

LOW PERMEABILITY CONCRETE FOR BRIDGE DECK OVERLAY

**FINAL REPORT
MLR-89-1**

JULY 1993

Highway Division



**Iowa Department
of Transportation**

Low Permeability Concrete
for Bridge Deck Overlay

Final Report
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By

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July 1993

TECHNICAL REPORT TITLE PAGE

1. REPORT NO.	2. REPORT DATE
MLR-89-1	July 1993
3. TITLE AND SUBTITLE	4. TYPE OF REPORT & PERIOD COVERED
Low Permeability Concrete for Bridge Deck Overlay	Final Report 4-89 to 7-93
5. AUTHOR(S)	6. PERFORMING ORGANIZATION ADDRESS
Robert F. Steffes Assistant to the Research Engineer	Iowa Department of Transportation Materials Department 800 Lincoln Way Ames, Iowa 50010
7. ACKNOWLEDGEMENT OF COOPERATING ORGANIZATIONS	
8. ABSTRACT	
<p>The penetration of chloride-ions from deicing salts into the portland cement concrete of bridge decks can cause corrosion and serious damage to the reinforcing steel. Concrete properties which prevent chloride penetration into the bridge deck and provide a good structural and economic wearing surface are desirable. A variety of mix designs have been tried in the past in search of improved performance and lower costs for bridge deck overlay concrete. A group of mixes with various designs have been tested in this project and results are being compared to determine which concrete mix appears to be the most cost effective and resistant to chloride penetration for bridge deck overlay use.</p>	
9. KEY WORDS	10. NO. OF PAGES
Concrete, permeability Chloride, bridge deck	43

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DISCLAIMER

The contents of this report reflect the views of the author and do not necessarily reflect the official views of the Iowa Department of Transportation. This report does not constitute any standard, specification or regulation.

INTRODUCTION

A major problem facing the Iowa highway industry is the chloride-ion penetration of bridge decks. Chloride-ions, from the deicing salts used for snow and ice removal, penetrate the bridge deck concrete and corrode the reinforcing steel. As steel corrodes, it expands and exerts stresses on the surrounding concrete. When the stresses exceed the strength of the concrete, cracks and delaminations form. Deterioration and spalling on bridge deck surfaces result from the cracks and delaminations.

If the reinforcing steel is protected by low permeability concrete and is not exposed to excessive chloride-ions, the steel may not corrode for a long period of time and the bridge deck will remain sound. Only when the chloride-ion content of the concrete exceeds the threshold value at the reinforcing steel, which is considered to be 1.5 pounds of chloride-ions per cubic yard of cement, is corrosion of the steel expected to occur.

Many different techniques to prevent corrosion of the reinforcing steel are being used. Galvanized reinforcing steel has been used. Epoxy coated steel is being used and is quite effective at resisting corrosion. The drawback with epoxy coated steel is that if the epoxy coating gets chipped off or cracks or has holidays (pinholes), the steel is no longer totally protected. Non-corrosive deicers and corrosion inhibited salts are available, but are considered quite expensive for use in the

field. A calcium nitrite additive can be used in concrete as a corrosion inhibitor, but it is also expensive. Cathodic protection, in which a metal net is placed within the bridge deck and electrically prevents the corrosion process, is also being used. A major drawback it has is that it needs continual monitoring and has electrical power requirements, making it somewhat impractical for some applications.

A possible solution to reduce chloride contamination is the use of admixtures such as latex, fly ash and silica fume in the portland cement concrete. These admixtures can reduce concrete permeability to chloride-ions and, thereby, delay or prevent corrosion of reinforcing steel. By experimenting with these admixtures along with the accepted "Iowa Dense" overlay mix design, we may be able to develop a low water/cement ratio concrete which would provide an improvement in resistance to penetration of chloride-ions. A concrete mix design for bridge deck overlays with lower permeability to chlorides and low drying shrinkage may prove to be economically viable in spite of the high cost of some of the admixtures used.

OBJECTIVE

The objective of this project is to evaluate a variety of low permeability concrete mix designs which have the potential to minimize chloride penetration for bridge deck overlays.

SCOPE

The scope of this project is to evaluate 10 concrete overlay mix designs covering the use of 4 different materials in various proportions to obtain permeability reduction. The 10 mixtures, including one standard 0-4 mix and one D-57 mix, were evaluated by the AASHTO T 277-89, "Rapid Determination of the Permeability of Portland Cement Concrete" (3) test for 90 days. Additional tests (AASHTO T 277-89) were performed at 180 and 360 days on the mixes to observe a longer term development of permeability reduction in the concrete. The 10 mixtures were tested by salt ponding for 90 days as per AASHTO T 259-80, "Resistance of Concrete to Chloride-Ion Penetration," (1) and AASHTO T 260-84, "Sampling and Testing for Total Chloride-Ion in Concrete and Concrete Raw Materials," (2) to obtain chloride content from the salt ponding. Drying shrinkage was monitored up to 180 days to determine any reduction in shrinkage achieved through these mix designs. Compression tests were also performed on each of the 10 mixes to evaluate the variations in strength from the different materials added to the concrete.

LAB PROCEDURES

Materials

1. Portland Cement: Type I, a lab blend of eight different portland cement sources.
2. Class F Fly Ash:
Clinton Fly Ash, Clinton, IA
3. Fine Aggregate:
Mississippi River Sand, Cordova, IL
4. Coarse Aggregate:
Martin-Marietta Limestone, Fort Dodge, IA
Coarse aggregate gradations are given in Appendix B

5. Water:
City of Ames
6. Silica Fume:
Force 10,000, W.R. Grace and Co.
7. Latex:
Modifier A., Dow Chemical Co.
8. Air Entraining Agent:
Ad-Aire Naturalized vinsol resin, single strength
Carter-Waters
9. Water Reducing Admixture:
WRDA 82, W. R. Grace and Co.
10. Super Water Reducer:
Daracem 100, W. R. Grace and Co.

Mixes

BRIDGE OVERLAY MIX PROPORTIONS

Mix No.	Type of Mix	Cement lbs/yd ³	Modifications	Fly Ash lbs/yd ³	Latex gal/yd ³	Silica Fume lbs/yd ³	Add mix
1	D-57	710	Standard				
2	0-4	823	IA Dense				WR
3	D-57/w Latex	710	Latex 1½ gal/SK		13.21		
4	D-57/w Latex	710	Latex 3½ gal/SK		26.42		
5	D-57-F	497	Fly Ash 30% F	266			WR
6	D-57/W Silica	710	Silica Fume 10%			71	SWR
7	0-4/w Latex	823	Latex 1½ gal/SK		15.3		
8	D-57-F/w Latex	497	Fly Ash 30% F Latex 1½ gal/SK	266	13.21		WR
9	C-4/w Silica	624	Silica Fume 11.3			71	SWR
10	D-57-F/w Silica	639	Fly Ash 10% F Silica Fume 10%	89		71	SWR

The slump and air data is given in Appendix C1.

From each mix:

Prepare two salt ponding slabs 12" x 12" x 4" and carry out 90 day tests according to AASHTO T 259-80

Prepare one 4" x 8" cylinder for 90, 180 and 360 day Rapid Determination of the Chloride Permeability according to AASHTO T 277-83.

Prepare three 4½" x 9" cylinders for compressive strength tests at 28 days according to AASHTO T 22-82.

Prepare one 4" cube for base chloride testing.

Prepare two 4" x 4" x 18" beams to measure drying shrinkage up to 180 days.

TESTING

90-Day Salt Ponding Test

For each mix, two 12" x 12" x 2" ponding slabs and a 4" cube were made. Each slab was made and tested in accordance with the requirements of AASHTO T 259-80, "Resistance of Concrete to Chloride-Ion Penetration." After the procedures in AASHTO T 259-80 were completed, three holes were drilled in each slab and in the cube. At each hole, powdered concrete samples were taken from depths of 1/16" to 1/2", 1/2" to 1", and 1" to 1 1/2" to determine the chloride content by AASHTO T 260-84, "Sampling and Testing for Total Ion in Concrete and Concrete Raw Materials."

Rapid Determination Chloride Permeability Test

Two 6" x 6" x 20" beams were made for each mix. Four-inch diameter cores were drilled from each beam and sawed to two-inch high specimens. Each core was then transferred to a moisture room at a 100% humidity and at 73°F ± 3°. After a 90 day curing period, the cores were taken out of the moisture room. They were then prepared for and tested in accordance with AASHTO T 277-89,

"Rapid Determination of the Chloride Permeability of Portland Cement Concrete." This test was performed at 90, 180 and 360 days on each mix to observe a long term development of permeability reduction in the concrete.

Other Tests

Each mix was measured for its shrinkage and loss of weight during curing. The 28-day compression strength test was also performed in accordance to AASHTO T 22-82, "Compressive Strength of Cylindrical Concrete Specimens."

MATERIAL COST ESTIMATES

The following material costs are based upon estimated industry averages. The objective of the cost evaluations is to determine the overall differences in material costs per cubic yard of concrete or the total difference in costs between some mixes for a hypothetical bridge deck.

TABLE 1
Material Costs
(Estimated Industry Averages)

Cement	\$65/ton
Fly Ash	\$13/ton
Fine Aggregate	\$5.10/ton
Coarse Aggregate	\$8.95/ton
Modifier A Latex	\$4.25/gal
Silica Fume Slurry	\$2.60/gal
WRDA 82	\$3.70/gal
Daracem 100	\$5.25/gal
Ad-Aire	\$2.20/gal

TABLE 2
Cost Comparison of Mixes

	<u>Material/yd³</u>	<u>Cost/yd³</u>
Mix #1	D-57	
	Cement 710 lbs	\$23.08
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Ad Aire 0.53 oz (0.07 oz/94 lbs cement)	<u>0.01</u>
		\$32.95
Mix #2	0-4	
	Cement 823 lbs	\$26.75
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Ad Aire 2.62 oz (0.3 oz/94 lbs cement)	0.05
	WR 24.6 oz	<u>0.71</u>
		\$37.37
Mix #3	D-57 With Latex	
	Cement 710 lbs	\$23.08
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Latex 13.21 gal (1.75 gal/94 lbs cement)	<u>56.14</u>
		\$89.08
Mix #4	D-57 With Latex	
	Cement 710 lbs	\$23.08
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Latex 26.42 gal (3.5 gal/94 lbs cement)	<u>112.29</u>
		\$145.23
Mix #5	D-57 With Fly Ash	
	Cement 497 lbs	\$16.15
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Fly Ash 266 lbs	1.73
	Ad Aire 9.06 oz (1.2 oz/94 lbs cement)	0.16
	WR 21.3 oz	<u>0.62</u>
		\$28.52
Mix #6	D-57 With Silica Fume	
	Cement 710 lbs	\$23.08
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Silica Fume 71 lbs	33.56
	Ad Aire 9.06 oz (1.2 oz/94 lbs cement)	0.16
	SWR 99.4 oz	<u>4.08</u>
		\$70.74

Mix #7	0-4 With Latex	
	Cement 823 lbs	\$ 26.75
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Latex 15.3 gal	<u>65.03</u>
		\$101.64
Mix #8	D-57 With Fly Ash and Latex	
	Cement 497 lbs	\$16.15
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Fly Ash 266 lbs	1.73
	Latex 13.21 gal	56.14
	WR 21.3 oz	<u>0.62</u>
		\$84.50
Mix #9	C-4 With Silica Fume	
	Cement 624 lbs	\$20.28
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Silica Fume 71 lbs	33.56
	Ad Aire 7.9 oz	<u>0.14</u>
		\$67.42
Mix #10	D-57 With Fly Ash and Silica Fume	
	Cement 639 lbs	\$20.77
	C Agg 1404 lbs	6.28
	F Agg 1404 lbs	3.58
	Fly Ash 89 lbs	0.58
	Silica Fume 71 lbs	33.56
	Ad Aire 12.2 oz (1.8 oz/94 lbs cement)	0.21
	SWR 99.4 oz	<u>4.08</u>
		\$69.06

TEST RESULTS

Test results are shown in the following figures:

Figure 1 - Rapid Determination of the Chloride Permeability of Concrete, AASHTO 277-89

Figure 2 - Resistance of Concrete to Chloride-Ion Penetration, AASHTO T 259-80

Figure 3 - Compressive Strength of Cylindrical Concrete Specimens, AASHTO T 22-82

Figure 4 - Drying Shrinkage of 18" Beams at 180 Days

Rapid Determination of the Chloride Permeability of Concrete, AASHTO 277-89

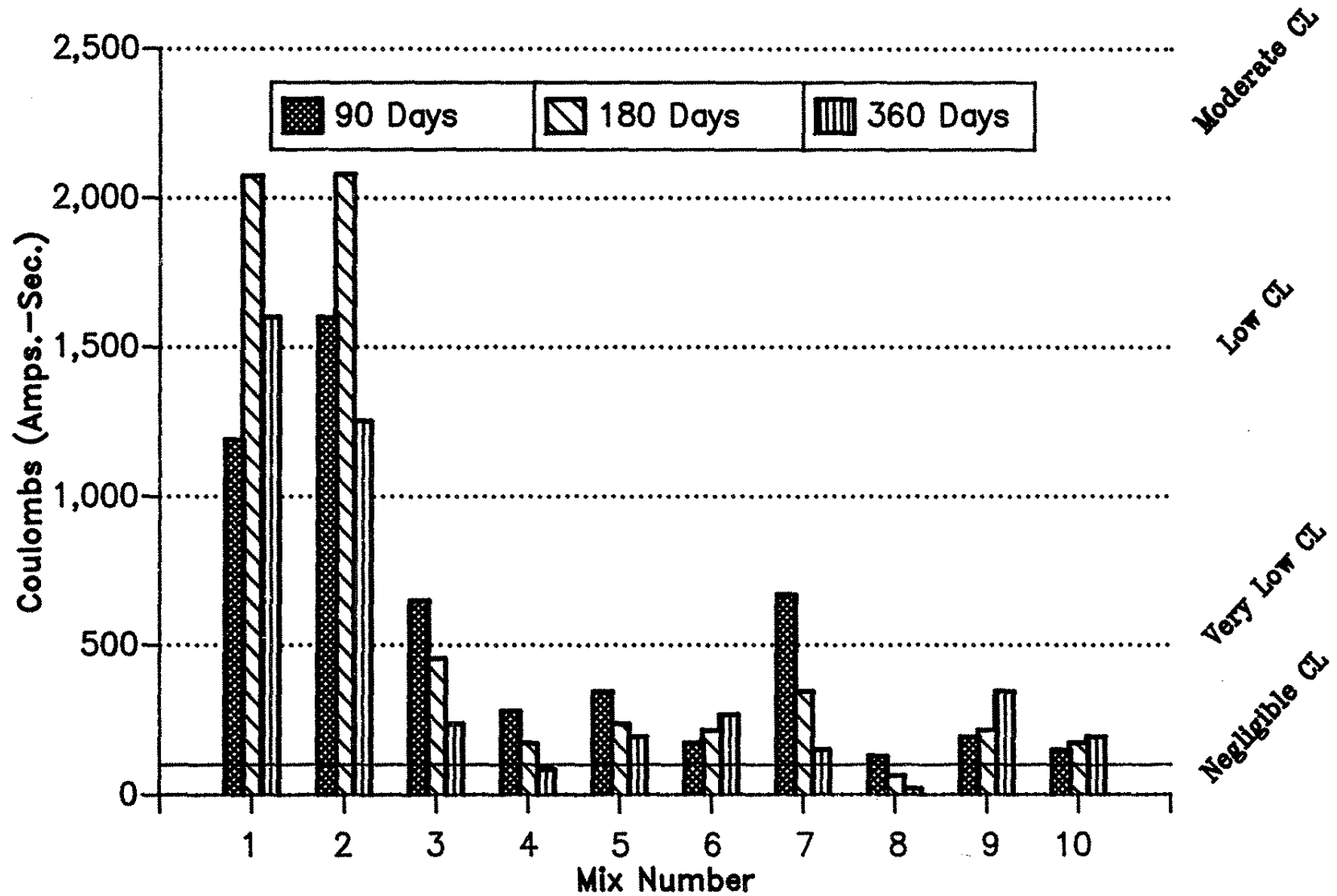


Figure 1

Resistance of Concrete to Chloride-Ion Penetration, AASHTO T 259-80

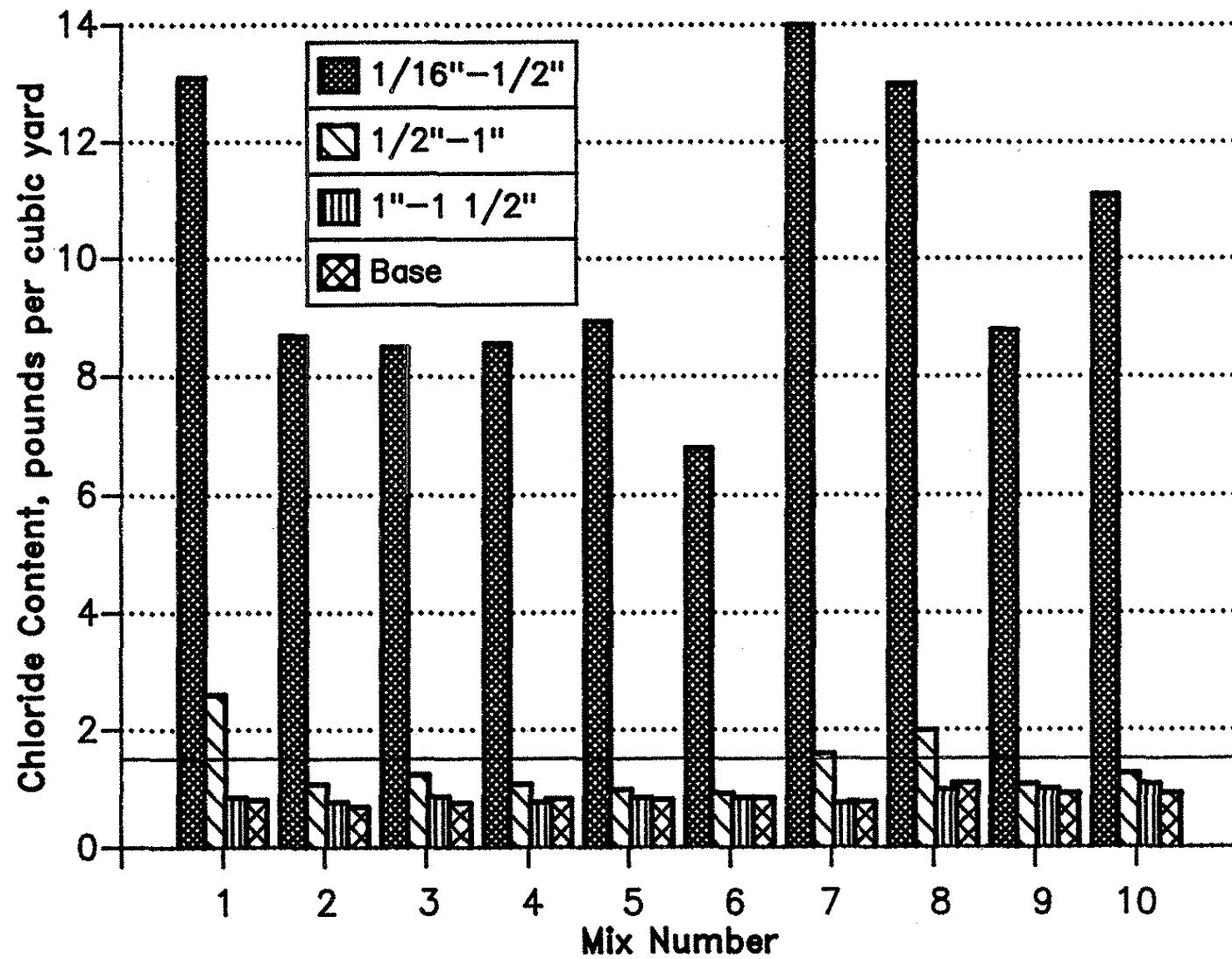


Figure 2

Compression Strength of Cylindrical Concrete Specimens, AASHTO T 22-82

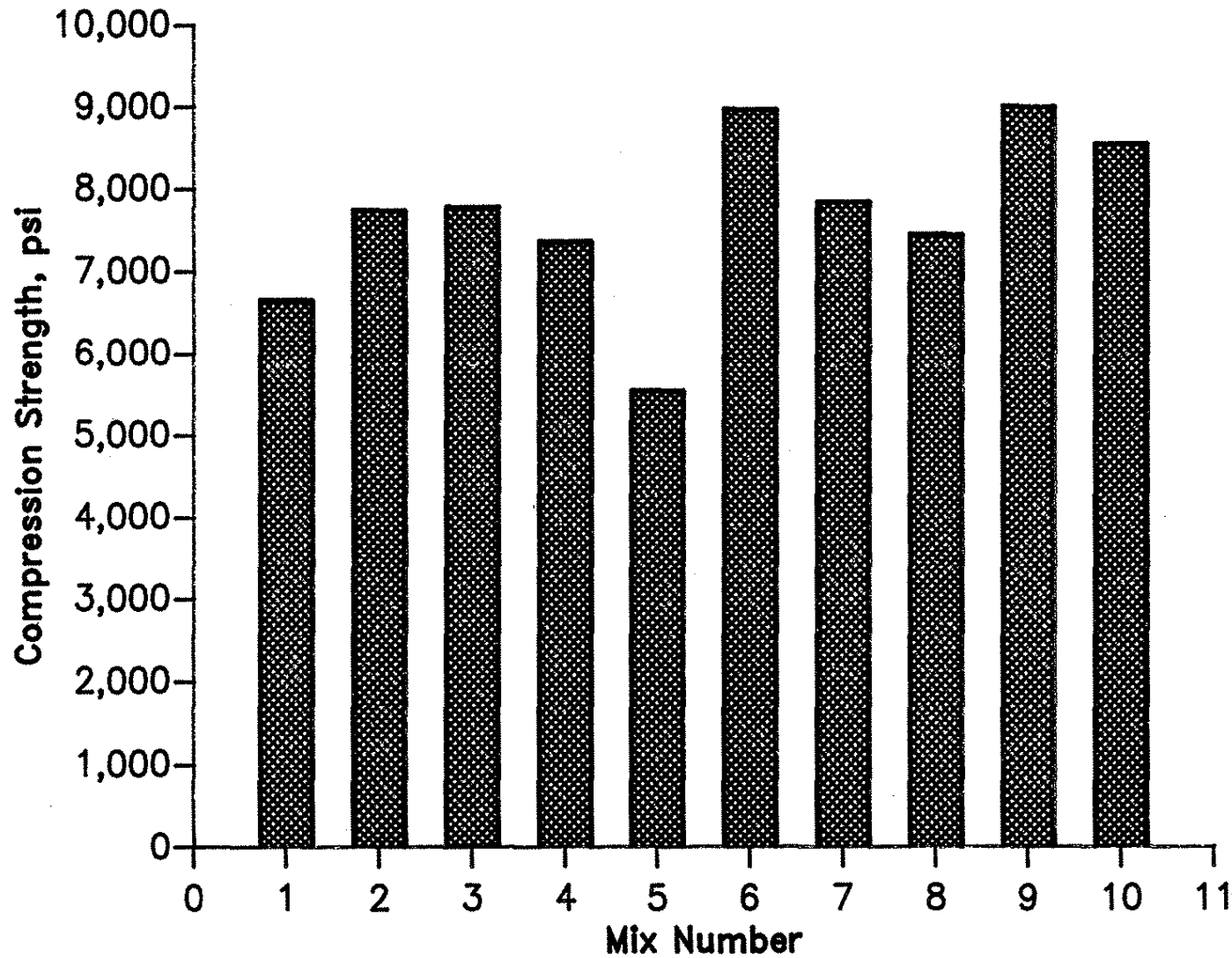


Figure 3

Drying Shrinkage of 18" Beams at 180 Days

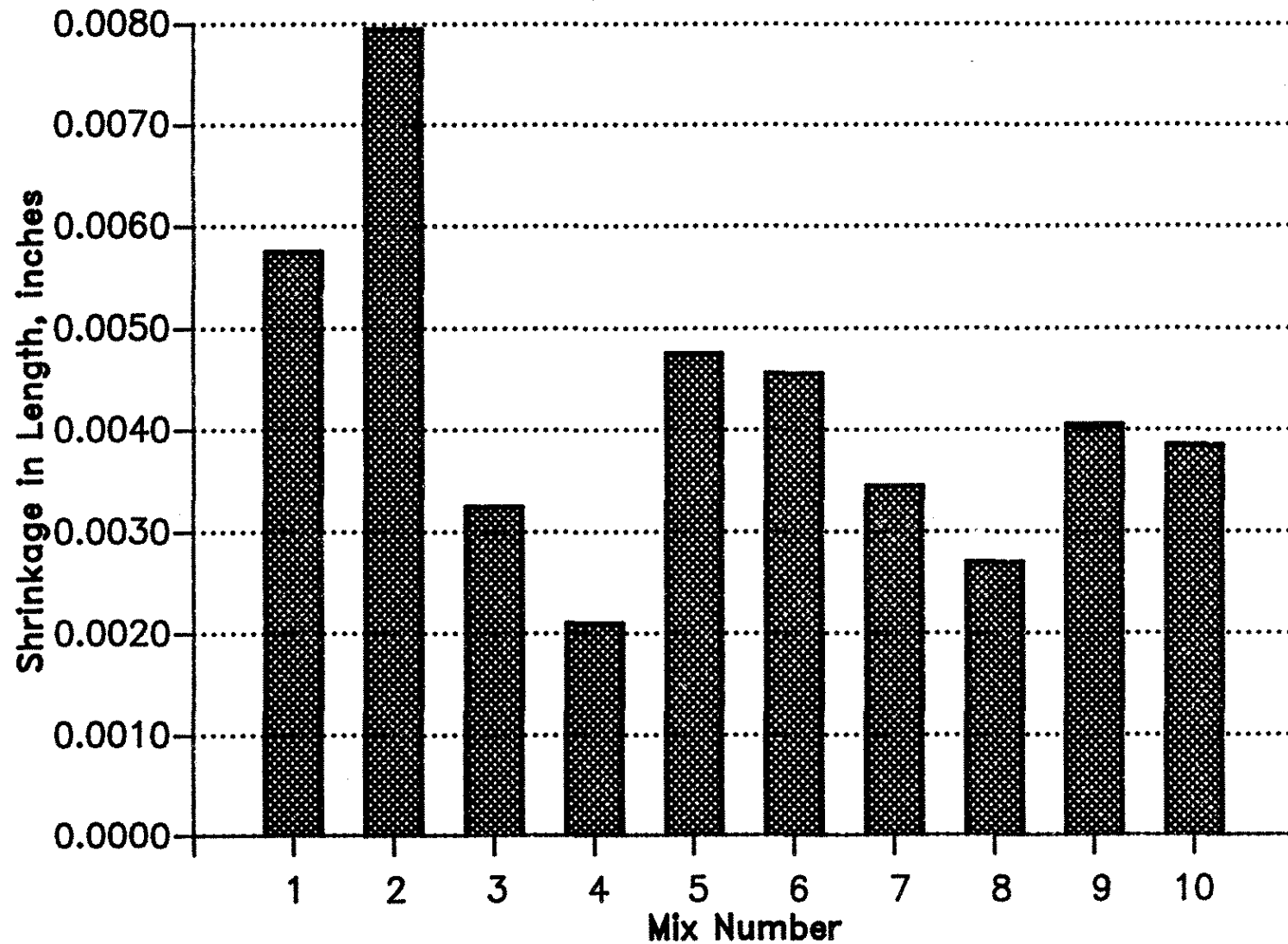


Figure 4

OBSERVATIONS

Some observations noted from results in Figures 1, 2, 3 and 4 were:

Figure 1 - Mixes Nos 6, 9 and 10 all have silica fume. A very interesting observation here is that all three of the mixes show a continuous increase in chloride permeability with time by the AASHTO T 277-89 test.

Mix No. 8, D-57-F with latex has the lowest chloride permeability, being very low in all three tests done at 90, 180 and 360 days by the AASHTO T 277-89 test.

Mixes Nos 1 and 2, the D-57 standard and the 0-4 IA dense, respectively, showed the highest chloride permeability by the AASHTO T 277 test.

Figure 2 - Mix No. 6, D-57 with 10% silica fume has the lowest overall percent chloride content by the AASHTO T 259 90-day salt ponding test.

Mix No. 8, D-57-F with latex has nearly the highest overall chloride content by the AASHTO T 259 test. The opposite indication was given by the AASHTO T 277 test.

There is not a consistent correlation of permeability to chlorides or chloride contents between the two test methods used for many mixes in this test.

Figure 3 - The three mixes with silica fume, No. 6, 9 and 10, had the highest values for compressive strength.

Mix No. 5, D-57-F, with 30% fly ash and 497 lbs/yd³ cement has the lowest 28-day compressive strength. Although comparatively the lowest, it still had the fairly high value of 5540 psi.

Figure 4 - Mix No. 2, 0-4 IA dense, showed the highest amount of shrinkage being 0.008 in. Mix No. 4, D-57 with latex, had the lowest amount of shrinkage, being 0.002 in.

From Table 2, the No. 2 mix, which is the commonly used 0-4 Iowa dense, has a material cost of \$37.37/yd³. Mix No. 5, D-57-F, has the lowest materials cost of \$28.52/yd³. This cost is 24% less than the Iowa dense mix currently used.

DISCUSSION OF RESULTS

90-Day Salt Ponding Test

The results of the chloride-ion content 90-day salt ponding tests are shown in Figure 2. At depths near the surface, 1/16" to

1/2", there were some irregular results. The 0-4 with latex mix had the highest chloride content. The D-57 with silica mix gave the best results at this depth. All of the mix designs exceeded the threshold value of 1.5 lbs chloride per cubic yard concrete at this depth.

At the depths between 1/2" and 1" only three of the mixes exceeded the threshold value. They were the D-57 mix, the 0-4 with latex mix and the D-57-F with latex mix. The 0-4 with latex mix showed higher permeability than the 0-4 mix without latex, and the D-57-F with latex mix permeability was also higher than the D-57-F mix without latex.

At the 1" to 1½" depths, all of the mixes were below the threshold value. The 0-4 with latex mix had the best results at this depth, however, it had poor values above this depth.

Rapid Determination Chloride Test

From results of AASHTO T 277 test, as shown in Figure 1, the chloride permeability of Mix Nos. 6, 9 and 10 were continuously increasing with time. This observation of increasing permeability should certainly be investigated further. It should also be noted that all 3 mixes which showed the increase in permeability with time contained silica fume and also, all three of these mixes contained super water reducer. This raises the question about long term effects on permeability from use of

silica fume and/or super water reducer. An increase in permeability could occur, over time, if there was also some proportional reduction in surface tension occurring, over time, within the system of pore channels.

CONCLUSIONS

From chloride content ponding test, Mix No. 6, D-57 with silica fume, had the lowest overall chloride content, as shown in Figure 2. However, the results of chloride permeability tests from AASHTO T 277-89 show continuously increasing permeability over the time span of 1 year for all 3 mixes having silica fume. As a result of this finding, these 3 mixes will not be proposed for field tests in this report.

Test results from AASHTO T 277-89 show that mix No. 8, D-57-F with latex, is a good mix as it has very low permeability. The material cost of this mix is not excessively high as only 1/2 of the prescribed amount of the expensive latex was used. The lower cost fly ash was added to compensate for the reduced amount of latex.

Another good mix with low permeability is mix No. 5, D-57-F. It also has the benefit of having the lowest material cost. With some adjustments in amounts of cement and fly ash, the compressive strength could be increased, if required, while still maintaining a very economical mix.

Both of the above proposed mixes, No. 8 and No. 5, have a much lower permeability than the commonly used Iowa dense mix No. 2, as shown in Figure 1.

Previous laboratory work done to compare concrete permeability to chloride by AASHTO T 259 and AASHTO T 277 tests gave results which were considered to have good correlation (4). Some test results found in this study showed chloride permeability to be extremely low by the AASHTO T 277 test method but not low by the AASHTO T 259 test method for the same mix design. The test results correlation was not real good, especially in Mix No. 8 when using the two different test methods.

Based upon costs given in Tables 1 and 2, the increase in cost for materials in Mix No. 8 which gave very low permeability (AASHTO T 277) compared to the Iowa dense Mix No. 2 would be \$1744.00, based on a 2" overlay for a 30' x 200' bridge deck using 37 yd³ concrete.

The reduction in materials cost from use of mix No. 5 compared to the Iowa dense mix would be \$327.00 on a 30' x 200' bridge deck. However, the main benefit from this mix would be the lower permeability of the concrete.

RECOMMENDATIONS

1. Based upon indications of increasing permeability over time for all mixes having silica fume and super water reducer, mixes Nos. 6, 9 and 10, it is recommended to run additional similar tests for evaluation beyond one year.
2. A bridge deck overlay using Mix No. 8, with fly ash and latex and mix No. 5, with fly ash, should be tried under a research project and compared with Mix No. 2, the Iowa dense design, possibly all on the same bridge. The AASHTO T 277 test indicated chloride permeability was much lower in the Mix No. 8 than in mix No. 2, the Iowa dense. Compared to mix No. 2, the increase in material cost for using Mix No. 8 on a bridge deck overlay 30' x 200' x 2", using 37 yd³ would be \$1744.00. The reduction in material cost when using mix No. 5 on the same deck should be \$327.00. An additional benefit from mix No. 5, beyond the economics, would be the very low permeability.

ACKNOWLEDGEMENTS

Appreciation is extended to the Iowa Department of Transportation, Materials Department, especially the Materials Laboratory, for their support in laboratory analysis. Special appreciation also goes to Kathy Davis and Steve Juhlin for their support in preparing this report.

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Appendix A
Properties of Concrete Mixes Used

Properties of Concrete Mixes Used													
Mix #	Type of Mix	Date Mixed	Cement lbs/yd ³	Fly Ash lbs/yd ³	Silica Fume lbs/yd ³	Latex gal/yd ³	F Agg lbs/yd ³	C Agg lbs/yd ³	Add-Mix oz/yd ³	W/C Ratio	Slump in.	Air %	Density lb/ft ³
1	D-57	4-17-89	710	---	---	---	1404	1404	---	.381	2.0	5.8	144.6
2	O-4	4-25-89	823	---	---	---	1404	1404	24.6 oz WR	.342	1.0	5.9	144.6
3	D-57 w/Latex	5-15-89	710	---	---	13.21	1404	1404	---	.289	2.25	3.6	148.2
4	D-57 w/Latex	5-17-89	710	---	---	26.42	1404	1404	---	.223	3.0	3.6	148.2
5	D-57-F	4-27-89	497	266	---	---	1404	1404	21.3 oz WR	.334	1.75	6.0	142.6
6	D-57 w/Silica	6-5-89	710	---	71	---	1404	1404	99.4 oz SWR	.266	1.25	5.5	145.4
7	O-4 /w Latex	5-22-89	823	---	---	15.3	1404	1404	---	.256	1.5	4.0	147.4
8	D-57-F /w Latex	5-24-89	497	266	---	13.21	1404	1404	21.3 oz WR	.250	1.5	3.4	148.2
9	C-4 /w Silica	6-7-89	624	---	71	---	1404	1404	87.2 oz SWR	.288	1.5	5.2	145.8
10	D-57-F /w Silica	6-14-89	639	89	71	---	1404	1404	99.4 oz SWR	.250	2.0	5.5	143.8

Appendix B
Coarse Aggregate Gradations for Mixes

Aggregate Gradation
for Mix Nos.
1, 3, 4, 5, 6, 8, 9, and 10

<u>Sieve No.</u>	<u>% Passing</u>
1"	100
3/4"	77
1/2"	40
3/8"	12
#4	.5
#8	.3
#200	0

Aggregate Gradation for
Mix Nos.
2 and 7

<u>Sieve No.</u>	<u>% Passing</u>
1"	100
3/4"	100
1/2"	100
3/8"	83
#4	10
#8	.5
#200	0

Appendix C
Test Results

Appendix C-1 - Rapid Determination of the Chloride Permeability
of Concrete, AASHTO 277-89

Appendix C-2 - Resistance of Concrete to Chloride-Ion
Penetration, AASHTO T 259-80

Appendix C-3 - Compressive Strength (28 Day)
and 18" Beam Drying Shrinkage (180 Day)

Appendix C-1

Rapid Determination of the Chloride Permeability of Concrete
By AASHTO T 277-89

Mix #	Type of Mix	W/C Ratio	Slump in.	Air %	Density	90 Days Coulombs Amp Sec	CL Perm. Standard	180 Days Coulombs Amp Sec	CL Perm. Standard	3600 Days Coulombs Amp Sec	CL Perm. Standard
1	D-57	.381	2.0	5.8	144.6	1190	Low	2074	Moderate	1600	Low
2	O-4	.342	1.0	5.9	144.6	1600	Low	2080	Moderate	1253	Low
3	D-57 /w Latex	.289	2.25	3.6	148.2	650	Very Low	454	Very Low	238	Very Low
4	D-57 /w Latex	.223	3.0	3.6	148.2	280	Very Low	173	Very Low	86	Negligible
5	D-57-F	.334	1.75	6.0	142.6	345	Very Low	238	Very Low	194	Very Low
6	D-57 /w Silica	.266	1.25	5.5	145.4	173	Very Low	216	Very Low	268	Very Low
7	O-4 /w Latex	.256	1.5	4.0	147.4	670	Very Low	346	Very Low	151	Very Low
8	D-57-F /w Latex	.250	1.5	3.4	148.2	130	Very Low	65	Negligible	22	Negligible
9	C-4 /w Silica	.288	1.5	5.2	145.8	194	Very Low	216	Very Low	346	Very Low
10	D-57-F /w Silica	.250	2.0	5.5	143.8	151	Very Low	173	Very Low	194	Very Low

Appendix C-2 Resistance of Concrete to Chloride-Ion Penetration AASHTO T 259-80									
Mix #	Type of Mix	W/C Ratio	Slump in.	Air %	Density lbs/ft ³	% Chloride Content by Weight Each Depth From Top of Slab			
						1/16"	1/8"	1/4"	Base
1	D-57	.381	2	5.8	144.6	0.347	0.069	0.023	0.021
2	O-4	.342	1.0	5.9	144.6	0.230	0.029	0.021	0.018
3	D-57 /w Latex	.289	2%	3.6	148.2	0.225	0.033	0.023	0.020
4	D-57 /w Latex	.223	3.0	3.6	148.2	0.226	0.029	0.021	0.022
5	D-57-F	.334	1%	6.0	142.6	0.236	0.026	0.023	0.022
6	D-57 /w Silica	.266	1.25	5.5	145.4	0.180	0.025	0.023	0.022
7	O-4 /w Latex	.256	1.5	4.0	147.4	0.370	0.042	0.020	0.021
8	D-57-F /w Latex	.250	1.5	3.4	148.2	0.344	0.053	0.026	0.029
9	C-4 /w Silica	.288	1.5	5.2	145.8	0.232	0.029	0.027	0.024
10	D-57-F /w Silica	.250	2.0	5.5	143.8	0.294	0.033	0.029	0.024

Appendix C-3

Compressive Strength (28 Day)
and 18" Beam Drying Shrinkage (180 Day)

Mix #	Type of Mix	W/C Ratio	Slump in.	Air %	Density	28 Day Compression Strength (psi)	180 Day Shrinkage in.	180 Day Weight Loss (ounces)
1	D-57	.381	2.0	5.8	144.6	6650	.00575	9.51
2	O-4	.342	1.0	5.9	144.6	7740	.00795	10.4
3	D-57 /w Latex	.289	2.25	3.6	148.2	7780	.00325	5.08
4	D-57 /w Latex	.223	3.0	3.6	148.2	7360	.00210	3.00
5	D-57-F	.334	1.75	6.0	142.6	5540	.00475	7.71
6	D-57 /w Silica	.266	1.25	5.5	145.4	8970	.00455	7.39
7	O-4 /w Latex	.256	1.5	4.0	147.4	7840	.00345	4.83
8	D-57-F /w Latex	.250	1.5	3.4	148.2	7450	.00270	4.09
9	C-4 /w Silica	.288	1.5	5.2	145.8	9010	.00405	7.46
10	D-57-F /w Silica	.250	2.0	5.5	143.8	8550	.00385	7.50

Appendix D
AASHTO T 259-80

*Standard Method of Test
for*

Resistance of Concrete to Chloride Ion Penetration

AASHTO DESIGNATION: T 259-80 (1990)

1. SCOPE

1.1 This method covers the determination of the resistance of concrete specimens to the penetration of chloride ion. It is intended for use in determining the effects of variations in the properties of concrete on the resistance of the concrete to chloride ion penetration. Variations in the concrete may include, but are not limited to, changes in the cement type and content, water-cement ratio, aggregate type and proportions, admixtures, treatments, curing and consolidation. This test method is not intended to provide a quantitative measure of the length of service that may be expected from a specific type of concrete.

2. TEST SPECIMENS

2.1 The specimens for use in this test shall be slabs made and cured in accordance with the applicable requirements of AASHTO T 126, "Making and Curing Concrete Test Specimens in the Laboratory."

NOTE 1—This method contemplates the use of a minimum of four specimens for each evaluation with each slab not less than 3 inches (76 mm) thick and 12 inches (305 mm) square.

2.2 For this test the specimens shall be removed from moist curing at 14 days of age unless earlier removal is recommended by the manufacturer of a special concrete. The specimens shall then be stored until 28 days of age in a drying room of the type specified by AASHTO T 160, Length Change of Cement Mortar and Concrete.

2.3 When the test method is used to evaluate concrete treatments, the slabs shall be fabricated from concrete having a cement factor of 658 lbs (229 kg) per cubic yard (0.76m³), a water-cement ratio

by weight of 0.5, and an air content of 6 ± 1 percent.

The concrete treatment shall be applied at 21 days of age and in accordance with the manufacturer's recommendations for field usage.

NOTE 2—If field application of a sealer by spraying is recommended, the sealer should be applied to the specimens by spraying rather than brushing.

2.4 When a special overlay material is to be evaluated, the concrete slab shall be cast 2 inches (51 mm) thick using the mix design specified under Section 2.3 and then the special overlay material shall be placed 1 inch (25 mm) thick, unless specified otherwise, according to the manufacturer's recommendations.

3. PROCEDURE

3.1 Immediately after the specified drying period stipulated in Section 2.2 (i.e. 29th day of specimen age), 0.125 ± 0.625 in. (3.2 ± 1.6 mm) of the slab surface shall be abraded using grinding or sand-blasting techniques if the concrete or treatment are to be subjected to the wearing effect of vehicular traffic. No water shall be used in the abrading process. If the concrete or treatment is to be used on surfaces not subject to wear from vehicular traffic then the abrading step shall be omitted.

3.2 Place approximately 0.75 in. (19 mm) high by 0.5 in. (13 mm) wide dams around the top edge of all slabs except one, which will then become the control slab. In lieu of this, a dam meeting these dimension requirements may be cast as an integral part of the slab. However, such previously cast dams shall not interfere with the abrasion of the surface as specified under Section 3.1.

3.3 All slabs shall then be returned to

the drying room as specified under Section 2.2 for an additional 13 days (i.e. until 42 days of age).

NOTE 3—The degree of saturation of the specimens at the time of ponding will affect chloride ingress. In general, water saturated concrete will absorb significantly less chloride during the 90 days of ponding than a drier but similar material. Thus, for proper definition of chloride ingress by this method, the requirements in Sections 2.2 and 3.1 through 3.3 (for a total of 28 days of air drying prior to ponding) must be followed.

3.4 The slabs with dams shall be subjected to continuous ponding with 3-percent sodium chloride solution to a depth of approximately 0.5 in. (13 mm) for 90 days. Glass plates shall be placed over the ponded solutions to retard evaporation of the solution. Placement of the glass plates shall not be done in such a manner that the surface of the slab is sealed from the surrounding atmosphere. Additional solution shall be added if necessary to maintain the 0.5 in. (13 mm) depth. All slabs shall then be returned to the drying room as specified under Section 2.2.

3.5 After 90 days of exposure the solution shall be removed from the slabs. The slabs shall be allowed to dry and then the surfaces shall be wire brushed until all salt crystal buildup is completely removed.

3.6 Samples for chloride ion analysis shall then be taken from all slabs in accordance with the procedure described in AASHTO T 260. These samples shall be obtained from each slab at each of the following depths unless otherwise directed by the specifying agency:

0.0625 in. (1.6 mm) to 0.5 in. (13 mm)
0.5 in. (13 mm) to 1.0 in. (25 mm)

The chloride content of each sample shall be determined in accordance with the instructions in AASHTO T 260.

NOTE 4—Many starter bits for use inside hollow rotary hammer pulverizing bits are significantly longer than the pulverizing bit. This results in a sampling depth which is greater in the center of the "core hole" than at the edges. To minimize this effect, the chuck end of the starter bit should be cut off such that its overall length does not exceed that of the outside bit by more than $\frac{1}{16}$ in. (i.e., such that the variation in sampling depth is not greater than $\frac{1}{16}$ in.). When it is desired to remove only $\frac{1}{16}$ in. (1.6 mm) of material from the surface of a test specimen or a bridge deck, it may be more convenient to complete that operation by use of a grinder. The sample can then be taken with the rotary hammer without fear of contamination from the salt on the surface of the item being sampled.

4. CALCULATIONS

4.1 The baseline chloride ion content for the test specimens shall be determined as the average chloride ion content of samples obtained from the 0.0625 in. (1.6 mm) to 0.5 in. (13 mm) and 0.5 in. (13 mm) to 1.0 in. (25 mm) depths within the slab that was not ponded with 3 percent sodium chloride solution.

4.2 The absorbed chloride ion content of each sample from the ponded slabs shall be determined as the difference between the total chloride ion content of that sample and the baseline value calculated in Section 4.1. If the result is less than zero, the result shall be reported as

zero. The average chloride ion absorbed at each sampling depth shall be calculated.

5. REPORT

5.1 Reporting shall include (1) each total chloride ion value determined in Section 3.6, (2) the average and maximum baseline chloride ion (Section 4.1), (3) each calculated absorbed chloride ion value determined in Section 4.2, (4) the average and maximum absorbed chloride ion values calculated in Section 4.2 for each depth, (5) a statement detailing whether or not the surface abrasion described in Section 3.1 was performed.

Appendix E
AASHTO T 260-84

Sampling and Testing for Total Chloride Ion in Concrete and Concrete Raw Materials

AASHTO DESIGNATION: T 260-84

1. SCOPE

1.1 This method covers procedures for the determination of the total chloride ion content or the water-soluble chloride ion content of aggregates, portland cement, mortar or concrete. The method is limited to materials that do not contain sulfides.

1.2 The age of concrete mortar, or hydrated portland cement at the time of sampling will have an effect on the water-soluble chloride ion content. Therefore, unless early age studies are desired, it is recommended that the material be well cured and at least 28 days of age before sampling.

1.3 This Standard provides for the determination of chloride ion content by two procedures: Procedure A, Determination of Total Chloride Ion Content and Water-Soluble Chloride Ion Content by Potentiometric Titration or Ion-Selective Electrode and Procedure B, Total Chloride Ion by Atomic Absorption.

PROCEDURE A—Total Ion and Water-Soluble Ion by Potentiometric Titration or Ion Selective Electrode

2. APPARATUS

2.1 Equipment for two methods of sampling are listed in Sections 2.1.1 or 2.1.2.

2.1.1 Core drill.

2.1.2 Rotary impact type drill with a depth indicator and drill or pulverizing bits of sufficient diameter to provide a representative sample of sufficient size for testing.

2.1.2.1 Sample containers capable of maintaining the sample in an uncontaminated state.

2.1.2.2 Spoons of adequate size to

collect the sample from the drilled holes.

2.1.2.3 A "blow out" bulb or other suitable means of removing excess pulverized material from the hole prior to re-drilling operations.

2.1.2.4 A device capable of determining the location and depth of steel reinforcement to $\pm \frac{1}{8}$ in. (± 3 mm).

2.2 Equipment for Chemical Testing

2.2.1 Chloride ion or silver/sulfide ion selective electrode and manufacturer-recommended filling solutions.

NOTE 1—Suggested electrodes are the Orion 96-17 Combination Chloride Electrode or the Orion 94-6 Silver/Sulfide Electrode or equivalents. The Silver/Sulfide electrode requires use of an appropriate reference electrode (Orion 90-02 or equivalent).

2.2.2 A millivoltmeter compatible with the ion electrode.

NOTE 2—Suggested millivoltmeter is the Orion Model 701 A Digital pH/mv meter or equivalent.

2.2.3 Magnetic stirrer and teflon stirring bars.

2.2.4 Burette with 0.1 ml graduations.

2.2.5 Balance complying with M 231, Class A.

2.2.6 Balance complying with M 231, Class G 2.

2.2.7 Hot plate, 250 to 400 C heating surface temperature.

2.2.8 Glassware, 100 and 250 ml beakers, filter funnels, stirring rods, watch glasses, dropper, wash bottles.

2.2.9 Sieve, U.S. Standard No. 50 (0.300 mm).

2.2.10 Whatman No. 40 and No. 41 filter papers (or equivalent).

NOTE 3—If equivalent filter papers are used, they should be checked to confirm they do not contain chloride which will contaminate the sample.

3. REAGENTS

3.1 Concentrated HNO_3 (sp gr 1.42).

3.2 Sodium chloride, NaCl , reagent grade (primary standard).

3.3 Standard 0.01 N NaCl solution. Dry reagent grade NaCl in an oven at 105 C. Cool, in a dessicator, weigh out approximately 0.5844 to the nearest 0.0001 gram, dissolve in distilled H_2O , and transfer to a 1 litre volumetric flask. Make up to the mark with distilled H_2O and mix. Calculate the exact normality as follows:

$$N_{\text{NaCl}} = (0.0100) \frac{W_{\text{actual}}}{0.5844}$$

W_{actual} = actual weight of NaCl
 N_{NaCl} = normality of NaCl solution

3.4 Standard 0.01 N AgNO_3 . Weigh 1.7 grams of reagent AgNO_3 , dissolve in distilled H_2O , filter into a 1 litre brown glass bottle, fill, and mix thoroughly. Standardize against 25.00 ml of the NaCl solution by the titration method given in Section 5.4. Calculate the exact normality as follows:

$$N_{\text{AgNO}_3} = \frac{(V_{\text{NaCl}})(N_{\text{NaCl}})}{V_{\text{AgNO}_3}}$$

N_{AgNO_3} = Normality of AgNO_3 Solution
 V_{NaCl} = Volume (ml) of NaCl Solution
 N_{NaCl} = Normality of NaCl Solution
 V_{AgNO_3} = Volume (ml) of AgNO_3 Solution

3.5 Distilled Water.

NOTE 4—Deionized water may be used in place of distilled water for samples where extreme precision and accuracy are not demanded.

3.6 Methyl orange indicator.

3.7 Ethanol, denatured or methanol, technical.

4. METHOD OF SAMPLING

4.1 Concrete Sample:

4.1.1 Determine the depth within the concrete for which the chloride content is desired.

NOTE 5—A convenient method of determining the location and depth of reinforcement bars is a pachometer capable of determining the location and depth of steel reinforcement to $\pm \frac{1}{8}$ in. (± 3 mm).

4.1.2 Core Method—Drill the core to chosen depth and retrieve.

4.1.2.1 When samples are received in the laboratory in other than pulverized condition, the sample shall be crushed and ground to a powder. All sawing or crushing shall be done dry (i.e. without water). All material shall pass a number 50 (0.300 mm) sieve. All pulverizing tools and sieves shall be washed with alcohol or distilled water and shall be dry before use with each separate sample (see note Section 4.1.3.7).

4.1.3 Pulverizing Method:

4.1.3.1 Set the rotary hammer depth indicator so that it will drill to $\frac{1}{2}$ in. (13 mm) above the desired depth.

4.1.3.2 Using a drill or pulverizing bit, drill until the depth indicator seats itself on the concrete surface.

4.1.3.3 Thoroughly clean the drilled hole and surrounding area utilizing the "blow out" bulb or other suitable means.

4.1.3.4 Reset the depth indicator to permit $\frac{1}{2}$ in. (13 mm) additional drilling.

4.1.3.5 Pulverize the concrete until the depth indicator again seats itself on the concrete.

NOTE 6—Care must be exercised during this pulverizing operation to prevent the drill bit from abrading concrete from the sides of the hole above the sampling depth. To insure against this, some users utilize an 0.25 in. (6 mm) smaller diameter bit in this step than that used in Section 4.1.3.2.

4.1.3.6 Collect at least 10 grams of the material remaining in the hole using a spoon and place in the sample container.

4.1.3.7 If the sample, as collected, does not completely pass a No. 50 (0.300 mm) sieve, additional pulverizing shall be performed in the laboratory until the entire sample is finer than 0.300 mm (No. 50 sieve).

NOTE 7—During sample collection and pulverizing, personnel shall use caution to prevent contact of the sample with hands, or other sources of body perspiration or contamination. Further, all sampling tools (drill bits, spoons, bottles, sieves, etc.) shall be washed with alcohol or distilled water and shall be dry prior to use on each separate sample. Alcohol is normally preferred for washing because of the rapid drying which naturally occurs.

4.2 Raw Material Sample:

4.2.1 Cement samples shall be taken and prepared as prescribed in AASHTO T 127, Sampling Hydraulic Cement.

4.2.2 Coarse and fine aggregate samples shall be taken as prescribed in AASHTO T 2, Sampling Stone, Slag, Gravel, Sand and Stone Block for Use in Highway Materials. Samples shall be reduced in accordance with AASHTO T 248, Reducing Field Samples of Aggregate to Testing Size.

4.2.3 Test samples shall contain the following minimum sizes:

cement—100 g, Sand—300 g,
coarse aggregate—3,000 g

4.2.4 Coarse aggregate samples shall be crushed to pass a No. 4 (4.75 mm) sieve and then reduced down to about 300 g. The final 300 g of coarse or fine aggregate shall be ground to a minus No. 50 (0.300 mm) sieve.

5. PROCEDURE

Two distinct procedures are presented here for determination of total chloride ion or water-soluble chloride ion content. For total chloride ion content follow 5.1 and 5.2, then continue with Section 5.4. For water-soluble chloride ion content follow 5.1 and 5.3, then continue with Section 5.4.

5.1 Weigh to the nearest milligram a 3 g powdered sample representative of the material under tests.

NOTE 8—Some users dry the sample to constant weight in a 105 C oven and determine the dry sample prior to analysis. This optional procedure provides a constant base for comparison of all results by eliminating moisture content as a variable. It is generally believed that drying is only necessary when very high accuracy is desired (see Reference 1 for data in this area).

5.2 Procedure for Total Chloride Ion Content:

5.2.1 Transfer the sample quantitatively to a beaker, add 10 ml of distilled H_2O , swirling to bring the powder into suspension. Add 3 ml of concentrated HNO_3 with continued swirling until the material is completely decomposed. Break up any lumps with a stirring rod and dilute with hot H_2O to 50 ml. Stir thoroughly to ensure complete sample digestion. Add five drops of methyl orange indicator and stir. If yellow to yellow-orange color appears, solution is not sufficiently acidic. Add additional concentrated HNO_3 dropwise with continuous stirring until a faint pink or red color persists in the solution. Cover with a watch glass, retaining the stirring rod in the beaker. Heat the acid solution or slurry to boiling on a hot plate at medium heat (250 to 400 C) and boil for about 1 minute. Remove from the hot plate, filter through double filter paper (Whatman No. 41 over No. 40 filter paper or equivalent).

5.2.2 Wash the filter paper ten times with hot distilled H_2O , being careful not to lift the paper away from the funnel surface. Finally, lift the filter paper carefully from the funnel and wash the outside surface of the paper with hot distilled H_2O ; then wash the tip of the funnel. The final volume of the filtered solution should be 125 to 150 ml. Cover with a watch glass and allow to cool to room temperature in an HCl fume-free atmosphere.

NOTE 9—Due to the presence of relatively insoluble materials in the sample, the solution generally will have a strong gray color, making the detection of indicator color difficult at times. Running of several trial samples is suggested to give the analyst practice in detecting the indicator color.

NOTE 10—A sample prepared to 100 percent passing 0.300 mm (No. 50 sieve) should generally allow determination of any expected chloride level with adequate precision an accuracy. Samples containing highly siliceous aggregates may require finer grinding to minimize bumping during step 5.2. This may also be the case when the concrete contains modifiers such as latex or polymer.

5.3 Procedure for Water-Soluble Chloride Ion Content:

5.3.1 Transfer the sample quantitatively to a beaker, add 60-70 ml distilled H_2O . Cover the beaker with a watch glass and bring to a boil on a hot plate-magnetic

stirrer using a small magnet. Boil for 5 minutes, then let stand for 24 hours in an HCl fume-free atmosphere.

5.3.2 Filter the clear supernatant liquid in the beaker through double filter paper (Whatman No. 41 over No. 40 or equivalent) into a 250 ml beaker; take care to quantitatively transfer any adhering drops on the watch glass, and use a stirring rod to aid transfer. Add sufficient hot distilled H₂O to cover any residue left in the original beaker, stir 1 minute on a magnetic stirrer, and filter into the 250 ml beaker with a swirling action. Wash the beaker and the stirring rod once into the filter with hot distilled H₂O. Wash the filter paper *once* with hot distilled H₂O. Lift the filter paper carefully from the funnel and wash the outside surface of the paper with hot distilled H₂O. Set aside the paper and wash the interior of the funnel and its tip with hot distilled H₂O. Finally, add 1-2 drops of methyl orange indicator to the 150 ml beaker; then add concentrated HNO₃ dropwise with continuous stirring until a permanent pink to red color is obtained. Make up the volume to 125 to 150 ml with distilled H₂O.

5.4 Three alternate methods are available to determine the Cl⁻ content of the solution. All methods utilize an ion selective electrode (Cl⁻ or Ag⁺) and all methods for the purpose of this analysis give results of essentially equal accuracy and precision.

5.4.1 Method 1: Potentiometric Titration—Fill the Cl⁻ or the Ag⁺ electrode with the solution(s) recommended by the manufacturer, plug it into the millivoltmeter (preferably the type with a digital rather than a dial readout), and determine the approximate equivalence point by immersing the electrode in a beaker of distilled H₂O. Note the approximate millivoltmeter reading (which may be unsteady in H₂O). Take the cooled sample beaker from Section 5.3 and carefully add 4.00 ml of 0.0100 N NaCl, swirling constantly. Remove the beaker of distilled H₂O from the electrode, wipe the electrode with absorbent paper, and immerse the electrode in the sample solution. Place the entire beaker-electrode assembly on a magnetic stirrer and begin gentle stirring. Using a calibrated buret, add gradually and record the amount of standard 0.01 N AgNO₃ solution necessary to bring the millivoltmeter reading to -40 mv of the equivalence point determined in distilled

H₂O. Then add standard 0.01 N AgNO₃ solution in 0.10 ml increments recording the millivoltmeter reading after each addition.

As the equivalence point is approached, the equal additions of AgNO₃ solution will cause larger and larger changes in the millivoltmeter reading. Past the equivalence point, the changes per unit volume will again decrease. Continue the titration until the millivoltmeter reading is at least 40 mv past the approximate equivalence point.

The endpoint of the titration usually is near the approximate equivalence point in distilled water and may be determined by (1) plotting the volume of AgNO₃ solution added versus the millivoltmeter readings. The endpoint will correspond to the point of inflection of the resultant smooth curve, or (2) calculating the differences in millivoltmeter readings between successive AgNO₃ additions and calculating the total volume of AgNO₃ which corresponds with each difference (i.e., the midpoints between successive additions).

Raw Data		Differences	
Titrant Volume	Millivolt Reading	Titrant Midpoints	Millivolt Difference
4.2 ml	130.0	4.25 ml	5.0
4.3 ml	135.0	4.35 ml	7.0
4.4 ml	142.0	4.45 ml	10.0
4.5 ml	152.0	etc.	

The endpoint will be near the midpoint which produced the largest change in millivoltmeter reading. It may be determined by plotting midpoints versus differences and defining the AgNO₃ volume which corresponds to the maximum difference on a smooth, symmetrical curve drawn through the points. However, it can usually be estimated accurately without plotting the curve by choosing the midpoint which corresponds to the maximum difference and adjusting for asymmetry, if any. In other words, if the differences on each side of the largest difference are not symmetrical, adjust the endpoint mathematically in the direction of the largest differences. Detailed examples of this adjustment are contained in Reference 1.

5.4.1.1 Calculations:

Determine the endpoint of the titration as described in Section 5.4.1 by either plotting a curve or estimating from the

numerical data. Calculate the percent Cl⁻ ion from the equation:

$$\text{Cl}^- \text{ percent} = \frac{(3.5453 (V_1 N_1 - V_2 N_2))}{W}$$

where:

V_1 = endpoint in ml of AgNO₃

N_1 = normality of AgNO₃

W = Weight of original concrete sample in grams

V_2 = Volume of NaCl solution added, in ml

N_2 = Normality of NaCl solution

5.4.2 Method II: Gran Plot Method—

This method is compatible with either a Cl⁻ or Ag⁺ ion selective electrode. Attach the electrode of choice to a compatible digital millivoltmeter after filling the required solutions as per the electrode manufacturer's instructions. Clean the electrode with distilled H₂O and pat dry with absorbent paper.

Weigh the solution and beaker from Section 5.3 without the watch glass and record the weight. Using a calibrated buret, titrate the sample to 225 mv ± 5 mv (Cl⁻ electrode) or 310 mv ± 5 mv (Ag⁺ electrode) with standard 0.01N AgNO₃ solution. Record the volume added and the millivoltmeter reading.

Continue to titrate in 0.50 ml increments recording the volume added and the millivoltmeter reading for each increment. Add and record the data for at least five increments. Empty, clean, dry and weigh the beaker. Subtract beaker weight from beaker + solution weight determined above to define solution weight.

Example shown in Figure 1. Additional information on the Gran Method is given in Reference 2.

5.4.2.1 Gran Method Calculations:

Calculate corrected values for each of the volumes recorded in Section 5.4.2 by the equation:

$$V_{\text{correct}} = \frac{V_{\text{record}}}{W/100}$$

W = Original solution weight in grams

V_{record} = Volumes recorded in ml

If any of the V_{correct} values are greater than 10, see Section 5.4.2.2. If less than 10, plot these corrected values versus the corresponding millivolt readings on Orion Gran Plot Paper (10 percent

volume corrected type with each major vertical scale division equal to 5 millivolts) or equivalent. Draw the best straight line through the points and read the endpoint at the intersection of the line with the horizontal axis of the graph. Calculate the actual endpoint by the equation:

$$E_a, \text{ ACTUAL ENDPOINT} = E_g \left(\frac{W}{100} \right)$$

where:

E_g = Endpoint determined from graph in ml

W = Weight of solution in grams

$$\text{Then percent Cl} = \frac{3.5453 E_a N}{W_c}$$

where:

E_a = Actual endpoint, in ml

N = Normality of AgNO_3 solution

W_c = Concrete sample weight in grams

5.4.2.2 Supplementary Gran Method Calculations:

When the V correct volumes determined in Section 5.4.2.1 are greater than 10, discard the values and follow the following procedure.

Choose a constant which, when subtracted from all V record volumes, yields values less than 10 ml.

NOTE 11—This constant, designated as X in the formulas below, is normally assigned an even value such as 5, 10, 15, 20, etc.

Calculate a revised solution weight W_r as

$$W_r = W + X$$

where:

W = Original solution in grams

X = The constant.

Then calculate corrected volumes for each recorded volume as:

$$V_{\text{correct}} = \frac{V_{\text{record}} - X}{W_r/100}$$

Plot these values and determine the graph endpoint E_g as described in Section 5.4.2.1. The actual endpoint E_a is then:

$$E_a = E_g \left(\frac{W_r}{100} \right) + X$$

where:

E_a = Actual endpoint in ml

E_g = Endpoint from graph in ml

W_r = Revised solution weight in grams

X = The constant chosen above.

Calculate the chloride content using the formula given in Section 5.4.2.1.

5.4.3 Method III: Automatic Titrator—This method is compatible with either a Cl^- or Ag^+ ion-selective electrode. The millivolt endpoint determination and testing procedure shall be in accordance with the instrument manufacturer's recommendation.

5.4.3.1 Automatic Titrator Calculations:

Having determined the endpoint with the automatic titrator, calculations will be identical with Section 5.4.2.1.

5.5 The percent chloride may be converted to pounds of Cl per cubic yard of concrete as follows:

$$\text{lbs Cl}^-/\text{yd}^3 = \text{percent Cl} \left(\frac{UW}{100} \right)$$

where:

UW = Unit weight of concrete per cubic yard

NOTE 12—A unit weight of 3,915 lbs/yd^3 is often assumed for normal structural weight concrete when the actual unit weight is unknown.

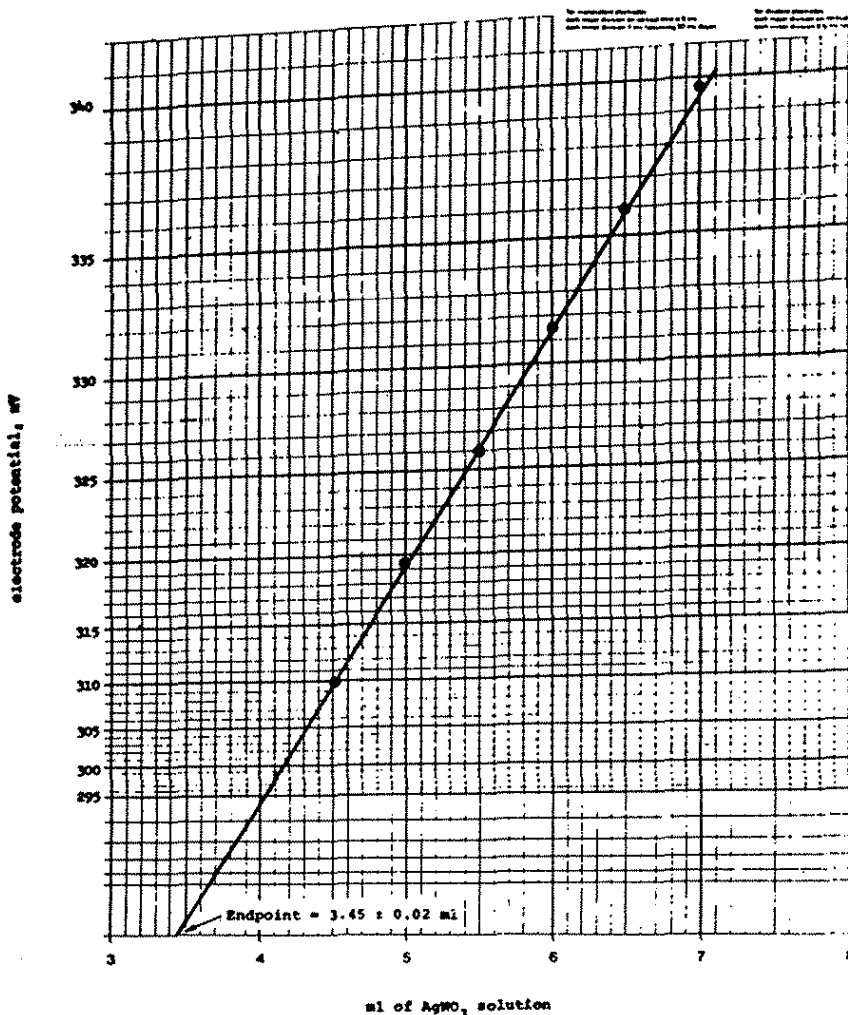


FIGURE 1 Use of Gran Method to Determine Endpoint in the Potentiometric Titration of an Acid Extract of Concrete

6. PRECISION

6.1 The precision statements presented below are based on guidelines presented in ASTM C 670, "Preparing Preci-

sion Statements for Test Methods for Construction Materials." They are, of necessity slightly different since the within-laboratory standard deviation was essentially constant over the chloride levels examined while the between-laboratory precision varied with chloride level. Both statements, however, are based on the difference two-sigma limit, (D2S). The (D2S) index is the difference between two individual test results that would be equaled or exceeded in the long run in only one case in 20 in the normal and correct operation of the chemical analysis.

6.2 Single-Operator—The single-operator standard deviation has been found to be 0.0024 percent chloride². Therefore, results of two properly conducted tests by the same operator on the same material should not differ by more than 0.0068 percent chloride².

6.3 Multilaboratory Precision—Criteria for judging the acceptability of chloride ion concentration test results obtained by different laboratories by this test method are given in the table entitled "Multilaboratory Precision."

PROCEDURE B—Total Ion by Atomic Absorption

7. APPARATUS

7.1 See Section 2.1 for sampling equipment.

7.2 Equipment for Chemical Testing.

7.2.1 Atomic Absorption Spectrophotometer.

NOTE 13—The text of this method is most applicable to the use of a Perkin Elmer Model 503 A.A., with a 2-inch path length burner and digital readout. Laboratories using instruments other than Perkin Elmer should utilize the method to the fullest extent possible.

7.2.2 Millipore filter assembly No. XX1104710 (47 mm in diameter), or equivalent. Millipore filter membrane No. HAWPO4700 (0.45 μ m membrane), or equivalent.

² The numbers represent, respectively, the (1S) and (D2S) limits as described in ASTM Recommended Practice C 670, for Preparing Precision Statements for Test Methods for Construction Materials.

Percent Chloride Concentration	Multilaboratory Precision	
	Standard Deviation ^a	Acceptable Range of Two Results ^a
0.0176	0.0030	0.0085
0.0268	0.0031	0.0088
0.0313	0.0032	0.0091
0.0592	0.0037	0.0105
0.1339	0.0048	0.0136
0.2618	0.0069	0.0195

NOTE—The figures given in Column 2 are the standard deviations that have been found to be appropriate for the chloride ion concentrations described in column 1. The figures given in Column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests.

^a These numbers represent, respectively, the (1S) and (D2S) limits as described in ASTM C 670, Recommended Practice for Preparing Precision Statements for Test Methods for Construction Materials.

NOTE 14—If equivalent filter membranes are used, they should be checked to confirm they do not contain chloride which will contaminate the sample.

7.2.3 100-mL volumetric flasks with glass stoppers (clear glass).

7.2.4 100-mL volumetric flasks (low actinic with glass stoppers).

7.2.5 Pipettes of suitable sizes, which meet or exceed the tolerances specified in NBS circular 602 for Class A Volumetricware.

7.2.6 Analytical balance sensitive to 0.0001 g complying with M 231 Class A.

7.2.7 Fisher filtrator (vacuum) with either a glass or plastic bell jar, tall enough to place a 100-mL volumetric flask underneath.

7.2.8 Hot plates (electric).

7.2.9 Vacuum source.

7.2.10 Vinyl tubing.

8. REAGENTS

8.1 Calcium Carbonate, Reagent Grade.

8.2 Hydrogen Peroxide (30%).

8.3 Methyl Orange.

8.4 Nitric Acid, Concentrated (sp gr 1.42).

8.5 Silver Nitrate, Reagent Grade (primary standard).

8.6 Sodium Chloride, Reagent Grade (primary standard).

8.7 Sodium Nitrate, Reagent Grade.

8.8 Water, Distilled.

9. METHOD OF SAMPLING

See Section 4 for method of sampling.

10. STANDARDIZATION

10.1 Dry a sufficient quantity of standard materials (AgNO₃, NaCl, CaCO₃, and NaNO₃) at 105°C to constant weight. Cool and retain in a desiccator.

10.1.1 Weigh a sufficient sample of each of the above standards to effect the following solutions:

Ag ⁺	— 100 ppm
Cl [−]	— 100 ppm
Na ⁺	— 100 ppm
Ca ⁺⁺	— 500 ppm

10.2 Add 10 mL of 1 + 9 HNO₃ to each of eight 100 mL low actinic volumetric flasks. Aliquot sufficient chloride solution so that each flask will contain a chloride ion concentration of 0.0, 0.1, 0.25, 0.50, 0.75, 1.0, 2.0, and 3.0 ppm respectively. The 0.0 concentration will be the 10 ppm silver standard.

10.3 Proceed with the eight volumetric flasks following Sections 11.7.1 through 11.7.3.

10.4 Set the operating parameters for A.A. in accordance with the manufacturer's procedures when using a silver lamp.

10.5 Aspirate the 10 ppm Ag⁺ standard with a zero chloride ion concentration, and set 10 ppm in the readout window.

10.6 Aspirate the remaining seven volumetrics containing 0.1 through 3.0 ppm chloride ion concentrations, and note the readings. Repeat this procedure at least three times on three different days to obtain an average reading. Develop the standardization data by aspirating randomly.

10.7 Prepare a curve on linear graph paper, plotting remaining Ag^+ ions determined by A.A. against the seven chloride standards (see Figure 2A).

11. PROCEDURE

11.1 The sample as received shall be made to pass a No. 50 sieve, after removing any free iron that may have been introduced in sample preparation. If received sample is excessively large, quartering may be necessary before grinding.

11.2 Dry sample at 105 C to constant weight and cool in dessicator.

11.3 Weigh to the nearest mg a one (1) g powdered sample representative of

the material under test. Transfer to a 150 mL beaker, and add 10 mL of dilute nitric acid (1 + 9) to dissolve as much of the sample as possible. Break up any lumps with a stirring rod.

11.4 If carbonates are present, let sample stand until all effervescence is completed.

11.5 If the solution is not acid at this point, add only enough nitric acid to produce a red color with methyl orange.

11.6 Heat the slurry on a hot plate to just under boiling, and digest for five minutes or until all reaction ceases. Remove from hot plate and cool. Vacuum filter (Fisher filtrator or equivalent through a 0.45 μm membrane (Millipore filter assembly No. XX1104710, Millipore filter membrane No. HAWP04700, or equivalents) into a 100 mL volumetric flask. Wash the precipitate with three or four small portions of distilled water. Dilute to volume (see Figure 2B).

11.7 Aliquot 10 mL of the filtered sample solution into a 100 mL volumetric flask (low actinic). Save balance of the sample; other dilutions may be needed if concentration is relatively high.

11.7.1 Add $\frac{1}{2}$ mL hydrogen peroxide and agitate for one minute.

11.7.2 Add 10 mL of the 100 ppm silver nitrate solution. Agitate the flask once again and let stand for one hour.

11.7.3 Vacuum filter, using above Millipore or equivalent equipment, into a 100 mL volumetric flask (low actinic) containing 5 mL of the sodium solution and 1 mL of the calcium solution. Wash the precipitate with three or four small portions of distilled water. Dilute to volume (see Figure 3). This filtrate contains the unreacted silver ions from the silver chloride precipitation which are found by standard atomic absorption procedures for silver, including measurements of suitable standards during the determinations.

11.7.4 Enter the curve (see Figure 1) with Ag^+ remaining, and determine the chloride concentration (ppm).

12. CALCULATION

Calculate the percentage of chloride in the concrete or concrete raw material, as indicated below:

$$\text{percent } \text{Cl}^- = \left(\frac{1}{V} \right) \left(\frac{\text{ppm } \text{Cl}^-}{W} \right)$$

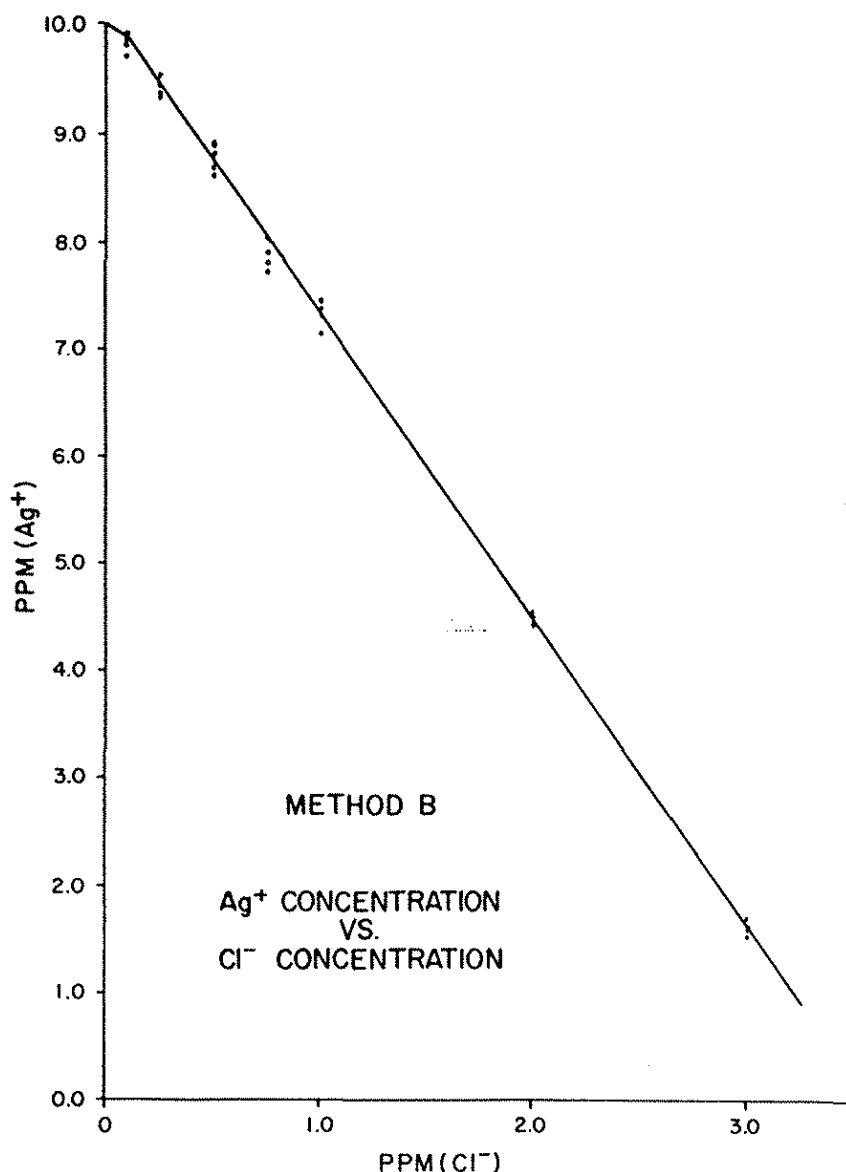


FIGURE 2A

where:

V = volume of aliquot used in Section 11.7, in millilitres,

W = weight of original concrete sample, in grams.

This equation assumes that the aliquot is diluted to 100 mL.

13. PRECISION

Data are being compiled that will be suitable for use in developing precision statements for this method.

METHOD B

SCHEMATIC SKETCH OF FILTRATION APPARATUS

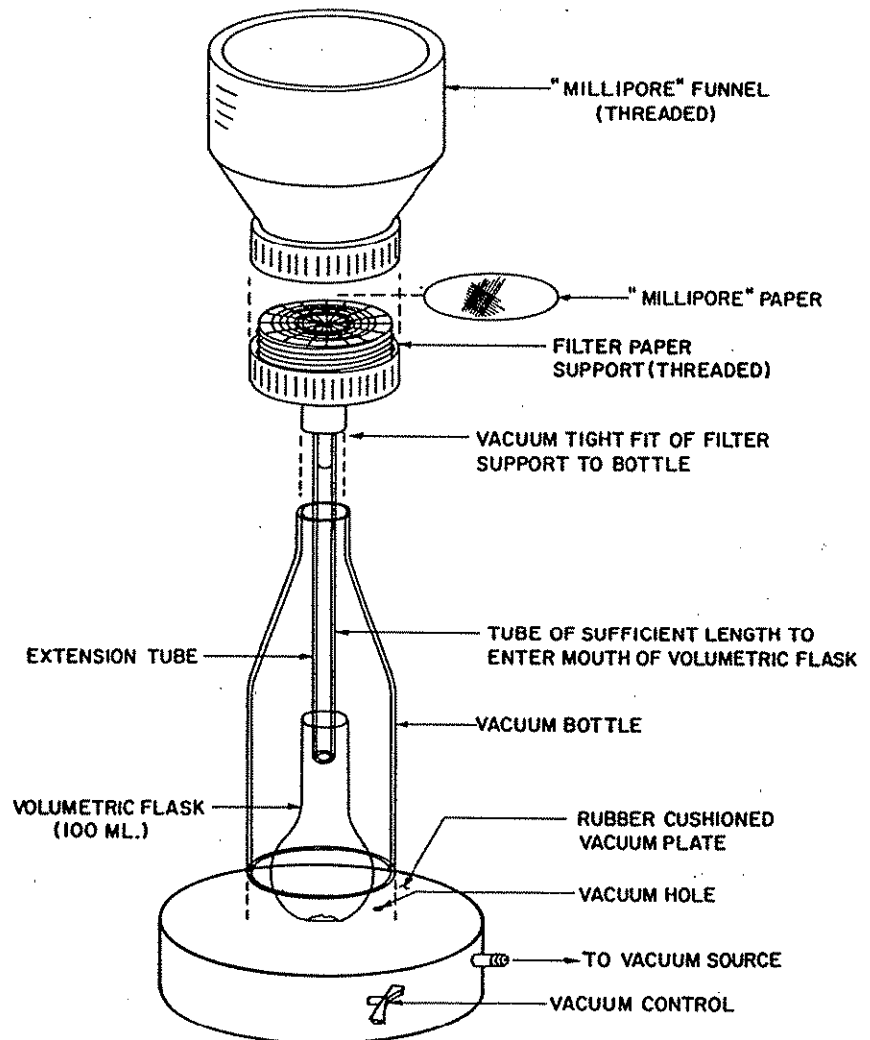


FIGURE 2B

Appendix F
AASHTO T 277-89

*Standard Method of Test
for*

Rapid Determination of the Chloride Permeability of Concrete

AASHTO DESIGNATION: T 277-89

1. SCOPE

1.1 This method covers the determination of the permeability of conventional portland cement and specialized, e.g., latex-modified and polymer, concretes to chloride ions. It consists of monitoring the amount of electrical current passed through 95 mm (3.75 in.) diameter by 51 mm (2 in.) long cores when one end of the core is immersed in a sodium chloride solution and a potential difference of 60 V dc is maintained across the specimen for 6 hours. The total charge passed, in coulombs, is related to chloride permeability.

2. REFERENCED DOCUMENTS

2.1 AASHTO Standards:

- T 24 Obtaining and Testing
Drilled Cores and Sawed
Beams of Concrete
- T 259 Resistance of Concrete to
Chloride Ion Penetration

3. SIGNIFICANCE AND USE

3.1 This method covers the laboratory evaluation of the relative permeability of concrete samples to chloride ions. The test results have shown good correlation with the results of 90-day chloride ponding tests (AASHTO T 259) on companion slabs cast from the same concrete mixes.

3.2 The method is suitable for specification acceptance, design purposes, service evaluation, and research and development.

3.3 Care should be taken in interpreting results of this test when it is used on surface-treated concretes. The results from this test on some such concretes show high chloride permeabilities, while 90-day chloride ponding tests on companion slabs show low permeabilities.

3.4 The method may be used on cores of diameters other than 95 mm (3.75 in.) and thickness other than 51 mm (2 in.). The values in Table 1 are not valid for any other size specimens, however, and no relationships have been established to adjust the values in that table for other specimen sizes. Data for specimens of other sizes may be used for relative comparisons of chloride permeabilities among specimens of the same size.

4. APPARATUS, REAGENTS, AND MATERIALS

4.1 Vacuum Saturation Apparatus (see Figure 1).

4.1.1 Separatory funnel—500 ml capacity.

4.1.2 Beaker—1,000 ml.

4.1.3 Vacuum desiccator¹—250 mm I.D.

4.1.4 Vacuum pump—capable of maintaining a pressure of less than 1 mm Hg (133 Pa) in desiccator.

4.1.5 Vacuum gage or manometer—accurate to ± 0.5 mm Hg (± 66 Pa) over range 0–10 mm Hg (0–13, 30 Pa) pressure.

4.2 Epoxy Coating Apparatus and Materials:

4.2.1 Epoxy resin—rapid setting, capable of sealing side surface of concrete cores.

4.2.2 Balance or scale, paper cups, wooden spatulas, and disposable brushes—for mixing and applying epoxy.

4.3 Specimen Sizing Equipment:

4.3.1 Movable bed diamond saw.

4.4 Voltage Application Apparatus, Reagents, and Materials:

4.4.1 Specimen-cell sealant—RTV silicone rubber or silicone rubber caulking.

4.4.2 Sodium chloride solution—3.0 percent by weight (reagent grade) in demineralized water.

4.4.3 Sodium hydroxide solution—0.3N, reagent grade.

4.4.4 Filter papers—No. 2, 90 mm diameter.

4.4.5 Digital voltmeter (DVM)—4½-digit, 200 mV full scale.

4.4.6 Digital voltmeter—3½ digit, 99.9 V full scale.

4.4.7 Shunt resistor—100 mV, 10 A rating.

4.4.8 Constant voltage power supply—0 – 80 V dc, 0 – 6 A, capable of holding voltage constant at 60 ± 0.1 V over entire range of currents.

4.4.9 Cable—two conductor, No. 14 (1.6 mm), insulated, 600 V.

4.4.10 Funnel—plastic, long stem.

4.4.11 Applied voltage cell (see Figures 2 and 3, Appendix).

4.4.12 Thermocouple wire and readout device (optional)—0–120 C (30–250 F) range.

5. TEST SPECIMENS

5.1 Obtain samples from the structure to be evaluated using a core drilling rig equipped with a nominal 4-in. (102 mm) diameter (3.75-in. (95 mm) actual I.D.) diamond-dressed core bit. Select and core samples following procedures in AASHTO Method T 24. Place the cores in a plastic bag for transport to the laboratory.

5.2 Using the diamond saw, cut a 2-inch (51 mm) slice from the top of the core, with the cut parallel to the top of the core. This slice will be the test specimen. Use a belt sander to remove any burrs on the end of the specimen.

¹ Desiccator must allow two hose connections, through rubber stopper and sleeve or through rubber stopper only. Each connection must be equipped with a stopcock.

6. CONDITIONING

6.1 Vigorously boil tapwater in a large (2L) Florence flask. Remove flask from heat, cap tightly, and allow water to cool to ambient temperature.

6.2 Allow specimen prepared in Section 5 to surface dry in air for 1 hour. Prepare approximately 10 g of rapid setting epoxy and brush onto sides of specimen. Place sample on sample-support stud while coating to ensure complete coating of sides. Allow coating to cure per manufacturer's instructions.

6.3 Check coating for tack-free surface. Place specimen in 1,000 ml beaker, then place beaker in vacuum desiccator. Seal desiccator and start vacuum pump.

Pressure should decrease to less than 1 mm Hg (1, 330 kPa) within a few minutes. Maintain vacuum for 3 hours.

6.4 Fill 500 ml separatory funnel with de-aerated water. With vacuum pump still running, open water stopcock and drain sufficient water into beaker to cover specimen (do not allow air to enter desiccator through this stopcock).

6.5 Close water stopcock and allow vacuum pump to run for 1 additional hour.

6.6 Close vacuum line stopcock, then turn off pump. Turn vacuum line stopcock to allow air to reenter desiccator.

6.7 Soak specimen under water in the beaker for 18 ± 1 hours.

7. PROCEDURE

7.1 Remove specimen from water, blot off excess water, and transfer specimen to can and seal temporarily.

7.2 If using two-part specimen-cell sealant, prepare approximately 20 g.

7.3 Place filter paper over one screen of the applied voltage cell; trowel sealant over brass shims adjacent to cast acrylic cell body. Carefully remove filter paper.

7.4 Press specimen onto screen; remove excess sealant which has flowed out of specimen/cell boundary. Cover exposed face of specimen with an imper-

meable material such as solid rubber sheeting. Place rubber stopper in cell vent-hole to restrict moisture movement. Allow sealant to cure per manufacturer's instructions.

7.5 Repeat steps 7.3 and 7.4 on second half of cell. (Specimen in applied voltage cell now appears as shown in Figure 4.)

7.6 Using the long stem funnel, fill left hand (-) side of cell, i.e., the side containing the top surface of the specimen, with 3.0 percent NaCl solution. Fill right hand (+) side of cell with 0.3N NaOH solution.

7.7 Attach lead wires to cell banana posts. Make electrical connections as shown in Figure 5. Turn power supply on, set to 60.0 ± 0.1 V, and record initial current reading (When the $4\frac{1}{2}$ -digit DVM specified in Section 4.4.5 is used with the 100 mV shunt, the DVM display can be read directly in milliamps disregarding the decimal point, i.e., 0.01 mV equals 1 milliamp).

7.8 Read and record current every 30 minutes. Monitor temperature inside of cell if desired (thermocouple can be installed through $\frac{1}{8}$ in. (3 mm) venthole in top of the cell).

NOTE 1—If temperature exceeds 190 F (88 C), discontinue test in order to avoid damage

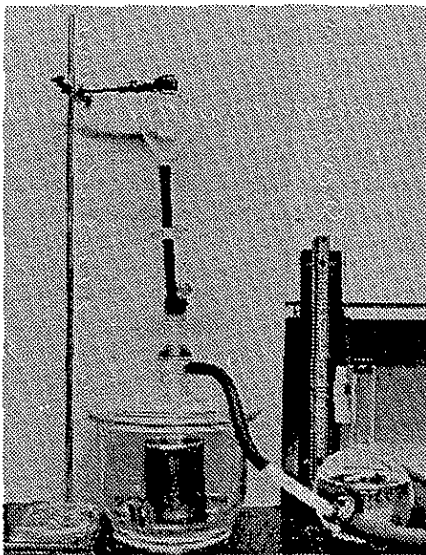


FIGURE 1 Vacuum Saturation Apparatus

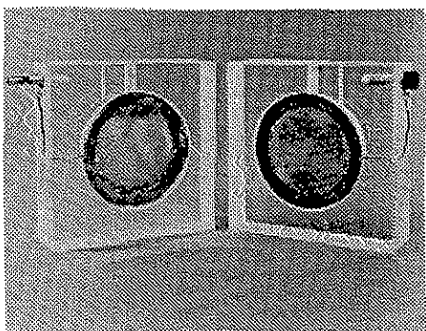


FIGURE 2 Applied Voltage Cell—Face View

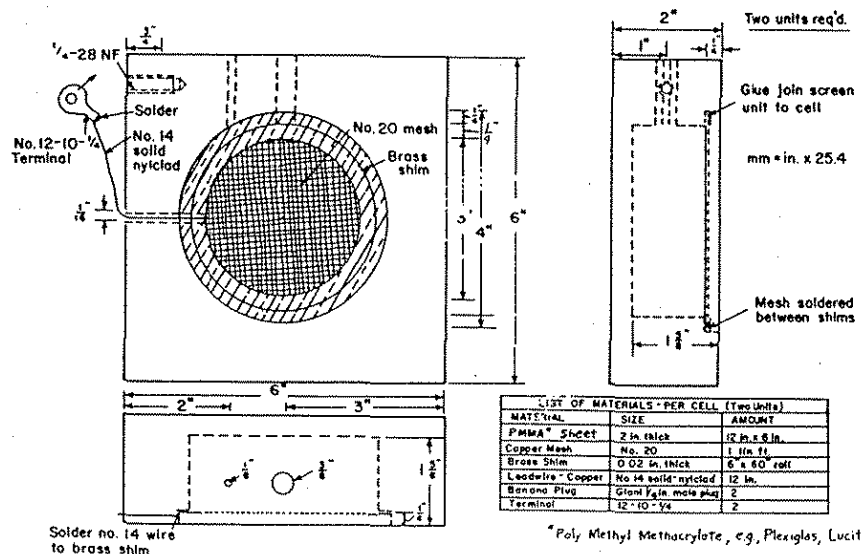


FIGURE 3 Applied Voltage Cell (Construction Drawing)

to cell. Such temperatures generally occur only for high permeability concretes and for specimens thinner than 51 mm (2 in.).

7.9 Terminate test after 6 hours.

7.10 Remove specimen. Rinse cell thoroughly in tapwater; strip out and discard residual sealant.

8. CALCULATION AND INTERPRETATION OF RESULTS

8.1 Plot current (in amperes) vs. time (in seconds). Draw a smooth curve through the data, and integrate the area underneath the curve in order to obtain

the ampere-seconds, or coulombs, of charge passed during the 6-hour test period.

NOTE 2—While conventional integration techniques such as planimetry or paper weighing can be used, programmable hand-held calculators which are now available can be used to numerically integrate the plots.

8.2 Use Table 1 to evaluate the test results. These values were developed from data on 3.75-in. (95 mm) diameter \times 2-in. (51 mm) long core slices taken from laboratory slabs prepared from various types of concretes.

NOTE 3—The terms in the middle column of Table 1 are not absolute. They are relative descriptions of the permeabilities of carefully prepared laboratory specimens.

9. REPORT

9.1 The report shall include the following:

9.1.1 Source of core, in terms of the structure and the particular location in the structure from which the core was obtained.

9.1.2 Identification number of core and specimen.

9.1.3 Location of specimen within core.

9.1.4 Type of concrete, including binder type, water-cement ratio, and other relevant data supplied with cores.

9.1.5 Description of specimen, including presence and location of reinforcing steel, presence and thickness of overlay, and presence and thickness of surface treatment.

9.1.6 Unusual specimen preparation, e.g., removal of surface treatment.

9.1.7 Test results, reported as the total charge passed over the test period and the maximum current recorded during the test period.

9.1.8 The chloride permeability equivalent to the calculated charge passed (from Table 1).

10. PRECISION AND BIAS

10.1 *Single-Operator Precision*—The single-operator coefficient of variation of a single test result has been found to be 12.3% (Note 4). Therefore the results of

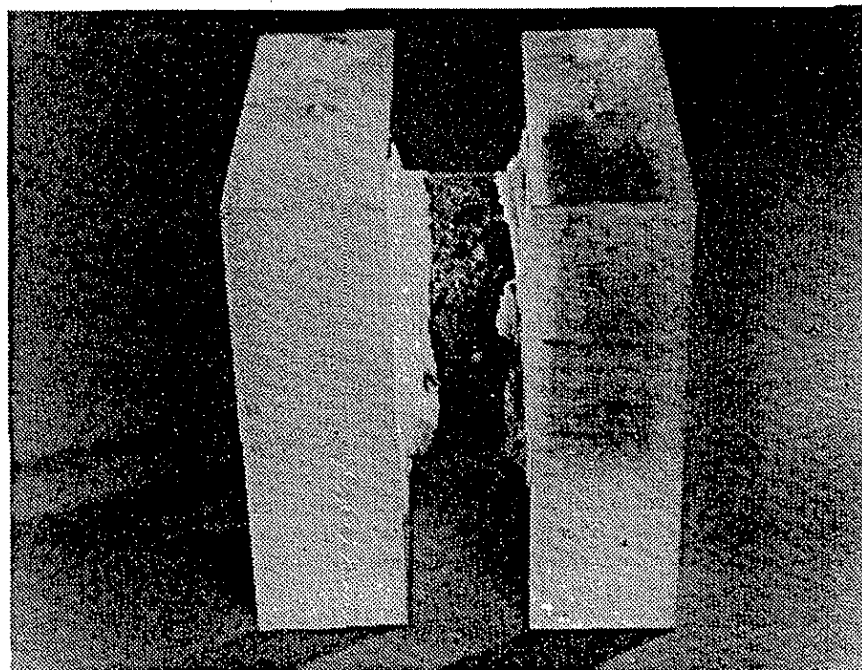


FIGURE 4 Specimen Ready for Test

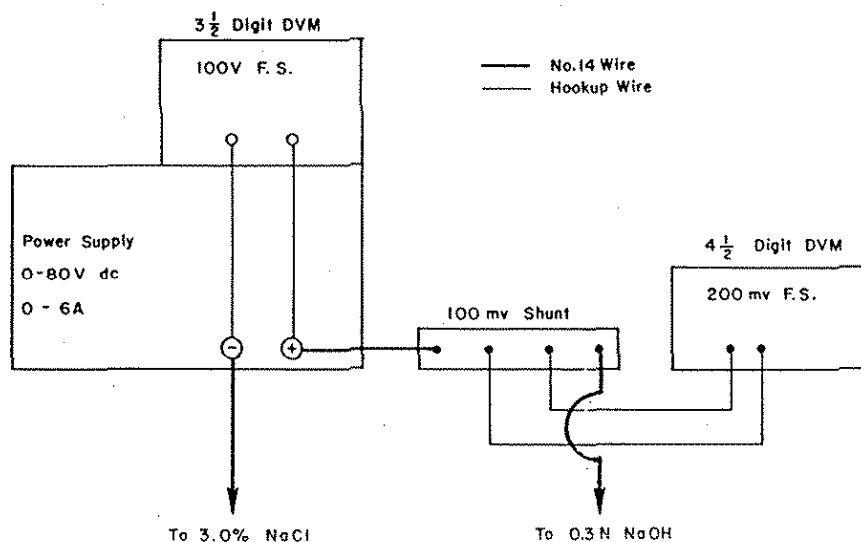


FIGURE 5 Electrical Block Diagram

TABLE 1 Chloride Permeability Based on Charge Passed (from Reference 2)

Charge Passed (coulombs)	Chloride Permeability	Typical of—
> 4,000	High	High water-cement ratio, conventional (>0.6) PCC
2,000-4,000	Moderate	Moderate water-cement ratio, conventional (0.4-0.5) PCC
1,000-2,000	Low	Low water-cement ratio, conventional (<0.4) PCC
100-1,000	Very Low	Latex-modified concrete Internally sealed concrete
<100	Negligible	Polymer impregnated concrete Polymer concrete

² Whiting, D., "Rapid Determination of the Chloride Permeability of Concrete," Report No. FHWA/RD-81/119, August 1981, available from NTIS, PB No. 82140724.

two properly conducted tests by the same operator on concrete samples from the same batch and of the same diameter should not differ by more than 35% (Note 4).

10.2 Multilaboratory Precision—The multilaboratory coefficient of variation of a single test result has been found to be 18.0% (Note 4). Therefore results of two properly conducted tests in different laboratories on the same material should not differ by more than 51% (Note 4). The average of three test results in two different laboratories should not differ by more than 29% (Note 5).

NOTE 4—These numbers represent, respectively, the (1S%) and (D2S%) limits as described in ASTM Practice C670, for Preparing Precision Statements for Test Methods for Construction Purposes. The precision statements are based on the variations in tests on three different concretes, each tested in triplicate in 11 laboratories. All specimens had the same actual diameters, but lengths varied within the range 51 ± 3 mm ($2 \pm \frac{1}{8}$ in.).

NOTE 5—Although the test method does not require the reporting of more than one test result, testing of replicate test specimens is usu-

ally desirable. The precision statement for the averages of three results is given since laboratories frequently will run this number of specimens. The percentage cited represents the (D2S%) limit divided by the square root of 3.

10.3 Bias—The procedure of this test method for measuring the chloride permeability of concrete has no bias because the value of this permeability can be defined only in terms of a test method.

APPENDIX—NOTES ON APPLIED VOLTAGE CELL CONSTRUCTION (REFER TO FIGURE 3)

1. ATTACHMENT OF LEAD WIRE TO SCREEN

Solder one end of the nylclad lead wire to the outer edge of the brass shim which holds the screen. The nylclad insulation should be removed prior to soldering by burning off with a propane torch and then removing the charred residue with wire wool.

2. ATTACHMENT OF SCREEN TO CELL

The screen is bonded to the cell by using a high quality waterproof adhesive. Scour both the screen shim and the cell lip with medium sandpaper prior to applying adhesive in order to obtain good metal to plastic bond. Apply a coating of adhesive to both cell and screen, run lead wire through $\frac{1}{16}$ in. (1.5 mm) hole inside of cell, then gently push screen into place on cell lip. Wipe excess adhesive off faceside of screen shim and place a weight on screen until adhesive has fully cured (24 hours).

3. ATTACHMENT OF LEAD WIRE TO BANANA PLUG

Solder a 12-10 $\frac{1}{4}$ ring terminal onto the bare end of the lead wire, keeping excess wire length to a minimum. Run the threaded end of the banana plug through the eyelet of the ring terminal, then thread banana plug into the $\frac{1}{4}$ -28 threaded hole in the side of the cell, tighten securely. Then fill the $\frac{1}{16}$ in. (1.5 mm) hole with clear silicone rubber caulk.

4. MATERIALS QUANTITIES AND COST

Some materials may not be available in the small quantities necessary to construct a single cell. In these cases package quantities have been quoted. Cast acrylic sheet stock will probably need to be precut by the suppliers, and the buyer will need to pay cutting charges unless he has another use for the full stock width.