (FIRST & LAST NAME)			MO	DAY	YEAR	TIME	
3	8	Bob Tester			09	15	14	10:30	
SAMPLED FROM					LIFT NO.	RDWY	STATION		
Roadway					1	EB	670+50		
ORIGINAL SOURCE			PROJECT ENGINEER / SUPERVISOR		PROJECT NUMBER		TRACS NUMBER		
XYZ COMMERCIAL			F. Bossy		F-099-9(9)		H999909C		
REMARKS									
EXAMPLE									
CONTACT PHONE NO. - 999-999-9999									

FIGURE 1

- Roadway Codes:**
 NB NORTHBOUND
 SB SOUTHBOUND
 EB EASTBOUND
 WB WESTBOUND
 RA RAMP A
 RB RAMP B
 RC RAMP C
 RD RAMP D
 FR FRONTAGE ROAD
 XR CROSS ROAD
 DE DETOUR
- Purpose Codes:**
 A ACCEPTANCE
 C CORRELATION
 P INDEPENDENT ASSURANCE
 I INFORMATIONAL
- Testing Lab Codes:**
 C CENTRAL LAB
 R REGIONAL LAB
 P PROJECT LAB

- Bituminous Mixes:**
 AC ASPHALTIC CONCRETE
 MA MINERAL AGGREGATE
 34 3/4" ASPHALTIC CONCRETE
 34F 3/4" FINE BAND 417 AC
 34K 3/4" COARSE BAND 417 AC
 12 1/2" ASPHALTIC CONCRETE
 12F 1/2" FINE BAND 417 AC
 12K 1/2" COARSE BAND 417 AC
 BM BASE MIX
 FC ACFC
 RD ASPHALT - RUBBER ASPHALTIC CONCRETE
 RF ASPHALT - RUBBER A.C. FRICTION COURSE
 409MI MISC. STRUCTURAL
 409SP MISC. STRUCTURAL (Special Mix)

- Soils and Aggregates:**
 AB AGGREGATE BASE (CLASS 1, 2, or 3)
 AS AGGREGATE SUBBASE (CLASS 4, 5, or 6)
 CM COVER MATERIAL (CLASS 1 or 2)
 CA COARSE AGGREGATE
 SG SUBGRADE
 BW BORROW
 BL BLOTTER MATERIAL
 DG DECOMPOSED GRANITE
 BF BACKFILL*
 *AP ALUMINUM PIPE
 *CP CONCRETE PIPE
 *MP METAL PIPE
 *PP PLASTIC PIPE
 *PV PVC PIPE
 *SL SLURRY
 *TR TRENCH BACKFILL

- Other Codes:**
 RP RECLAIMED ASPHALT PAVEMENT
 C COARSE
 F FINE
 O OTHER
 GR GRANULATED RUBBER
 CB CRASH BARREL SAND
 RR RIP RAP

Not all codes used by FAST are listed above. (See Appendix C of Series 900 of the ADOT Materials Testing Manual for a listing of other codes used by FAST. FAST may revise codes, delete codes, or add codes at various times. Individuals must assure that they are utilizing the current FAST codes.)

FIGURE 2

SAMPLING SOILS AND AGGREGATES

(An Arizona Method)

1. SCOPE

- 1.1 This method describes the methods which are to be used when sampling soils and aggregates.
- 1.2 Sampling is equally as important as the testing, and the individual doing the sampling shall use every precaution to obtain samples that will be representative of the materials being sampled.
- 1.3 This test method may involve hazardous material, operations, or equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of any regulatory limitations prior to use.
- 1.4 Table 1 shall be used to determine minimum sample weights based on the size of aggregate. The amount of material required may be greater depending on the tests that are to be performed on the material.

Table 1 Minimum Sample Sizes		
Nominal Maximum Aggregate Size *	Sample Mass	
	lbs	kg
Fine Aggregate		
#8	22	10
#4	22	10
Coarse Aggregate		
3/8"	22	10
1/2"	35	15
3/4"	55	25
1"	110	50
1-1/2"	165	75
2"	220	100
2-1/2"	275	125
3"	330	150
* The smallest sieve opening through which the entire amount of material, by specification, is permitted to pass.		

2. SAMPLING FROM STOCKPILES

- 2.1 In sampling materials from stockpiles it is difficult to ensure unbiased samples, due to the segregation which often occurs when the material is stockpiled with coarser particles rolling to the outside base of the pile. If power equipment is available then it would be advantageous to enlist the use of that equipment to develop a separate, small sampling pile composed of materials drawn from various levels and locations in the main stockpile. Once a small sampling pile has been established then a sample shall be taken from that pile by taking several increments and combining.
- 2.2 The stockpile may also be sampled by placing a wood or metal shield upslope from the point of sampling to prevent loose aggregate from sliding down into the sampling area. Remove approximately 3 to 6 inches of material from the sampling area. Utilizing a square point shovel, take a sample near the top, at the middle and near the bottom of the stockpile. The sample taken at each location shall be one shovelful of material. Repeat this operation at the sampling locations as shown in Figure 1, and combine all samples taken from the stockpile.

3. SAMPLING FROM BINS

- 3.1 A sample shall be taken by passing a sampling device through the entire cross-section of the flow of material as it is being discharged (see Figures 2 and 3). Sufficient material shall be allowed to pass at the beginning of discharge to ensure uniformity of material before the sample is taken. Repeat sampling procedure as necessary until the desired amount of material from each bin is obtained. Material from each bin shall be properly identified.

4. SAMPLING FROM A CONVEYOR BELT

- 4.1 Sampling from a conveyor belt may be performed either while the conveyor belt is running (by using a sampling device which diverts or intercepts the flow of material) or by taking a sample while the conveyor belt is stopped. The stopped belt method is also used when approving a sampling device used for sampling while the belt is running.

4.1.1 If the sample is obtained while the conveyor belt is running, samples of the aggregate shall be taken utilizing a sampling device to divert or intercept the entire flow of material in such a manner that all portions of the flow are diverted or intercepted for an equal amount of time.

4.1.2 Samples may be obtained by stopping the conveyor belt and sampling the full width of the belt utilizing a template which is shaped to the same contour of the belt. All material which is within the template area shall be removed, utilizing a brush to obtain all the fine aggregate material.

5. SAMPLING FROM A WINDROW

5.1 Figure 4 illustrates the method used to sample a windrow. At each point in the windrow where a sample is to be taken, remove sufficient material from the top of the windrow so that a representative sample can be obtained from the center of the freshly exposed top of the windrow using a square point shovel. The sample taken at each sampling location shall be one shovelful of material. Repeat the sampling as necessary, at the required number of locations in the windrow, to obtain the desired amount of material. The samples taken shall be combined.

6. SAMPLING FROM THE ROADWAY

6.1 In the case of sampling material in-place from the roadway, at least 3 samples shall be taken with a shovel at equally distributed locations across the width of the roadway. It may be necessary to use a hammer and chisel or similar tools to cut the hole in the compacted roadway. Care shall be taken to obtain all material from the hole which is dug. The samples taken shall be combined.

7. REDUCING FIELD SAMPLES TO TESTING SIZE

7.1 The reduction of samples to obtain the amount required for particular tests shall be performed in accordance with AASHTO T 248.

8. SAMPLE IDENTIFICATION

- 8.1 Each sample shall be identified by an accompanying sample ticket. Sample tickets shall be filled out as required to provide necessary information. The remarks area of the sample ticket shall be used as necessary to provide additional information, including the phone number of an individual who can be contacted regarding the sample.
- 8.2 The source of the sample shall be the “original source” of the material, as indicated on the sample ticket.
- 8.3 An example of a completed sample ticket used by ADOT for construction projects is shown in Figure 5. Commonly used codes for filling out the sample ticket are shown on the back side of the sample ticket (see Figure 6).
- 8.4 The sample ticket consists of three copies. The center copy is kept by the person submitting the sample, the original copy is included inside the sample container, and the third copy is attached to the sample container. When filling out sample tickets, make certain information is clear and easily read on all three copies.

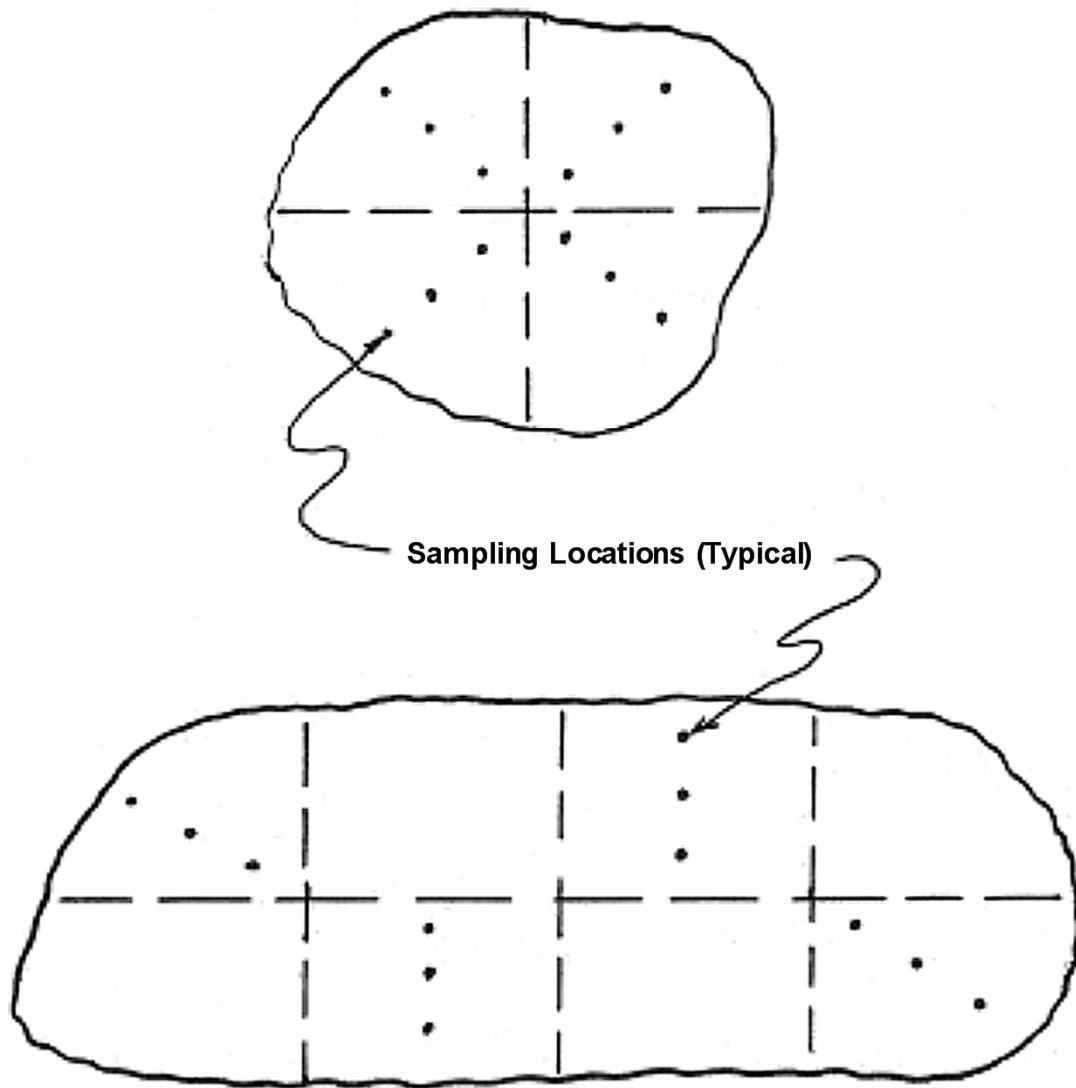
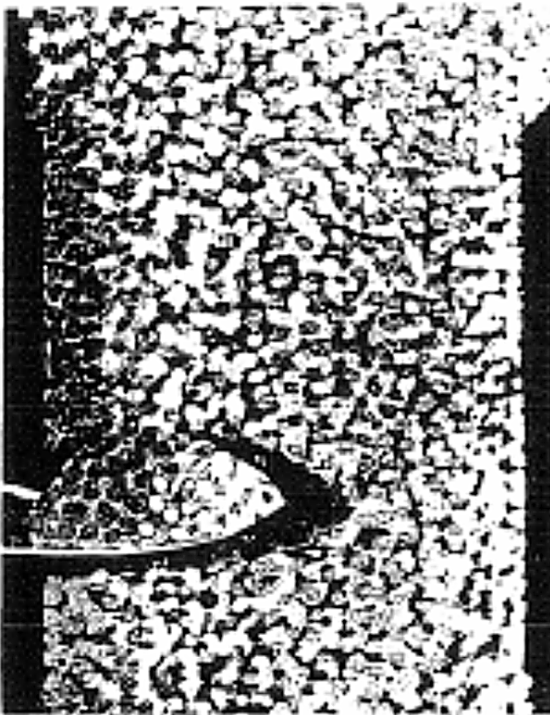


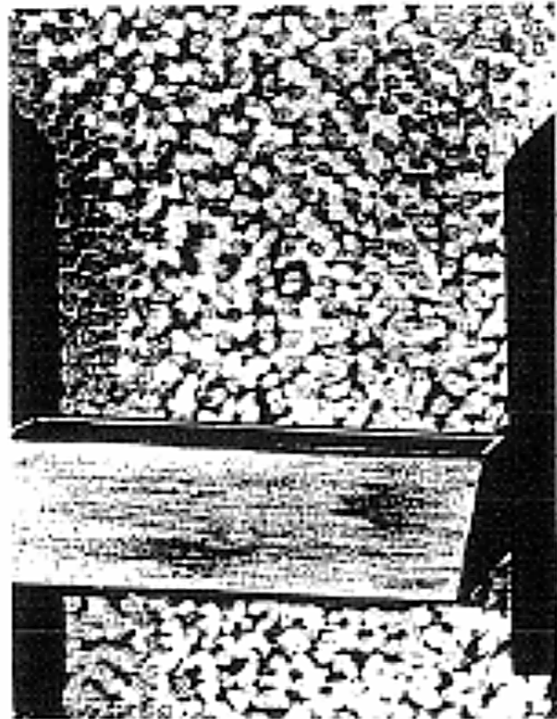
Illustration of Sampling Locations for Different Stockpile Types

FIGURE 1



WRONG

When aggregate is passed over a screen, the fines tend to drop through immediately and accumulate on one side of the hopper. A sample taken with a shovel or other small container will not be representative.

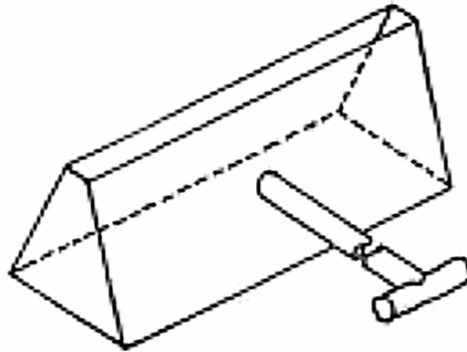


RIGHT

A sample taken by inserting the sampling device through the full flow of material will yield a representative sample. The restricted opening prevents the sampling device from filling all at once.

Illustration of Bin Sampling

FIGURE 2



Typical Bin Sampler

FIGURE 3

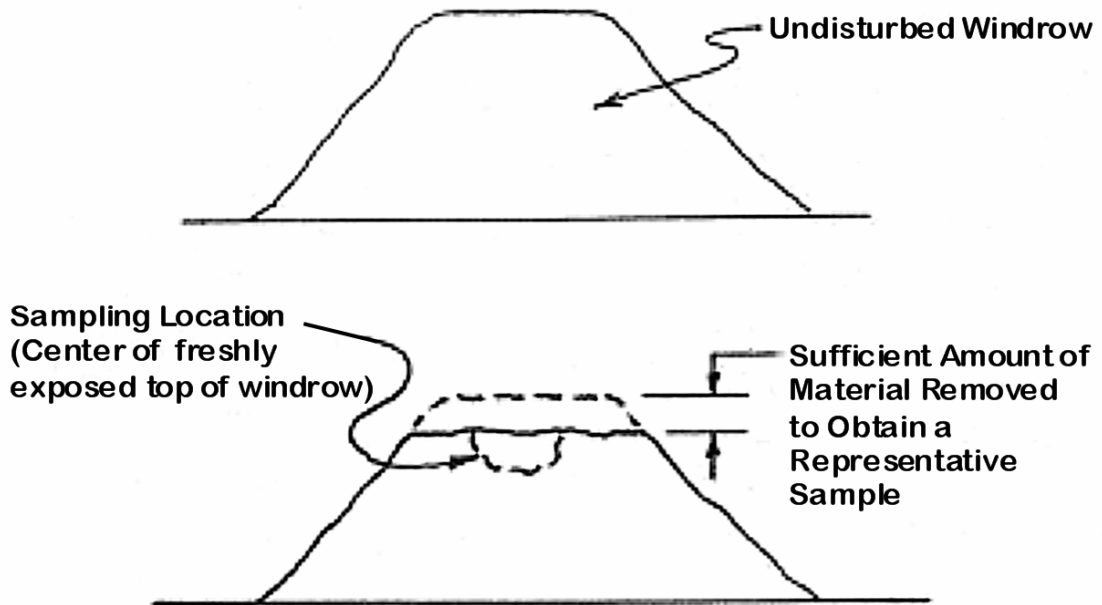


Illustration of Sampling From a Windrow

FIGURE 4

PRESS FIRMLY USING A BALL-POINT PEN WHILE FILLING OUT FORM
ARIZONA DEPARTMENT OF TRANSPORTATION
SAMPLE TABULATION
SOIL, AGGREGATE, & BITUMINOUS MIXES

ADOT		USE CAPITAL LETTERS		44-9346 R0714	
LAB NUMBER	ORG NUMBER	MATL	TYPE	PUR-POSE	TEST LAB
	9999	MA	12	A	P
TEST NO.	LOT OR SUFFIX	SAMPLED BY (FIRST & LAST NAME)		MO	DAY
		JOL DOGOOD		09	15
SAMPLED FROM		YEAR	TIME	MILITARY TIME	
STOCKPILE		14	15		
		LIFT NO.	RDWY	STATION	
ORIGINAL SOURCE	PROJECT ENGINEER / SUPERVISOR	PROJECT NUMBER	TRACS NUMBER		
XYZ Commercial	F. Bossy	F-099-9(9)	H999909C		
REMARKS					
EXAMPLE					
CONTACT PHONE NO. - 555-555-5555					

FIGURE 5

- Roadway Codes:**
 NB NORTHBOUND
 SB SOUTHBOUND
 EB EASTBOUND
 WB WESTBOUND
 RA RAMP A
 RB RAMP B
 RC RAMP C
 RD RAMP D
 FR FRONTAGE ROAD
 XR CROSS ROAD
 DE DETOUR
- Purpose Codes:**
 A ACCEPTANCE
 C CORRELATION
 P INDEPENDENT ASSURANCE
 I INFORMATIONAL
- Testing Lab Codes:**
 C CENTRAL LAB
 R REGIONAL LAB
 P PROJECT LAB
- Bituminous Mixes:**
 AC ASPHALTIC CONCRETE
 MA MINERAL AGGREGATE
 34 34" ASPHALTIC CONCRETE
 34F 3/4" FINE BAND 417 AC
 34K 3/4" COARSE BAND 417 AC
 12 1/2" ASPHALTIC CONCRETE
 12F 1/2" FINE BAND 417 AC
 12K 1/2" COARSE BAND 417 AC
 BM BASE MIX
 FC ACFC
 RD ASPHALT - RUBBER ASPHALTIC CONCRETE
 RF ASPHALT - RUBBER A.C. FRICTION COURSE
 409M MISC. STRUCTURAL
 409SP MISC. STRUCTURAL (Special Mix)
- Soils and Aggregates:**
 AB AGGREGATE BASE (CLASS 1, 2, or 3)
 AS AGGREGATE SUBBASE (CLASS 4, 5, or 6)
 CM COVER MATERIAL (CLASS 1 or 2)
 CA COARSE AGGREGATE FA FINE AGGREGATE
 SG SUBGRADE SB STRUCTURE BACKFILL
 BW BORROW NG NATURAL GROUND
 BL BLOTTER MATERIAL EM EMBANKMENT
 DG DECOMPOSED GRANITE TS TOP SOIL
 BF BACKFILL* BM BEDDING MATERIAL*
 *AP ALUMINUM PIPE
 *CP CONCRETE PIPE
 *MP METAL PIPE
 *PP PLASTIC PIPE
 *PV PVC PIPE
 *SL SLURRY
 *TR TRENCH BACKFILL
- Other Codes:**
 RP RECLAIMED ASPHALT PAVEMENT
 C COARSE
 F FINE
 O OTHER
 GR GRANULATED RUBBER
 CB CRASH BARREL SAND
 RR RIP RAP

Not all codes used by FAST are listed above. (See Appendix C of Series 900 of the ADOT Materials Testing Manual for a listing of other codes used by FAST. FAST may revise codes, delete codes, or add codes at various times. Individuals must assure that they are utilizing the current FAST codes.)

44-9346 R0714

FIGURE 6

BULK SPECIFIC GRAVITY AND BULK DENSITY OF COMPACTED BITUMINOUS MIXTURES

(A Modification of AASHTO Designation T 166 and PP 75)

1. SCOPE

- 1.1 This procedure covers the determination of bulk specific gravity and bulk density of specimens of compacted bituminous mixtures.
- 1.2 This test procedure consists of three methods for determining bulk specific gravity and bulk density. These methods are referred to as "Method A", "Method C (Rapid Test)", and "Vacuum Method".
- Note:** If it is desired to use "Method B", which is not included in this procedure, AASHTO T 166 shall be referred to. Method B should not be used with samples that contain open or interconnecting voids and/or absorb more than 2.0 percent water by volume. Method B is not acceptable for specimens that have more than six percent air voids.
- 1.3 Method A should not be used with samples that contain open or interconnecting voids and/or absorb more than 2.0 percent water by volume as determined in Subsection 7.4. If the percent water absorbed by the specimen exceeds 2.0 percent, either Method C, Vacuum Method, or AASHTO T 275 "Bulk Specific Gravity (G_{mb}) of Compacted Hot Mix Asphalt (HMA) Using Paraffin-Coated Specimens" shall be used.
- 1.4 Referee testing shall be performed in accordance with "Method C".
- 1.5 This test method involves hazardous material, operations, and equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
- 1.6 See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

2. TEST SPECIMENS

- 2.1 Test specimens may be either laboratory molded specimens or samples from an existing bituminous pavement.
- 2.2 Size of specimens - It is recommended that: (1) the diameter of cylindrically molded or cored specimens, or the length of the sides of sawed specimens, be at least four times the maximum size of the aggregate and, (2) the thickness of specimens be at least one and one half times the maximum size of the aggregate.
- 2.3 Specimens obtained from bituminous pavements shall be taken with a core drill, diamond or carborundum saw, or by other suitable means.
- 2.4 Care shall be taken to avoid distortion, bending, or cracking of specimens during and after the removal from the pavement or mold. Specimens shall be stored in a safe, cool place.
- 2.5 Test specimens shall be free from paper, tape, soil, and other foreign materials not intended to be tested as part of the specimen.
- 2.6 If desired, specimens may be separated from other pavement layers by sawing or other suitable means. Care shall be exercised to ensure that specimens are not damaged during the separation process.

3. APPARATUS

- 3.1 Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus for this test procedure shall consist of the following:
 - 3.1.1 Balance - A balance capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M 231, except the readability and sensitivity of any balance utilized shall be at least 0.1 gram. The balance shall be equipped with a suitable suspension apparatus and holder to permit weighing the specimen while suspended from the center of the balance.

- 3.1.2 Suspension Apparatus - The wire(s) suspending the holder shall be the smallest practical size to minimize any possible effects of a variable immersed length. The suspension apparatus shall be constructed to enable the container to be immersed to a depth sufficient to cover it and the test sample during weighing.
- 3.1.3 Water bath - The water bath for immersing the specimen in water while suspended under the balance shall be equipped with an overflow outlet or a clearly marked "fill line" for maintaining a constant water level. The level of the water shall be adjusted before testing each specimen, if necessary, to maintain a constant water level.
- 3.1.4 Oven - An oven capable of drying specimens at either 125 ± 5 °F (Method A) or 290 ± 10 °F (Method C).
- 3.1.5 Vacuum Drying Device (For Vacuum Method) – Conforming to the requirements of AASHTO PP 75. Automatic controls of the unit shall be calibrated by the manufacturer prior to initial use.

METHOD A

4. PROCEDURE

- 4.1 Dry the specimen to constant mass. Constant mass shall be defined as the mass at which further drying at 125 ± 5 °F does not alter the mass by more than 0.1 gram. Samples saturated with water shall initially be dried overnight at 125 ± 5 °F and then weighed at two-hour drying intervals. Recently molded laboratory samples, which have not been exposed to moisture, do not require drying.
- 4.2 Cool the specimen to room temperature at 77 ± 9 °F and record the dry mass to the nearest 0.1 gram as "A".
- 4.3 Immerse the specimen in water at 77 ± 2 °F for 4 ± 1 minutes and record the immersed mass to the nearest 0.1 gram as "C". Care shall be exercised to ensure that no trapped air bubbles exist under the specimen.
- 4.4 Remove the specimen from the water, quickly damp dry the specimen surface by blotting with a damp towel, and as quickly as possible determine and record the surface-dry mass to the nearest 0.1 gram as "B". Any water that seeps from the specimen during the surface-dry

weighing operation is considered as part of the specimen. The determination of the immersed mass and surface-dry mass of each specimen will be completed before the next specimen is submerged for its immersed mass determination.

Note: A terry cloth towel has been found to work well. Damp is considered to be when no water can be wrung from the towel.

Note: If desired, the sequence of testing operations may be changed to expedite the test results. For example, first the immersed mass, "C", can be taken, then the surface-dry mass, "B", and finally the dry mass, "A".

- 4.5 Calculate the bulk specific gravity, bulk density, and percent water absorption by volume in accordance with Section 7.

METHOD C (RAPID TEST)

5. PROCEDURE

- 5.1 This procedure can be used for testing specimens which are not required to be saved and which contain a substantial amount of moisture. Specimens obtained by methods that introduce moisture, such as wet coring or sawing, can generally be tested the same day by this method.
- 5.2 Ensure the specimen is at room temperature (77 ± 9 °F).
- 5.3 Immerse the specimen in water at 77 ± 2 °F for 4 ± 1 minutes and record the immersed mass to the nearest 0.1 gram as "C". Care shall be exercised to ensure that no trapped air bubbles exist under the specimen.
- 5.4 Remove the specimen from the water, quickly damp dry the specimen surface by blotting with a damp towel, and as quickly as possible determine and record the surface-dry mass to the nearest 0.1 gram as "B". Any water that seeps from the specimen during the surface-dry weighing operation is considered as part of the specimen. The determination of the immersed mass and surface-dry mass of each specimen will be completed before the next specimen is submerged for its immersed mass determination.

- 5.5 Place the specimen in a large, flat-bottom drying pan of known weight. Place the pan and specimen in a 290 ± 10 °F oven. Leave the specimen in the oven until it can be easily separated to the point where particles of the fine aggregate portion are not larger than 1/4 inch. During separation of material, ensure that no material is lost. Place the separated specimen in the 290 ± 10 °F oven and dry to constant mass. (Constant mass shall be determined as follows: Dry the sample for a minimum of 1 hour at 290 ± 10 °F. Record the weight of the sample to the nearest 0.1 gram. Continue drying and weighing until the weight does not change more than 0.1 gram at drying intervals of a minimum of 30 minutes.)
- 5.6 Cool the pan and specimen to room temperature at 77 ± 9 °F. Weigh the pan and specimen, subtract the mass of the pan, and record the dry mass of the specimen to the nearest 0.1 gram as "A".
- Note:** For Method C, the drying of specimens to constant weight may be accomplished in a microwave oven, as described in Arizona Test Method 719.
- 5.7 Calculate the bulk specific gravity, bulk density, and percent water absorption by volume in accordance with Section 7.

VACUUM METHOD

6. PROCEDURE

- 6.1 This procedure can be used for testing specimens, which are to be saved, and which contain a substantial amount of moisture. Specimens obtained by methods that introduce moisture, such as wet coring or sawing, can generally be tested the same day by this method.
- 6.2 Turn on the vacuum drying device. Follow the manufacturer's recommendations for warm up and self-test procedures.
- 6.3 Run the vacuum drying device without any specimens. The pressure reading on the display should indicate a known dry point value as recommended by the manufacturer. If the indicated dry point is not achieved, refer to the manufacturer's trouble shooting instructions.
- 6.4 Ensure the specimen is at room temperature (77 ± 9 °F).

- 6.5 Remove any standing water from the surface of the specimen by using a paper towel or an absorptive cloth.
- 6.6 Dry the cold trap and the specimen chamber. Place the specimen inside the vacuum chamber.
- Note:** Two 4-inch diameter specimens, that are less than 3-inches in thickness can be placed side-by-side in the chamber during a single drying cycle. Larger specimens shall be placed in the chamber individually.
- Note:** Water and/or ice may buildup in the moisture trap during a drying cycle. Wipe off any free standing water in the moisture trap between drying specimens. This will expedite specimen drying.
- 6.7 Place the lid on the vacuum chamber and press the lid down to ensure secure contact between the lid and the chamber. Press the appropriate key on the vacuum drying device to begin the drying process.
- 6.8 The vacuum drying device will automatically stop when the specimen is dry. The unit shall be calibrated to sense the "dry specimen condition". The "dry specimen condition" shall be determined from the calibrated pressure at which no water remains in the chamber. The pressure is monitored throughout the drying process to ensure that the "dry specimen condition" pressure is achieved in the device.
- 6.9 Remove the specimen from the vacuum drying device. Weigh the specimen and record the dry mass of the specimen to the nearest 0.1 gram as "A".
- 6.10 Immerse the specimen in water at 77 ± 2 °F for 4 ± 1 minutes and record the immersed mass to the nearest 0.1 gram as "C". Care shall be exercised to ensure that no trapped air bubbles exist under the specimen.
- 6.11 Remove the specimen from the water, quickly damp dry the specimen surface by blotting with a damp towel, and as quickly as possible determine and record the surface-dry mass to the nearest 0.1 gram as "B". Any water that seeps from the specimen during the surface-dry weighing operation is considered as part of the specimen. The determination of the immersed mass and surface-dry mass of each specimen will be completed before the next specimen is submerged for its immersed mass determination.

Note: If desired, the sequence of testing operations may be changed to expedite the test results. For example, first the immersed mass, "C", can be taken, then the surface-dry mass, "B", and finally the dry mass, "A".

- 6.12 Calculate the bulk specific gravity, bulk density, and percent water absorption by volume in accordance with Section 7.

7. CALCULATION

- 7.1 Calculate and record the bulk specific gravity of the specimen to the nearest 0.001 as follows:

$$\text{Bulk Specific Gravity} = \frac{A}{B - C}$$

Where: A = mass of dry specimen
B = mass of surface-dry specimen
C = mass of immersed specimen

- 7.2 Calculate and record the bulk density of the specimen to the nearest 0.1 lb/cu ft by multiplying the bulk specific gravity by 62.3 lbs/cu ft.

- 7.3 For laboratory molded specimens of 1/2-inch or 3/4-inch asphaltic concrete mixes, the range of bulk density results for three replicate specimens shall not differ by more than 2.5 lbs/cu ft. For laboratory molded specimens of asphaltic concrete Base Mixes, the range of bulk density results for three replicate specimens shall not differ by more than 3.0 lbs/cu ft. If the respective requirement is not met, the entire set of specimens shall be discarded and a new set of specimens shall be prepared and tested.

- 7.4 Calculate and record the percent water absorbed by the specimen to the nearest 0.01 percent (on volume basis) as follows:

$$\text{Percent Water Absorption by Volume} = \frac{B - A}{B - C} \times 100$$

8. REPORT

- 8.1 The method that was used.
- 8.2 The bulk specific gravity to the nearest 0.001.
- 8.3 The bulk density to the nearest 0.1 lb/cu ft.
- 8.4 The absorption to the nearest 0.01 percent.

DETERMINATION OF AIR VOIDS IN COMPACTED BITUMINOUS MIXTURES

(A Modification of AASHTO Designation T 269)

1. SCOPE

- 1.1 This procedure is used to determine the air voids in compacted bituminous mixtures. It is applicable for specimens which are either laboratory compacted or field compacted (for example, cores).
- 1.2 See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

2. CALCULATION

- 2.1 For specimens which are either Marshall laboratory compacted or field compacted (e.g., cores), the percent air voids shall be calculated using the bulk density of the compacted bituminous mixture (Arizona Test Method 415) and maximum density of the mixture from the Rice Test (Arizona Test Method 417).
 - 2.1.1 The percent air voids are calculated by the following equation:
$$\text{Percent Air Voids} = \left[1 - \frac{\text{Bulk Density}}{\text{Maximum Density}} \right] \times 100$$
 - 2.1.1.1 An example of the calculations is given in Figure 1.
 - 2.1.1.2 A blank form for performing the calculations is given in Figure 3.
- 2.2 For specimens which are gyratory laboratory compacted, the percent air voids shall be calculated using the average relative density of the compacted bituminous mixture at N_{design} (AASHTO T 312).

2.2.1 The percent air voids are calculated by the following equation:

$$\text{Percent Air Voids} = (100) - (\text{Average Relative Density, \% } G_{mm}, \text{ at } N_{\text{design}})$$

2.2.1.1 An example of the calculations is given in Figure 2.

2.2.1.2 A blank form for performing the calculations is given in Figure 4.

3. REPORT

3.1 The percent air voids shall be reported to the nearest 0.1%.

CALCULATION OF AIR VOIDS FOR MARSHALL LABORATORY COMPACTED SPECIMENS OR FIELD COMPACTED SPECIMENS				
Specimens Compacted by: Hand <input type="checkbox"/> Mechanical <input checked="" type="checkbox"/> 4 inch <input checked="" type="checkbox"/> 6 inch <input type="checkbox"/> ; Core <input type="checkbox"/>				
Specimen I.D.	1	2	3	Average
Specimen Height	2.516	2.515	2.519	
Bulk Specific Gravity, Bulk Density, and Absorption (Arizona Test Method 415: Method A <input checked="" type="checkbox"/> , Method C <input type="checkbox"/> , or Vacuum Method <input type="checkbox"/>)				
A = Mass in grams of specimen in Air	1155.9	1155.4	1158.2	
B = Mass in grams of SSD specimen in Air	1156.9	1156.3	1159.2	
C = Mass in grams of specimen in Water	647.9	649.6	651.8	
G_{mb} = Bulk Specific Gravity = $A/(B - C)$	2.271	2.280	2.283	2.278
% Absorption = $[(B - A)/(B - C)] \times 100$	0.20	0.18	0.20	
Bulk Density = ($G_{mb} \times 62.3$ lbs./cu. ft.)	141.5	142.0	142.2	
Range of Bulk Density values (lbs./cu. ft.)	0.7			
Average Bulk Density = (Average $G_{mb} \times 62.3$ lbs./cu. ft.)				141.9
Maximum Density (lbs./cu. ft.) [from Rice Test]	149.4			
Notes:				
The Individual specimen heights are reported to the nearest 0.001 inch.				
The Individual specimen masses are reported to the nearest 0.1 gram.				
The Individual bulk specific gravities are reported to the nearest 0.001.				
The average bulk specific gravity is calculated, and reported to the nearest 0.001, using the individual bulk specific gravities which have been reported to the nearest 0.001.				
The individual bulk densities are reported to the nearest 0.1 lb./cu. ft.				
The average bulk density is reported to the nearest 0.1 lb./cu. ft.				
The maximum density [from Rice Test] is reported to the nearest 0.1 lb./cu. ft.				
$\text{Percent Air Voids} = \left[1 - \frac{\text{Average Bulk Density}}{\text{Maximum Density from Rice Test}} \right] \times 100 = \left[1 - \frac{141.9}{149.4} \right] \times 100 = 5.0\%$				

**EXAMPLE AIR VOIDS CALCULATION FOR
 MARSHALL LABORATORY COMPACTED SPECIMENS**

FIGURE 1

CALCULATION OF AIR VOIDS FOR GYRATORY LABORATORY COMPACTED SPECIMENS			
Specimen I.D.	1	2	Average
h_{ini} = Height, in mm, of specimen at N_{ini} (8 gyrations)	128.7	129.3	
h_{des} = Height, in mm, of specimen at N_{des} (100 gyrations)	117.0	117.4	
h_{max} = Height, in mm, of specimen at N_{max} (160 gyrations)	115.6	116.0	
Bulk Specific Gravity and Absorption (Arizona Test Method 415: Method A <input type="checkbox"/> , Method C <input type="checkbox"/> , or Vacuum Method <input type="checkbox"/>)			
A = Mass, in grams, of specimen at N_{max} in Air	4747.4	4744.6	
B = Mass, in grams, of SSD specimen at N_{max} in Air	4759.4	4756.0	
C = Mass, in grams, of specimen at N_{max} in Water	2752.7	2751.2	
G_{mb} = Bulk Specific Gravity of specimen at N_{max} = $\frac{A}{B - C}$	2.366	2.367	
% Absorption = $[(B - A)/(B - C)] \times 100$	0.60	0.57	
G_{mm} = Maximum Specific Gravity [from Rice Test]	2.449		
*Relative Density, % G_{mm} , of specimen at N_{ini}	86.8	86.7	86.8
*Relative Density, % G_{mm} , of specimen at N_{des}	95.5	95.5	95.5
*Relative Density, % G_{mm} , of specimen at N_{max}	96.6	96.7	96.7
$* \text{Relative Density, \% } G_{mmx} = \frac{(G_{mb}) \times (h_{max})}{(G_{mm}) \times (h_x)} \times 100$ <p>Where: %G_{mmx} = Relative Density, %G_{mm}, of specimen at N_{ini}, N_{des}, or N_{max} G_{mb} = Bulk Specific Gravity of specimen at N_{max} h_{max} = Height, in mm, of specimen at N_{max} G_{mm} = Maximum Specific Gravity [from Rice Test] h_x = Height of specimen, in mm, at N_{ini}, N_{des}, or N_{max}</p>			
Notes:			
The Individual specimen heights are reported to the nearest 0.1 mm.			
The Individual specimen masses are reported to the nearest 0.1 gram.			
The Individual bulk specific gravities are reported to the nearest 0.001.			
The maximum specific gravity [from Rice Test] is reported to the nearest 0.001.			
The individual relative densities are reported to the nearest 0.1 percent.			
The average relative density for each set of specimens (at N_{ini} , N_{des} , and N_{max}) is calculated, and reported to the nearest 0.1 percent, using the corresponding individual relative densities which have been reported to the nearest 0.1 percent.			
Three specimens are used when referee testing is performed.			
Percent Air Voids = (100) – (Average Relative Density, % G_{mm}, at N_{des}) = (100) – (95.5) = 4.5%			

**EXAMPLE AIR VOIDS CALCULATION FOR
 GYRATORY LABORATORY COMPACTED SPECIMENS**

FIGURE 2

CALCULATION OF AIR VOIDS FOR MARSHALL LABORATORY COMPACTED SPECIMENS OR FIELD COMPACTED SPECIMENS				
Specimens Compacted by: Hand <input type="checkbox"/> Mechanical <input type="checkbox"/> 4 inch <input type="checkbox"/> 6 inch <input type="checkbox"/> ; Core <input type="checkbox"/>				
Specimen I.D.				Average
Specimen Height				
Bulk Specific Gravity, Bulk Density, and Absorption (Arizona Test Method 415: Method A <input checked="" type="checkbox"/> , Method C <input type="checkbox"/> , or Vacuum Method <input type="checkbox"/>)				
A = Mass in grams of specimen in Air				
B = Mass in grams of SSD specimen in Air				
C = Mass in grams of specimen in Water				
$G_{mb} = \text{Bulk Specific Gravity} = A/(B - C)$				
% Absorption = $[(B - A)/(B - C)] \times 100$				
Bulk Density = $(G_{mb} \times 62.3 \text{ lbs./cu. ft.})$				
Range of Bulk Density values (lbs./cu. ft.)				
Average Bulk Density = $(\text{Average } G_{mb} \times 62.3 \text{ lbs./cu. ft.})$				
Maximum Density (lbs./cu. ft.) [from Rice Test]				
Notes: The Individual specimen heights are reported to the nearest 0.001 inch. The Individual specimen masses are reported to the nearest 0.1 gram. The Individual bulk specific gravities are reported to the nearest 0.001. The average bulk specific gravity is calculated, and reported to the nearest 0.001, using the individual bulk specific gravities which have been reported to the nearest 0.001. The individual bulk densities are reported to the nearest 0.1 lb./cu. ft. The average bulk density is reported to the nearest 0.1 lb./cu. ft. The maximum density [from Rice Test] is reported to the nearest 0.1 lb./cu. ft.				
Percent Air Voids = $\left[1 - \frac{\text{Average Bulk Density}}{\text{Maximum Density from Rice Test}} \right] \times 100 = \left[1 - \frac{(\quad)}{(\quad)} \right] \times 100 = \underline{\quad}\%$				

FIGURE 3

CALCULATION OF AIR VOIDS FOR GYRATORY LABORATORY COMPACTED SPECIMENS			
Specimen I.D.			Average
h_{ini} = Height, in mm, of specimen at N_{ini} (8 gyrations)			
h_{des} = Height, in mm, of specimen at N_{des} (100 gyrations)			
h_{max} = Height, in mm, of specimen at N_{max} (160 gyrations)			
Bulk Specific Gravity and Absorption (Arizona Test Method 415: Method A <input type="checkbox"/> , Method C <input type="checkbox"/> , or Vacuum Method <input type="checkbox"/>)			
A = Mass, in grams, of specimen at N_{max} in Air			
B = Mass, in grams, of SSD specimen at N_{max} in Air			
C = Mass, in grams, of specimen at N_{max} in Water			
G_{mb} = Bulk Specific Gravity of specimen at N_{max} = $\frac{A}{B - C}$			
% Absorption = $[(B - A)/(B - C)] \times 100$			
G_{mm} = Maximum Specific Gravity [from Rice Test]			
*Relative Density, % G_{mm} , of specimen at N_{ini}			
*Relative Density, % G_{mm} , of specimen at N_{des}			
*Relative Density, % G_{mm} , of specimen at N_{max}			
$* \text{Relative Density, \% } G_{mmx} = \frac{(G_{mb}) \times (h_{max})}{(G_{mm}) \times (h_x)} \times 100$ <p>Where: %G_{mmx} = Relative Density, %G_{mm}, of specimen at N_{ini}, N_{des}, or N_{max} G_{mb} = Bulk Specific Gravity of specimen at N_{max} h_{max} = Height, in mm, of specimen at N_{max} G_{mm} = Maximum Specific Gravity [from Rice Test] h_x = Height of specimen, in mm, at N_{ini}, N_{des}, or N_{max}</p>			
Notes: The Individual specimen heights are reported to the nearest 0.1 mm. The Individual specimen masses are reported to the nearest 0.1 gram. The Individual bulk specific gravities are reported to the nearest 0.001. The maximum specific gravity [from Rice Test] is reported to the nearest 0.001. The individual relative densities are reported to the nearest 0.1 percent. The average relative density for each set of specimens (at N_{ini} , N_{des} , and N_{max}) is calculated, and reported to the nearest 0.1 percent, using the corresponding individual relative densities which have been reported to the nearest 0.1 percent. Three specimens are used when referee testing is performed.			
Percent Air Voids = (100) – (Average Relative Density, % G_{mm}, at N_{des}) = (100) – (_____) = _____ %			

FIGURE 4

SULFATE IN SOILS

(An Arizona Method)

1. SCOPE

- 1.1 This test method describes a procedure for determining sulfate content in soil using a turbidimeter. The sulfate content determined is defined in terms of the method and may be called water soluble sulfate.
- 1.2 This test method involves hazardous material, operations, and equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
- 1.3 See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.
- 1.4 The extraction procedure, Subsections 4.1 through 4.4, is the same as is used in Arizona Test Method 736, "Chloride In Soils".

2. APPARATUS

- 2.1 Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus for this test procedure shall consist of the following:
 - 2.1.1 Turbidimeter - with at least $\pm 2\%$ accuracy to 1000 NTU (nephelometric turbidity units) and at least $\pm 5\%$ accuracy to 4000 NTU, with sample cuvettes.
 - 2.1.2 Balances or scales:
 - 2.1.2.1 One balance or scale capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M 231, except the readability and sensitivity shall be at least 0.1 gram.

- 2.1.2.2 An analytical balance capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M231, except the readability and sensitivity shall be at least 0.1 milligram.
- 2.1.3 Beaker(s) - three, 200 mL capacity each.
- 2.1.4 Erlenmeyer flask - 500 mL capacity with stopper.
- 2.1.5 Volumetric flask - 1000 mL capacity, accurate to 0.3 mL.
- 2.1.6 Reagent dispensing bottle - 500 mL capacity, capable of repeatedly dispensing 5 mL of reagent.
- 2.1.7 Reagent storage bottles - three, one-liter capacity, each with cap.
- 2.1.8 Dropping bottle - 60 mL, with dispensing tip or dropper.
- 2.1.9 Centrifuge tube - 50 mL, with cap.

3. REAGENTS

- 3.1 Conditioning Reagent.
 - 3.1.1 Into a reagent storage bottle, add 300 mL demineralized water, 30 mL Reagent Grade Concentrated Hydrochloric Acid, 100 mL Reagent Grade Isopropyl Alcohol, 75 grams Reagent Grade Sodium Chloride, and 50 mL Reagent Grade Glycerol. Mix well. Transfer to a reagent dispensing bottle as needed.
- 3.2 Sulfate Standard Solution, 0.100 mg/mL (100 ppm).
 - 3.2.1 Into a 1000 mL volumetric flask, add 147.9 mg Reagent Grade Anhydrous Sodium Sulfate. Fill the flask to the 1000 mL mark with demineralized water. Mix well. Transfer to a reagent storage bottle.
- 3.3 Barium Chloride Reagent Solution, 1%.
 - 3.3.1 Into a 1000 mL volumetric flask, add 10 grams of Reagent Grade Barium Chloride Powder, Anhydrous. Fill the flask to the 1000 mL mark with demineralized water. Mix well. Transfer to a reagent dispensing bottle as needed, and the remainder to a reagent storage bottle.

- 3.4 Nitric Acid, 20%.
- 3.4.1 Measure 10 mL concentrated nitric acid into a suitable size beaker containing approximately 40 mL demineralized water and mix well. Transfer to a dropping bottle.

CAUTION: Exercise extreme caution in preparing and using the Nitric Acid solution. It must be properly labeled and treated as a hazardous material.

- 3.5 Demineralized water.

4. PROCEDURE

- 4.1 Weigh 100.0 ± 0.1 grams of soil passing a No. 10 sieve into a 500 mL Erlenmeyer flask.
- 4.2 Weigh 300.0 ± 0.1 grams demineralized water into the flask.
- 4.3 Stopper the flask, shake vigorously, and let the mixture stand undisturbed for one hour.
- 4.4 Carefully, with minimal disturbance of the sediment, decant 50 mL of the extract solution into a centrifuge tube.
 - 4.4.1 If the decanted extract solution is clear, proceed to Subsection 4.5.
 - 4.4.2 If the decanted extract solution is not clear, place the tube into the centrifuge and centrifuge at a minimum of 5000 RPM for 10 minutes. If the solution is still not clear, add 2 drops of 20% Nitric Acid solution to the tube and centrifuge again. Repeat until the centrifugate is clear.
- 4.5 Transfer 20.0 mL of the clear extract solution (or centrifugate) into a 200 mL beaker. This is the sample aliquot, "ALSAM."
 - 4.5.1 Dispense 5 mL of Conditioning Reagent and 5 mL of Barium Chloride Reagent Solution into the beaker, and dilute to 100 mL. This is the "Sample Solution."
- 4.6 Transfer 20.0 mL of Sulfate Standard Solution into a second 200 mL beaker. This is the standard aliquot "ALSTD."

- 4.6.1 Dispense 5 mL of Conditioning Reagent and 5 mL of Barium Chloride Reagent Solution into this beaker, and dilute to 100 mL. This is the "Standard Solution."
- 4.7 Dispense 5 mL of Conditioning Reagent and 5 mL of Barium Chloride Reagent Solution into a third 200 mL beaker, and dilute to 100 mL. This is the "Reagent Blank Solution."
- 4.8 Stir the three solutions and let them stand undisturbed for at least 15 minutes.
- 4.9 Rinse a sample cuvette with demineralized water and then with freshly stirred Reagent Blank Solution. Immediately transfer enough Reagent Blank Solution to fill the cuvette to the mark.
- 4.9.1 Place the cuvette into the turbidimeter and read the turbidity to the nearest 0.1 NTU. The turbidity of the Reagent Blank Solution is recorded as "TBBNK".
- 4.10 Rinse a sample cuvette with demineralized water and then with freshly stirred Sample Solution. Immediately transfer enough Sample Solution to fill the cuvette to the mark.
- 4.10.1 Place the cuvette into the turbidimeter and read the turbidity to the nearest 0.1 NTU. Record the turbidity of the Sample Solution as "TBSAM."
- 4.11 Rinse a sample cuvette with demineralized water and then with freshly stirred Standard Solution. Immediately transfer enough Standard Solution to fill the cuvette to the mark.
- 4.11.1 Place the cuvette into the turbidimeter and read the turbidity to the nearest 0.1 NTU. Record the turbidity of the Standard Solution as "TBSTD."
- 4.12 Compare the values of "TBSAM" and "TBSTD." If "TBSAM" is larger than "TBSTD", repeat Subsections 4.5 and 4.5.1 using a suitably smaller sample aliquot. Record this volume, to the nearest 0.1 mL, as "ALSAM." Repeat Subsections 4.10 and 4.10.1.

5. CALCULATION AND REPORT

- 5.1 Calculate sulfate content in the soil in parts per million, "S", and report to the nearest 10 ppm as follows:

$$S = 300 \times \left[\frac{\text{ALSTD} \times \text{TBSAM}}{\text{ALSAM} \times \text{TBSTD}} \right]$$

Where:

ALSTD = 20.0 mL (Size of the standard aliquot).

TBSTD = Turbidity corresponding to ALSTD.

ALSAM = Size of the sample aliquot, mL.

TBSAM = Turbidity corresponding to ALSAM.

Note: If the turbidity of the Reagent Blank Solution "TBBNK" is 0.40 or higher, for greater accuracy, the value of "TBBNK" shall be subtracted from "TBSAM" and from "TBSTD" before calculating S above.

CHLORIDE IN SOILS

(An Arizona Method)

1. SCOPE

- 1.1 This test method describes a procedure for determining chloride content in soil by a standard addition technique using a chloride electrode. The chloride content is defined in terms of the method and may be called water soluble chloride.
- 1.2 This test method involves hazardous material, operations, and equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.
- 1.3 See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.
- 1.4 The extraction procedure, Subsections 4.1 through 4.4, is the same as is used in Arizona Test Method 733, "Sulfate In Soils".

2. APPARATUS

- 2.1 Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus shall consist of the following:
 - 2.1.1 Tall-form beakers - two, 200 mL each (Pyrex #1060 or equivalent), calibrated to indicate 100 mL volume.
 - 2.1.2 An analytical balance capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M231, except the readability and sensitivity of any balance utilized shall be at least 0.001 gram.
 - 2.1.3 Magnetic stirrer and stirring bar.

- 2.1.4 Erlenmeyer flask(s) - 500 mL capacity with stopper(s).
- 2.1.5 Chloride electrode system - (Orion Ionplus #9617 combination chloride electrode or equivalent), suitable for use in a 200 mL tall-form beaker.
- 2.1.6 Specific ion meter - (Orion EA 940 or equivalent), suitable for use with the chloride electrode system.
- 2.1.7 Thermometer - accurate to at least 0.5 °C.
- 2.1.8 Pipettes - 1 mL, 5 mL, and 10 mL capacity, accurate to 1%.
- 2.1.9 Sample bottle - 200 mL capacity or larger, with cap.
- 2.1.10 Dropping bottle - 60 mL, with dispensing tip or dropper.
- 2.1.11 Centrifuge tube - 50 mL, with cap.

3. REAGENTS

- 3.1 Chloride Standard Solution, 1.000 mg/mL (1000 ppm).
 - 3.1.1 Fisher Scientific # LC13000-1 or equivalent. Alternatively, the Chloride Standard Solution may be made by transferring 1.648 gram dried primary standard sodium chloride into a 1000 mL volumetric flask and diluting to 1000 mL with demineralized water.
- 3.2 Nitric Acid, 20%.
 - 3.2.1 Measure 10 mL concentrated nitric acid into a suitable size beaker containing approximately 40 mL demineralized water and mix well. Transfer to a dropping bottle.

CAUTION: Exercise extreme caution in preparing and using the Nitric Acid solution. It must be properly labeled and treated as a hazardous material.

- 3.3 Demineralized Water.

4. PROCEDURE

- 4.1 Weigh 100.0 ± 0.1 grams of soil passing a No. 10 sieve into a 500 mL Erlenmeyer flask.
- 4.2 Weigh 300.0 ± 0.1 grams demineralized water into the flask.
- 4.3 Stopper the flask, shake vigorously, and let the mixture stand undisturbed for one hour.
- 4.4 Carefully, with minimal disturbance of the sediment, decant 50 mL of the extract solution into a 50 mL centrifuge tube.
- 4.4.1 If the decanted extract solution is not cloudy, proceed to Subsection 4.5.
- Note:** The decanted extract solution does not have to be clear, as it is required to be in Arizona Test Method 733.
- 4.4.2 If the decanted extract solution is cloudy, place the tube into the centrifuge and centrifuge at a minimum of 5000 RPM for 10 minutes.
- 4.5 Pipette a 10.0 mL aliquot of the extract solution (or centrifugate) into a 200 mL beaker. Record this volume as "A". Dilute to the 100 mL mark with demineralized water and add three drops of Nitric Acid, 20%. This is the "Sample Reading Solution."
- 4.6 Place a stirring bar into the beaker, place the beaker onto the magnetic stirrer, insert the electrodes, and initiate stirring. Stirring shall be at a constant moderate rate, such that the vortex created by stirring does not expose the tips of the immersed electrodes. The rate of stirring shall be constant throughout the procedure.
- 4.7 After the reading has stabilized, record the initial reading to the nearest millivolt, as " E_1 ".
- 4.8 Add 1.00 mL of Chloride Standard Solution.
- 4.9 After the reading has stabilized, record the final reading to the nearest millivolt, as " E_2 ".
- 4.10 Calculate ΔE . ($\Delta E = E_1 - E_2$).

Note: " ΔE " is calculated and recorded initially as " ΔE_o " for the Sample Reading Solution. It is subsequently also calculated and recorded as " ΔE_b " for the Reagent Blank Reading Solution.

- 4.11 If " ΔE_o " (for the Sample Reading Solution) is less than 18, repeat Subsections 4.5 through 4.10 with a suitably smaller size aliquot, recording this aliquot volume as "A."
- 4.12 Prepare a Reagent Blank Reading Solution by placing 100 mL of demineralized water into a second 200 mL beaker and adding 3 drops of Nitric Acid 20%. Repeat Subsections 4.6 through 4.10, calculating and recording " ΔE_b " (for the Reagent Blank Reading Solution).

5. CALCULATIONS AND REPORT

- 5.1 Calculate chloride concentration, " C_o " in the Sample Reading Solution and " C_b " in the Reagent Blank Reading Solution, and record each to the nearest 0.001 mg/mL, as follows:

$$C_o = \frac{1}{\left[101 \times 10^{[(\Delta E_o)/S]} \right] - 100}$$

$$C_b = \frac{1}{\left[101 \times 10^{[(\Delta E_b)/S]} \right] - 100}$$

Where:

S = Electrode slope, as determined in accordance with the manufacturer's recommendations. (The slope should equal approximately 59 millivolts for a properly functioning electrode.)

- 5.2 Calculate chloride concentration in the soil in parts per million, "C", and report to the nearest 10 ppm as follows:

$$C = \frac{300,000 \times (C_o - C_b)}{A}$$