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C. Kasper

Corrosion Research Laboratory Checklist

FM 5-516 Florida Method of Test for Determining Low-Levels of Chloride in Concrete and Raw Materials

		P	F	N/A
Sample Preparation				
1.	Sample material larger than 1.3 cm (0.5in.) is crushed such that the largest particle is no larger than 1.3 cm (0.5 in).			
2.	Clean the crusher in between samples to ensure no cross contamination.			
3.	Reduce the sample size to 400 g (14 oz) following any procedure in ASTM C702.			
4.	Dry sample(s) in an oven for 4 hours at 110 ^{o} C (230^{o} F) or until constant mass is achieved. Cool completely.			
5.	Pulverize sample such that it passes through a 50-mesh sieve.			
Note: For cement, pass the sample through a 20-mesh sieve.				
6.	Clean the pulverizer in between samples to ensure no cross contamination.			
7.	Store samples in a covered container to keep clean and dry.			
8.	Laboratory Control Sample (LCS): Prepare a hardened concrete LCS and a cement LCS with chloride concentrations near 0.1 lb./yd^3. Store in a covered container to keep clean and dry.			
Preparing Reagents				
9.	5% Nitric Acid Solution: Under fume hood, fill half of a 2000-mL volumetric flask with deionized water. Add 150 mL of approx. 70% concentrated HNO_3 to the volumetric flask. Dilute to volume with deionized water. Mix well.			
10.	0.1 N Silver Nitrate Solution: Place 16.987 g of reagent grade $AgNO_3$ into a 1000-mL volumetric flask and add deionized water to volume. Mix thoroughly. Alternatively, a pre-mixed 0.1 $AgNO_3$ standard solution may be used.			
11.	0.01 N Silver Nitrate Solution: Weekly, transfer 50.0 mL 0.1 $AgNO_3$ to 500-mL flask add deionized water to volume. Mix thoroughly.			
12.	ISA Solution: Dissolve 15.0 g of reagent grade KNO_3 in 100-mL volumetric flask and dilute with deionized water to volume. Mix well. Store in a chemically resistant bottle.			
13.	Store the Silver Nitrate solutions in brown, chemically resistant bottles away from light sources.			
14.	1000 ppm Cl Standard Solution: Dry reagent grade NaCl for 2 hours in a 140^{o} C oven. Cool in a desiccator. Place 1.649 g of NaCl into a 1000-mL volumetric flask add deionized water, bring to volume. Alternatively, a premade 1000 mg/L Cl Standard Solution can be purchased.			
15.	100 ppm Cl Standard Solution: Obtain NIST traceable 1000 mg/L Cl Standard Solution. Transfer 10.0 mL into 100-mL volumetric flask, finish filling the flask with deionized water. Mix well.			

16.	10 ppm Chloride Solution: Transfer 10.0 mL of 100 ppm solution to a 100-mL volumetric flask. Dilute to volume with deionized water and mix well.			
Note: The 10 ppm is used to determine the voltage potential threshold and is typically only done once or when changing electrode manufacturer or type.				
17.	3 ppm Chloride Check Sample (CCS): To make a single 3 ppm CCS, transfer 3.00 mL of 100 ppm Cl Standard Solution to a 100-mL volumetric flask. Add deionized water to bring to volume. Mix well. To make a bulk supply of 3 ppm, transfer 30.0 mL of 100 ppm Cl Standard Solution to a 1000-mL volumetric flask add deionized water to bring to volume. Mix well.			
Procedure				
18.	Maintenance of combination Cl electrode: Refresh the solution daily or every 36 analyses (12 samples) or a lapse of more than 1 hour. Follow the manufacturer's instructions to rejuvenate the electrode. This could include: polish membrane, flush and fill with the manufacturers recommended solution.			
19.	Condition in a solution of 100 mL deionized water, 1 mL ISA, and 2.5 mL of 0.01 N Silver Nitrate. Stir at a moderate and constant rate for 10 minutes. Remove electrode, rinse with deionized water and pat dry with a lint-free tissue.			
20.	Check Silver Nitrate Dispenser (0.01 N $AgNO_3$ and the 0.1 N $AgNO_3$) by dispensing (10) 0.5 mL aliquots into a 5-mL volumetric flask and verify mark. If necessary, adjust the dispenser and re-check. Check after every 36 analyses (12 samples).			
21.	Prepare triplicate samples by weighing out in accordance with Table 1 of the method. Place each sample in a 100-mL beaker.			
Note: If testing coarse or fine aggregate, place 0.5 g of material on a watch glass and add a few drops of 5% HNO_3 if it fizzes, weigh out 3.0000 g (±0.0005 g) of aggregate and transfer to a 100-mL beaker. If the aggregate does not fizz, weight out 3.0000 g (±0.0005 g) of aggregate and 2.0000 g (±0.0005 g) of LCS Cement, add to a 100-mL beaker.				
22.	Place watch glasses over the beakers to prevent contamination.			
23.	Add 5.0 mL of deionized water to each beaker.			
24.	Slowly add 35.0 mL of 5% HNO_3, to each beaker being careful to avoid excess frothing.			
25.	Use a clean glass rod for each sample to mix thoroughly. Stir each sample gently, avoiding frothy overflow. Continue to stir until there is no more frothing.			
26.	Waft the sample or use a commercially available kit to determine the presence of slag (rotten egg smell).			
27.	If sample has a rotten egg smell, add an additional 1 to 2 mL of concentrated Nitric Acid.			
28.	If samples do not contain slag, boil for 3-5 minutes, if the sample contains slag boil for 10-12 minutes. Use a hot plate, set temperature to a minimum of 250 °C (482 °F).			
29.	Remove the samples from the hot plate and filter through a funnel containing a Whatman No. 41 filter paper (or comparable product) into a 100-mL flask. Pour off the liquid and let it filter through first.			
30.	Rinse the residue out of the beaker (using hot deionized water) into the funnel. Repeat rinsing being careful not to go over the mark on the flask. Allow to cool to room temperature (about 1 hour).			
31.	Add deionized water to the mark (bottom of the meniscus should be at line). Do not overfill. Cap flask.			

Note: If testing grout check the pH prior to titration (after filtering and filling to volume). The pH should be between 2 and 9, if not start over and adjust the mass either up or down to get it in that range.				
Note: Run the 0.01 and 0.1 blank correction daily or every 36 analyses (12 sample).				
32.	Add 100 mL of deionized water to a beaker.			
33.	Add 1.00 mL of ISA.			
34.	Immerse the electrode in the solution and stir at a moderate and constant speed.			
35.	Add 1.00 mL of 0.01 N Silver Nitrate and record the scaling potential.			
36.	Make four more 0.50-mL additions of Silver Nitrate and record the potential and total volume of titrant after each increment.			
37.	Remove and rinse the electrode with deionized water.			
38.	When not testing, keep the electrode in a beaker with clean deionized water.			
39.	Calculate and verify that the end point is 0 ±0.100 and is between the upper control limit (UCL) and the lower control limit (LCL). If outside of those limits, verify that all data was entered correctly. It may require rejuvenating the electrode, condition, and re-test the 0.01 blank.			
40.	When the 0.01 blank meets the above requirements, prepare the 0.1 blank.			
41.	Add 100 mL of deionized water to a beaker.			
42.	Add 1.00 mL of ISA.			
43.	Immerse the electrode in the solution and stir at a moderate and constant speed.			
44.	Add 1.00 mL of 0.10 N Silver Nitrate and record the scaling potential.			
45.	Make four more 0.50-mL additions of Silver Nitrate and record the potential and total volume of titrant after each increment.			
46.	Remove and rinse the electrode with deionized water.			
47.	When not testing, keep the electrode in a beaker with clean deionized water.			
48.	Calculate and verify that the end point is 0 ±0.100 and is between the upper control limit (UCL) and the lower control limit (LCL). If outside of those limits, verify that all data was entered correctly. It may require making fresh solutions or rejuvenating the electrode, condition, and re-test the 0.01 and 0.1 blank.			
Note: Run the 3-ppm chloride check sample daily or every 36 analyses (12 sample).				
49.	Transfer 100-mL of the 3 ppm CCS to a beaker and add 1.00 mL of ISA.			
50.	Immerse the electrode in the solution and stir the solution at a moderate and constant speed.			
51.	Add 0.01 N Silver Nitrate titrant in 0.50-mL increments until the scaling potential is reached or just exceeded. Record the total titrant added and the potential.			
52.	Make four more 0.50-mL additions, recording the potential and the total volume of titrant after each increment.			
53.	Remove and rinse the electrode with deionized water.			
54.	Determine the endpoint and subtract the 0.01 blank endpoint, calculate the concentration in ppm and verify it is between 2.85 and 3.15 and is between the upper control limit (UCL) and the lower control limit (LCL). If outside of those limits,			

	verify that all data was entered correctly, and if it was, test a new CCS sample. If 3 ppm CCS continues to fail, prepare fresh solutions and test again.			
Note: Run the Lab Control Sample (LCS) daily or every 36 analyses (12 sample).				
55.	Empty contents of flask into a beaker.			
56.	Immerse the electrode in the solution and stir the solution at a moderate and constant speed.			
57.	Add 0.01 N Silver Nitrate titrant in 0.5 mL increments until the scaling potential of the blank is reached or just exceeded. Record the total titrant added and the potential.			
58.	Make four 0.50-mL additions, recording the potential and total volume of titrant after each increment.			
59.	Remove and rinse the electrode with deionized water.			
60.	Determine the endpoint and subtract the 0.01 blank endpoint and verify it is between the upper control limit (UCL) and the lower control limit (LCL). If outside of those limits, verify that all data was entered correctly and test a new LCS sample if necessary. If the LCS continues to fail, prepare fresh solutions and test again.			
Note: Run the chloride sample.				
61.	Empty contents of flask into a beaker.			
62.	Immerse the electrode in the solution and stir the solution at a moderate and constant speed.			
63.	Use 0.01 N Silver Nitrate solution if the initial mV reading is greater than the potential measured on a 10 ppm chloride solution or use 0.1 N Silver Nitrate solution if it is less than the potential measured on a 10 ppm chloride solution.			
64.	Add the Silver Nitrate titrant in 0.50-mL increments until the scaling potential of the corresponding blank is reached or just exceeded. Record the total titrant added and the potential.			
65.	Make four 0.50-mL additions, recording the potential and total volume of titrant after each increment.			
66.	Remove and rinse the electrode with deionized water.			
67.	Repeat the titration for the remaining replicates.			
68.	Determine the endpoint and subtract the corresponding blank endpoint and then calculate the chloride concentration in lbs/yd^3.			
69.	Check to see if the range for the sample set is less than 0.080 lbs/yd^3 when titrating samples with 0.01 N Silver Nitrate (0.80 lbs/yd^3 if titrating samples with 0.10 N Silver Nitrate) if the sample set is out of range this indicates a failure, and the sample set must be re-analyzed.			

Remarks:

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C. Kasper

Date: _____ Technician: _____ Observer: _____

Technician's E-mail Address: _____

Employer's/Supervisor's E-mail Address: _____