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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.	For concrete, crush sample such that the largest particle is no larger than 0.5 inches.			
2.	Clean the crusher in between samples to ensure no cross contamination.			
3.	Reduce the sample size to 400 g using a rock splitter.			
4.	Dry sample(s) in an oven for 4 hours at 110 ^{o} C (230^{o} F) Cool completely.			
5.	Pulverize sample such that it passes through a 50-mesh sieve.			
6.	Clean the pulverizer in between samples to ensure no cross contamination.			
7.	For cement, pass the sample through a 20-mesh sieve.			
8.	Store powdered samples in a covered container to keep clean and dry.			
Preparing Reagents				
9.	5% Nitric Acid Solution: Under fume hood, fill 1800 mL of DI water in a 2000-mL flask. Add 150 mL of approx. 70% concentrated HNO_3. Add 50 mL more of DI water.			
10.	0.100 N Silver Nitrate Solution: Place 16.987 g of $AgNO_3$ into a 1000-mL flask and add DI water.			
11.	0.010 N Silver Nitrate Solution: Weekly, transfer 50 mL 0.10 $AgNO_3$ to 500-mL flask-add DI water.			
12.	ISA Solution: Dissolve 15 g of KNO_3 in 100 mL of DI water.			
13.	Store the Silver Nitrate and Potassium Nitrate in opaque bottles and store in the dark.			
14.	1000 mg/L Cl Standard Solution: Dry NaCl reagent for 2 hours in a 105^{o} C oven. Cool. Place 1.649 g of NaCl in 1000 mL of DI water. Alternatively, a premade 1000 mg/L Cl Standard Solution can be purchased. Use for Electrode Calibration Curve.			
15.	100 mg/L Cl Standard Solution: Obtain NIST traceable 1000 mg/L Cl Standard Solution. Transfer 10 mL into 100-mL flask, finish filling the flask with DI water. Use for the daily 3 ppm Cl Check Standard.			
Procedure				
16.	Maintenance of combination Cl electrode: Refresh the solution daily or every 10 samples (30 analyses). Polish membrane. Condition for 10 minutes in a solution of DI water, ISA, and Silver Nitrate.			
17.	Check Silver Nitrate Dispenser by dispensing (10) 0.5 mL into a 5-mL flask and verify mark. If there are bubbled in the dispenser, place valve in recirculate position.			

18.	Prepare triplicate samples by weighing out 4.0000 g (±0.0005 g) of concrete or 2.0000 g (±0.0005 g) of cement, placing each sample in a 100-mL beaker			
19.	Place watch glasses over the beakers to prevent contamination.			
20.	Add 5.0 mL of DI water to each beaker. Place a clean stir rod in each beaker. Stir each sample.			
21.	Slowly add 35.0 mL of 5% HNO_3, to each beaker being careful to avoid excess frothing.			
22.	Stir each sample gently, avoiding frothy overflow. Continue to stir until there is minimal frothing.			
23.	Waft the sample or use a commercially available kit to determine the presence of slag (rotten egg smell).			
24.	If sample has a rotten egg smell, add an additional 1 to 2 mL of concentrated Nitric Acid.			
25.	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 250° C.			
26.	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.			
27.	Rinse the residue out of the beaker into the funnel. Repeat this 2-3 times using 30 mL of hot DI water. Allow to cool to room temperature (about 1 hour).			
28.	Add DI water to the mark (bottom of the meniscus should be at line). Do not overfill.			
Note: Run the 0.01 and 0.10 blank correction daily or every 10 sample (30 analyses).				
29.	Add 100 mL of DI water to a 250-mL beaker for the 0.01 blank.			
30.	Add 1 mL of ISA.			
31.	Place the beaker on the magnetic stir plate and add a clean stir bar to the beaker.			
32.	Immerse the electrode in the solution and stir at a low-medium speed.			
33.	Add 1.0 mL of 0.01 N Silver Nitrate and record the scaling potential.			
34.	Make four 0.5 mL additions of Silver Nitrate and record the potential and total volume of titrant after each increment.			
35.	Remove and rinse the electrode and stir bar with DI water.			
36.	Record the above data in the Chloride Database. Run another blank if an error message is received or the control charts are violated.			
37.	Add 100 mL of DI water to a 250-mL beaker for the 0.10 blank.			
38.	Add 1 mL of ISA.			
39.	Place the beaker on the magnetic stir plate and add a clean stir bar to the beaker.			
40.	Immerse the electrode in the solution and stir at a low-medium speed.			
41.	Add 1.0 mL of 0.10 N Silver Nitrate and record the scaling potential.			
42.	Make four 0.5 mL additions of Silver Nitrate and record the potential and total volume of titrant after each increment.			
43.	Remove and rinse the electrode and stir bar with DI water.			
44.	Record the above data in the Chloride Database. Run another blank if an error message is received or the control charts are violated.			
Note: Run the 3-ppm chloride check sample daily or every 10 sample (30 analyses).				

45.	Transfer 3 mL of 100-ppm Chloride Standard Solution into a 100-mL flask add DI water to bring up to volume. Transfer to a 250-mL beaker and add 1 mL of ISA.			
46.	Place the beaker on the magnetic stir plate and add a clean stir bar to the beaker.			
47.	Immerse the electrode in the solution and stir the solution at a low-medium speed.			
48.	Add 0.01 N Silver Nitrate titrant in 0.5 mL increments until the scaling potential in 33 is exceeded. Record the total titrant added and the potential.			
49.	Make four 0.5 mL additions, recording the potential and the total volume of titrant after each increment.			
50.	Remove and rinse the electrode and stir bar with DI water.			
51.	Record the above data in the Chloride Database. Run another 3-ppm Cl Standard if an error message is received or the control charts are violated. If results are not greater than 2.9 or less than 3.1, it is a fail.			
Note: Run the Lab Control Sample (LCS) daily or every 10 sample (30 analyses). Run this sample from (18 to 28).				
52.	Empty contents of flask into a 250-mL beaker.			
53.	Place the beaker on the magnetic stir plate and add a clean stir bar to the beaker.			
54.	Immerse the electrode in the solution and stir the solution at a low-medium speed.			
55.	Use 0.01 N Silver Nitrate solution if the initial mV reading is more than 100 mV and 0.1 N Silver Nitrate solution if less than 100 mV.			
56.	Add 0.01 N (or 0.10 N) Silver Nitrate titrant in 0.5 mL increments until the scaling potential of the blank (in line 33 for 0.01 N or line 41 for 0.10 N) is exceeded. Record the total titrant added and the potential.			
57.	Make four 0.5 mL additions, recording the potential and total volume of titrant after each increment.			
57.	Remove and rinse the electrode and stir bar with DI water.			
59.	Record the above data in the Chloride Database. Run another LCS if the control charts are violated. This sample will not indicate a failure, it is necessary to check the control charts.			
Note: Run the chloride sample from (18 to 28).				
60.	Empty contents of flask into a 250-mL beaker.			
61.	Place the beaker on the magnetic stir plate and add a clean stir bar to the beaker.			
62.	Immerse the electrode in the solution and stir the solution at a low-medium speed.			
63.	Use 0.01 N Silver Nitrate solution if the initial mV reading is more than 100 mV and 0.1 N Silver Nitrate solution if less than 100 mV.			
64.	Add 0.01 N or (0.10 N) Silver Nitrate titrant in 0.5 mL increments until the scaling potential of the blank (in line 33 for 0.01 N or line 41 for 0.10 N) is exceeded. Record the total titrant added and the potential.			
65.	Make four 0.5 mL additions, recording the potential and total volume of titrant after each increment.			
66.	Remove and rinse the electrode and stir bar with DI water.			
67.	Repeat the steps in (18 to 28) and (60 to 66) for the two other extractions.			
68.	Enter the data in the chloride database.			
69.	If the sample range is greater 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) the software will indicate a failure and the sample must be re-analyzed.			

Remarks:

Date: _____ Technician: _____ Observer: _____

Technician's E-mail Address: _____

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