GENERAL

NZS 3121\(^1\) sets out requirements for testing levels of chloride in concrete.

As a precaution against reinforcement corrosion, NZS 3101\(^2\) and NZS 3109\(^3\) require that the maximum total chloride ion content of steel reinforced concrete shall be measured for compliance:

EITHER

*Based on measurements of chloride content arising from aggregate, mixing water (including slurry water) and admixtures*

OR

*Directly measured from a sample of hardened concrete*

Acid soluble chloride ion contents per m\(^3\) of concrete shall not exceed the following limits –

- 0.5 kg/m\(^3\) for prestressed concrete
- 0.8 kg/m\(^3\) for concrete in an environment exposed to moisture or chloride
- 1.6 kg/m\(^3\) for concrete in a dry or moisture protected environment

Ultimately it is the responsibility of the concrete supplier to verify that the concrete complies within these requirements.

TEST METHODS

Wherever possible, aggregate samples shall be tested by a laboratory accredited by IANZ for appropriate test methods. If it is not practical or economical to use a laboratory accredited for a particular test method, then the analysis can be performed by a laboratory with independent accreditation for measurement of similar aggregate properties, or a laboratory that has been independently audited and found to comply with NZS ISO/IEC 17025\(^4\) or AS/NZS ISO 9001\(^5\).

NZS 3121 requires that:

a) The aggregate supplier shall provide test data on the acid soluble chloride ion content (by wet chemistry) of each aggregate and sand constituent to either AS 1012.20\(^6\) or BS EN 1744.5\(^7\).

b) An alternative aggregate test method is x-ray fluorescence spectroscopy (XRF). XRF measures total chloride in a sample, which can be greater than the acid-soluble chloride content.

Therefore compliance testing by XRF first requires calibration against tests for acid soluble chlorides. Consequently XRF is better suited to measuring the consistency of chloride contents of a particular aggregate supply (or concrete) over time, rather than for compliance testing a small number of samples (<5).

c) The admixture supplier shall provide the chloride content of any admixtures used.

d) Within NZS 3121 there is an upper limit 0.10% of chloride in mix water for reinforced concrete exposed to moisture or chloride in service, and 0.05% for prestressed concrete tested to ASTM D 512\(^8\). This requirement shall need to include slurry water. The chloride in the mix water shall be included in total chloride content of the concrete mix.

The calculation of chloride content of fresh concrete from the mix components is given in IB 83\(^9\) which can be downloaded from the Cement & Concrete Association of New Zealand (CCANZ) – www.ccanz.org.nz

e) Wet chemistry test methods for determining acid soluble chloride ion content of hardened concrete shall be based on BS 1881-124\(^10\), AS 1012.20 or ASTM C1152\(^11\).
TEST FREQUENCY

The aggregate supplier shall test for chloride on a regular basis to ensure that the processing of aggregate and sand keeps within the chloride limits initially declared to the concrete producer.

Aggregates, both coarse aggregate and sand, shall have their acid soluble chloride levels tested by the aggregate supplier at a frequency agreed between aggregate supplier and concrete producer. Results shall meet limits declared to the concrete producer, expressed as % chloride by mass of aggregate or sand supplied.

Where sea dredged and or beach deposits are the source of aggregate materials, the aggregate supplier shall test for chloride on a regular basis to ensure that the process keeps within agreed chloride limits declared to the concrete producer. Testing shall be carried out initially, and at each change of aggregate source, thereafter with a minimum of one test annually for marine aggregates.

Where inland quarries or pits are the source of aggregate materials, initially a single confirming test shall be carried out.

ACCURACY OF TEST METHODS

Two methods of testing hardened concrete for chlorides have been evaluated through a testing programme undertaken on the NZRMCA’s behalf by Opus Central Laboratories, on hardened concrete samples produced by the industry. The formal report is held by NZRMCA – Comparison of XRF and AS 1012.20 Test Methods, Opus Central Laboratories, June 2009.

The industry samples covered a range of chloride contents up to 1.8 kg/m3 which was established by chloride admixture dosing. The concrete plants used for manufacture of concrete from which samples were taken, used basalt aggregates or greywacke aggregates.

As different New Zealand Standards quote the use of the one or two methods, it was deemed necessary to cross check that the methods produced similar results. Currently acceptable test methods to determine chloride content of hardened concrete are:
As different New Zealand Standards quote the use of the one or two methods, it was deemed necessary to cross check that the methods produced similar results. Currently acceptable test methods to determine chloride content of hardened concrete are:

- NZS 3101:2006 accepts Acid Soluble Chloride test to ASTM C1152 (AS 1012.20) and XRF
- NZS 3109:1997 accepts Acid Soluble Chloride test to ASTM C1152 (AS 1012.20)
- NZS 3121:2015 accepts Acid Soluble Chloride test to ASTM C1152 (AS 1012.20) (BS1881-124) and XRF

The report showed that either AS 1012.20 or XRF was satisfactory for the likely degrees of accuracy required for construction evaluation. However, various issues in the testing programme were encountered as follows:

MATTERS ARISING FROM COMPARATIVE TESTS

1. During the first iteration of results, there appeared to be 0.2 kg/m³ difference between XRF and the acid soluble test. This evaluation was examined and it was determined that the difference had occurred from using an ‘out of date’ reference sample used to set up the XRF equipment. The tests were then repeated and the XRF results were in agreement with the acid soluble tests.

2. The chloride dosage rate used was from nil to 1.8 kg/m³. While parallel testing by XRF after the correction described in 1. above, and by the acid soluble test gave equal results, these results were approximately 0.4 kg/m³ above the calculated dosage content for all basalt concretes, including the sample that had no chloride added.

   After closer evaluation, it appears that constituents in some basalt may enter into the evaluation methods used to identify chloride, whilst in reality this is chloride bound in the basalt material and does not contribute to corrosion arising from free chlorides present in the concrete. The degree of fineness of grinding the specimen in preparation for testing appears to influence the degree of variation experienced. Hence it is important when using basalt materials to have accurate information on potential chloride dosage to establish chloride levels by calculation.

3. Chloride testing carried out on two sources of marine sands taken saturated off the barge to two different test methods, were within allowable NZS 3101 levels.

SUMMARY

Where it is likely that chloride limits on a project basis are to be checked from the hardened concrete, it is recommended the acid soluble test method to BS 1881 Part 124, ASTM 1152 or AS 1012.20 be used or at least used to establish a reference link before using the XRF method. This concurs with NZS 3121.

Most routine checks of hardened concrete for chloride content could be done by wet chemistry as in general, ‘standard concretes’ are well below the NZS 3101 and NZS 3109 chloride thresholds.

For plants using basalt materials there may be a correction needed as some basalts may give an inflated reading of soluble chloride by 0.3 - 0.4 kg/m³. A portion of this chloride is bound and cannot contribute to reinforcement corrosion. Where such readings are above the maximum allowable concrete levels, calculations should be carried out to show that the levels of total chloride based on individual chloride levels for the components of the concrete are below the allowable.

Testing for chloride levels of marine based aggregates, shall be carried out initially and at each change of aggregate source, thereafter once annually. Confirmatory static chloride test levels of other concrete components (admixtures, mix water) shall be kept for reference.
REFERENCES

1. NZS 3121: 2015 *Water and aggregate for concrete*
2. NZS 3101: 2006 *Concrete structures standard*
3. NZS 3109: 1997 *Concrete construction*
4. NZS ISO/IEC 17025: 2005 *General requirements for the competence of testing and calibration laboratories*
5. AS/NZS ISO 9001: 2016 *Quality management systems - Requirements*
7. BS EN 1744-5: 2006 *Tests for chemical properties of aggregates. Determination of acid soluble chloride salts*
8. ASTM D512 - 12 *Standard test methods for chloride ion in water*
9. CCANZ IB 83 *Chloride content of fresh concrete*
11. ASTM C1152 - 04 *Standard test method for acid-soluble chloride in mortar and concrete*