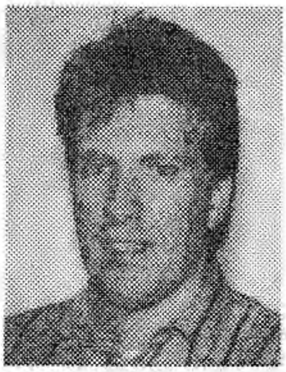
MEASUREMENT OF CHLORIDES IN CONCRETE - AN EVALUATION OF TWO DIFFERENT SAMPLING TECHNIQUES



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ABSTRACT
A test has been performed to evaluate two different sampling techniques and their influence on the results of chloride measurements in concrete. The two techniques used were dry drilling using a hand held drill and water-cooled drilling of cores using a jig-mounted drill. Samples were taken from concrete specimens cast with 0.0%, 1.0 % and 3.0 % chloride content by weight of cement. The specimens also differed in maximum aggregate sizes. The specimens containing 0.0 % chloride were after hardening exposed to chloride solution to produce a concentration gradient. The analysis shows good accuracy and precision in measured chloride contents for cores drilled from specimens of known concentration. Correlation between parallel cores is good.
Measured chloride contents from dry drilled dust show poorer but acceptable accuracy and precision only for max. aggregate sizes below drilling diameter. It is concluded that a bore diameter above maximum aggregate size is essential to obtain sufficient accuracy and precision in chloride measurements.

KEY WORDS: concrete structures, chlorides, sampling techniques

1 INTRODUCTION

This investigation was designed to evaluate the influence of the sampling technique on the results of chloride measurements in concrete. The analysis of chlorides in concrete has received great current interest with the increasing knowledge about the extent of the damaging effects of chlorides on steel reinforcement. Chlorides penetrate into the concrete over a period of time. The depth of penetration is dependent on factors such as concrete permeability, humidity and chloride exposure. Therefore, in addition to the usual measurements of chlorides, there is often a wish to map the amount of penetration of chlorides into the concrete through the measurement of concentration gradients or profiles. This makes great demands not only to the chemical analysis /1/, but also to the sampling technique.

Several sampling techniques for chloride analysis are in normal use. Among these are:

- chiselling by hand or machine
- dry drilling by using hand held drill
- water-cooled drilling of cores using a jig-mounted drill
- grinding using a profile grinding equipment

The chiselling technique suffers from poor controllability and is not suitable for accurate recording of chloride concentration gradients in concrete. Profile grinding is a highly accurate and promising sampling technique, but it's use is limited due to it's recent development.

This work deals with the second and third techniques, dry drilling and water-cooled drilling of cores, of which dry drilling is the most commonly used due to low costs and simplicity. Core drilling normally gives the best accuracy and precision, provided that the size of the aggregate is not too large compared to the core diameter. The technique requires heavier equipment on the sampling site and additional cutting before chemical analysis. It is therefore more costly in use. Both techniques are suitable for measurement of chloride profiles in concrete.

Following factors are investigated:

- influence of aggregate size on analysed chloride content
- number of sample parallels needed to meet requirement for adequate accuracy and precision, comparison of the two techniques
- ability to detect chloride profiles, comparison of the two techniques
- effect of chloride concentration

For this purpose 296 different samples were taken from concretes cast with known chloride content and concretes with a laboratory induced chloride profile.

2 SPECIMENS

2.1 Test specimens

The specimens consisted of twenty 100 mm cubes for measurement of compressive strength, and twenty-four beams (150 mm x150 mm x 470 mm) for chloride content testing. All the beams and twelve of the cubes were cast with chloride contents of 1.0% and 3.0 % by weight related to the weight of the cement.

In addition, four plates (150 mm x 400 mm x 700 mm) were made without chlorides. Two of these plates were exposed to chloride solution during a cycling process. The cycling process consisted of drying at 50 °C for two weeks followed by immersion in 3% Cl-solution (chlorides added to water as NaCl) to achieve a chloride profile.

2.2 Plates

For mix proportions see table 2.1.

Table 2.1 Mix proportions for plates.

Material	Mix III and IV
	kg/m^3
Cement type RP 38 (ASTM Type III)	371
Water	167
Fine aggregate, 0-8 mm	1047
Coarse aggregate, 8-11.2 mm	476
Coarse aggregate, 11.2-16 mm	381
W/c-ratio	0.45
Vol-% paste	28.8

Seven days after casting, the plates were demolded and stored under exposure to laboratory air. The cubes were demolded after one day and stored in water until tested for compressive strength after 28 days. Plasticizers, which could affect the chloride measurements, were avoided.

The plates were left in storage for 17 months before commencing the cycling process.

2.3 Beams

The beams were made with three different maximum aggregate sizes and two different chloride concentrations, giving six different concrete recipes. Each of the recipes were used to make four beams (150 mm x 150 mm x 470 mm) for chloride measurements and cubes for compressive strength tests. The chlorides were dissolved in a part of the mix-water and then poured into the concrete mix.

For mix proportions see table 2.2.

Both the beams and the cubes were demolded one day after casting. The cubes were stored in water until tested for compressive strength after 28 days. Each beam was sealed separately with a strong plastic foil, placed with the others and finally covered with a second plastic foil. The beams were then stored in this way for fifteen months before sampling.

The compressive strength and density of the hardened concrete are shown in table 2.3. All figures are mean values obtained from the testing of two specimens.

Table 2.2 Mix proportion for beams.

Material	Mix kg/m³					
	1	2	3	4	5	6
Rapid cement RP 38	371	371	371	371	371	371
Water	167	167	167	167	167	167
Sand 0-8	952	952	1046	1046	1047	1047
Coarse aggregate 8-11.2	952	952	476	476	190	190
Coarse aggregate 11.2-16	-	-	381	381	190	190
Coarse aggregate 16-32	-	-	-	-	476	476
NaCl	6.12	18.35	6.12	18.35	6.12	18.35
W/c-ratio	0.45	0.45	0.45	0.45	0.45	0.45
Vol.paste (%)	28.8	28.8	28.8	28.8	28.8	28.8
Vol.aggregate (%)	71.2	71.2	71.2	71.2	71.2	71.2
Chloride content by m-% of cement	1.0	3.0	1.0	3.0	1.0	3.0

Table 2.3 Mechanical properties of hardened concrete (plates and beams).

Properties of hardened concrete	Mix							
	1	2	3	4	5	6	III	IV
Compressive strength (MPa)	58.1	54.8	56.1	53.5	55.0	52.0	54.1	51.9
Density (g/cm³)	2.438	2.434	2.420	2.428	2.407	2.441	2.440	2.421

3 EXPERIMENTAL

3.1 Sampling

3.1.1 Beams

To determine a representative mean value of the chloride content in the beams, a 50 mm thick slice was cut from the centre of each beam, as shown in Fig. 3.1. Each slice was cut to form an inner and outer part. The outer part consisted of the outer 25 mm of the slice measured from the beam surface. Each part was dried, grounded and analysed separately.

Two cores (Ø 98 mm) were drilled from each beam using a jig mounted water cooled drill. Four slices with thickness 8 mm were cut from each core at the following depths measured from the concrete surface: 0-10 mm, 10-20 mm, 20-30 mm, 30-40 mm. The water cooling was kept at a minimum to reduce the danger of chlorides being washed out.

Five dry drilled samples were collected from each beam by drilling five 40 mm deep holes with diameter 20 mm. Dry drilled samples and cores were collected according to a fixed pattern. The drill was stabilized during sampling by a template to avoid damaging the concrete surface. To determine the chloride profile, dust samples were collected at the following depths: 0-10 mm, 10-20 mm, 20-30 mm, 30-40 mm. Between each drilling step dust was removed from the holes using compressed air.

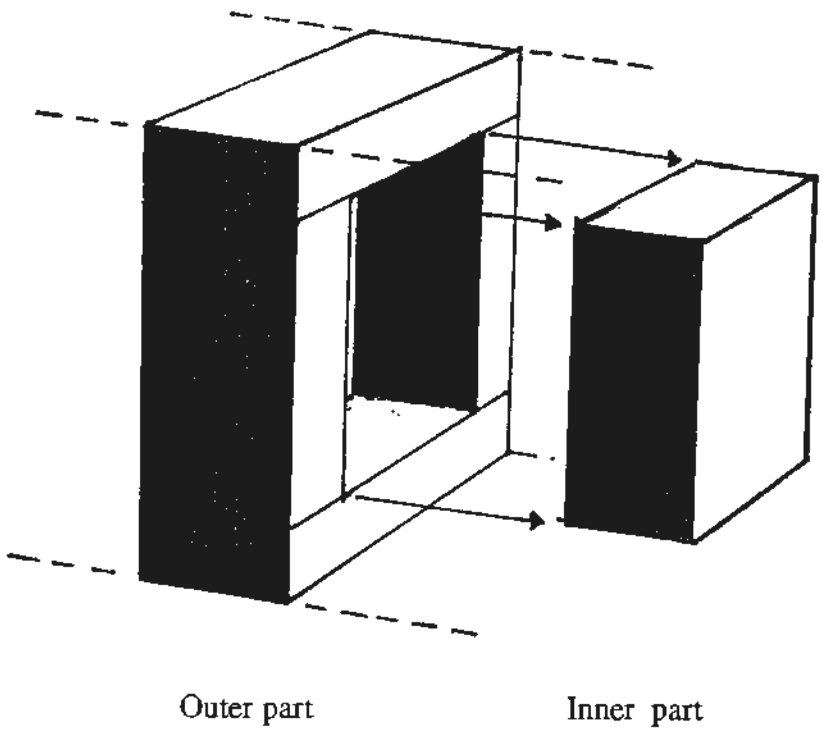

Fig. 3.1 Exploded view of 50 mm slice as cut from the centre of each beam.

3.1.2 Plates

One core with diameter 98 mm was drilled from each plate using a water cooled core drilling equipment. As for beams the water cooling was kept at a minimum to avoid loss of chlorides. Six slices with thickness 8 mm were cut from each of the cores. These slices were cut at the following depths measured from the concrete surface: 0-10 mm, 10-20 mm, 20-30 mm, 30-40 mm, 40-50 mm and 50-60 mm.

Samples were also collected by dry drilling of ten 40 mm deep holes (Ø 20 mm) in each slab. Drilling of the first five samples was done by using a hand held drill. The last of the ten samples were taken using the same template that was used for the beams. The samples were collected at the following depths: 0-10 mm, 10-20 mm, 20-30 mm and 30-40 mm using the same procedure as in section 3.1.1. Between each drilling step, dust was removed from the holes by using compressed air.

3.2 Grinding

The mass of the outer and inner parts of the beam slices were 1500 g and 1200 g respectively. Prior to the chloride analysis, the concrete dust from each part was homogenized in a rotating chamber.

The slices from the cores were subjected to the same treatment.

Samples collected by dry drilling were used without additional grinding, and thus only dried before chemical analysis.

3.3 Chemical analysis

The analysis of chlorides was based on the Norwegian Standard, NS 4756 Chloride, potentiometric method /2/. An automatic titration unit of type Metrohm 686, Titroprocessor/Metrohm 665, Dosimat, was used. NS 4756 corresponds to Finnish Standard SFS 3006, Danish Standard DS 239 and Swedish Standard SS 02 81 36.

Chlorides were extracted from the concrete using a modified Norwegian Standard NS 3671 procedure /3/. This procedure was based on extraction with nitric acid and water, leaving the chlorides in an aqueous solution for chemical analysis. The chloride content was determined by titration with silver nitrate:

$$Ag^{+} + Cl^{-} \leftrightarrow AgCl$$

A silver electrode was used to monitor the change in potential in the solution.

A number of 35 samples of documented concrete reference dust containing known chloride concentrations /4/ were randomly incorporated in the sample set to reduce the danger of possible errors in the analysis.

4 DISCUSSION

There are two main possible causes for uncertainties in the chloride measurements in this work. The first is the uncertainty in the chemical analysis. The second is the uncertainty inherent in the specimens. The autotitration equipment used in this work has proved to give good accuracy and precision /4/. The uncertainty in the chemical analysis may therefore be regarded as low compared to the inherent uncertainty. The uncertainty connected to the specimens is governed by several factors. Among these are:

- sample volume and number of samples
- maximum aggregate diameter
- bore size
- chloride concentration

4.1 Sample volume and number of samples

The sample volume will have a great influence on the test results. As a consequence of the concrete being a composite material consisting of paste and a coarse fraction of aggregate, a decrease in sample volume will lead to a reduction in both precision and accuracy. The results show that the chosen diameter of Ø 98 mm, gives measurements with very good precision and accuracy. The results from sampling by dry drilling, however, show results with reduced precision and accuracy. This is illustrated in Figures 4.1, 4.2, 4.3 and 4.4, and does probably reflect the effect of differences in sample volume achieved with the two techniques. To get the same amount of sample volume using a 20 mm drill as with 98 mm diameter drilled cores approximately 20 holes have to be drilled. This is of course neither a practical nor an economical solution to the problem. Often no more than three parallel holes are drilled when samples are collected from field constructions.

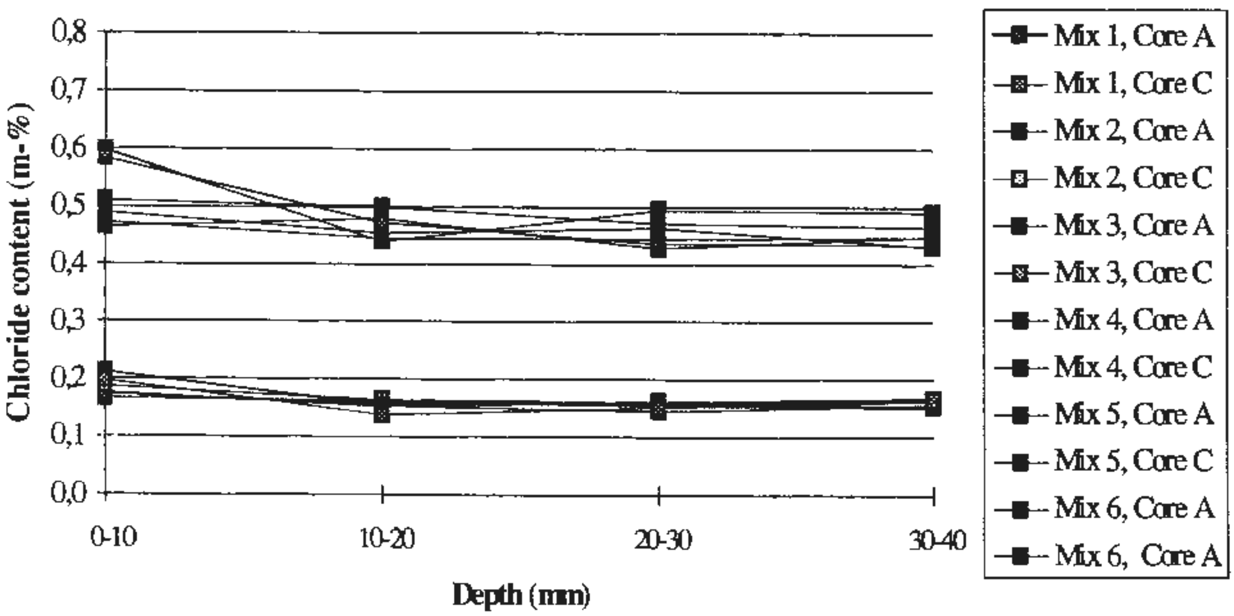

Fig. 4.1 Chloride content of beam slices. Chloride content in % by weight of concrete.

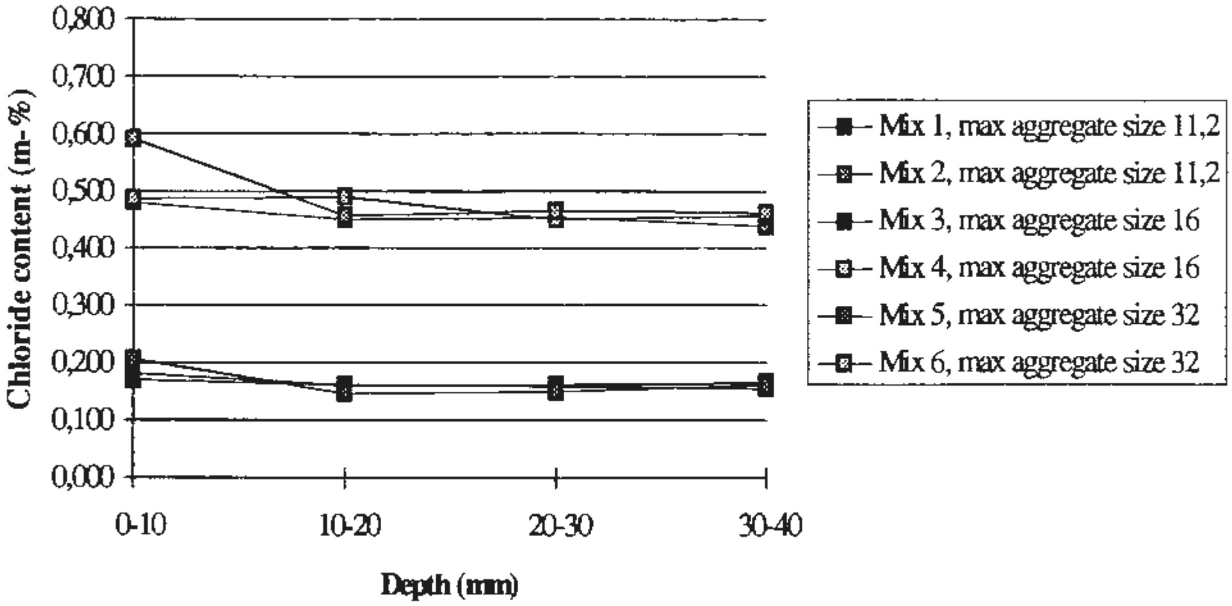

Fig. 4.2 Mean values of chloride content of beam slices. Chloride content in % by weight of concrete.

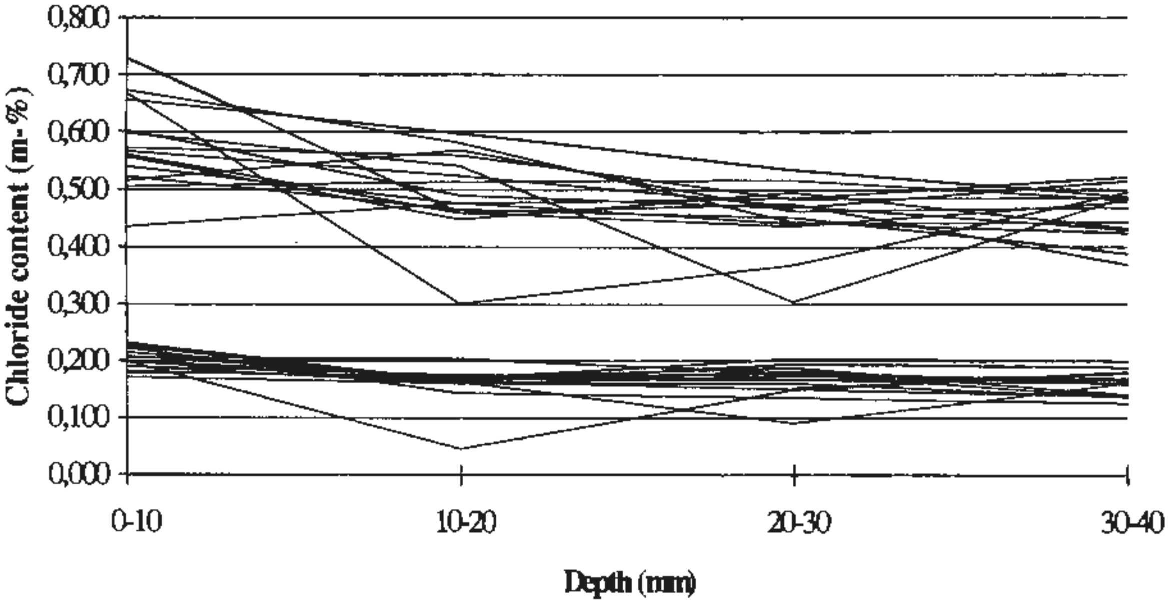

Fig. 4.3 Chloride content of dry drilled dust from beams. Chloride content in % by weight of concrete.

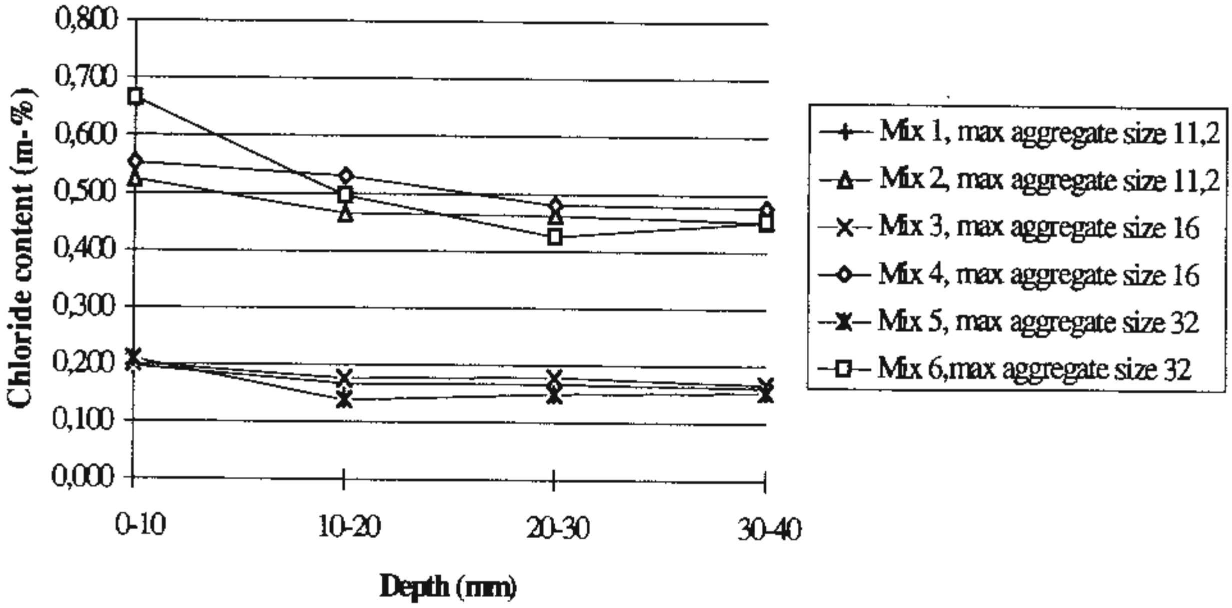

Fig. 4.4 Mean values of chloride content of dry drilled dust from beams. Chloride content in % by weight of concrete.

4.2 Effect of maximum aggregate diameter

Chlorides in concrete exist mainly in the hydrated cement paste. When regarding the danger of corrosion to reinforcement steel it is therefore the chloride content expressed in relation to the amount of cement that is important. The presence of aggregate will add an uncertainty to the determination of a chloride content and therefore displace the ratio between the amount of chloride and cement paste. The larger the aggregate diameter is compared to the sampling bore diameter, the larger is the uncertainity. This is also demonstrated by the results that are obtained in this work. The results are visualized in Fig 4.5 below, where observed maximum deviations are plotted against different maximum aggregate diametres.

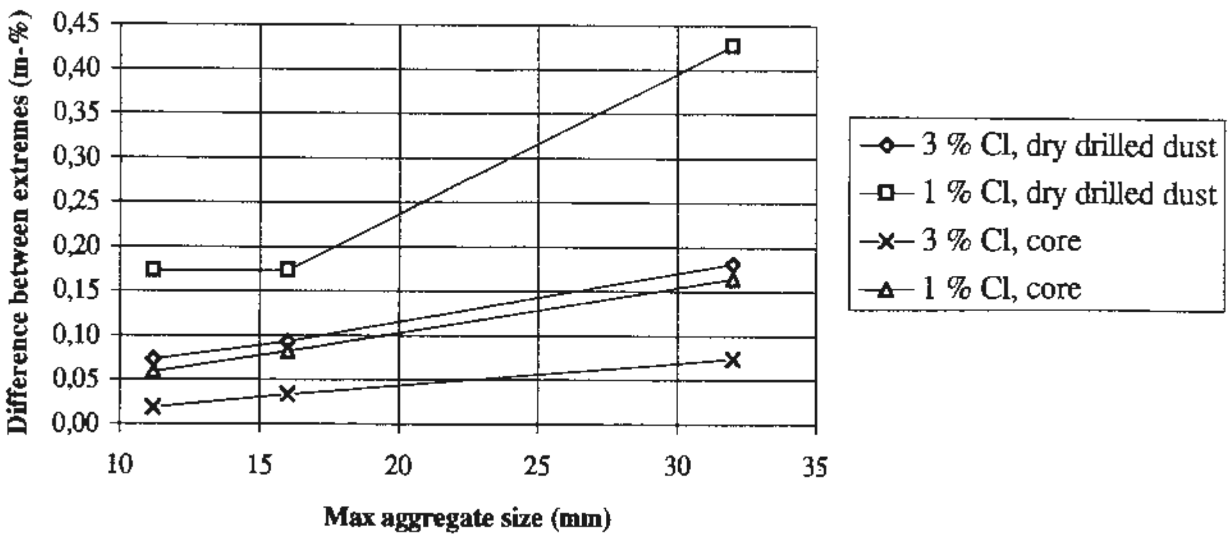

Fig 4.5 Maximum observed deviations in chloride measurements plotted vs. maximum aggregate diameter. The two lower curves represent values measured in cores. The two upper curves represent dry drilled samples. Chloride content in % by weight of concrete.

The differences in deviations observed with the two sampling techniques is apparent. With 5 dry drilled samples as used here, there is also a distinct change in deviation between aggregate diametres above and below the bore diameter.

There is no significant difference between drilling with or without a template.

4.3 Bore size

As seen in the previous section, the bore size is a critical parameter. It has importance both for cores and dry drilled dust. The effect is considerably larger, however, for the latter sampling technique.

A bore diameter of 20 mm must be considered to be close to an upper practical limit for hand held operation. Five samples may therefore only be recommended for concretes with maximum aggregate sizes smaller than 16 mm. Larger aggregate sizes will require more samples to achieve acceptable certainty.

4.4 Effect of chloride concentration

The certificates from Taylor Woodrow /4/ reported an increase in standard deviation for samples with a decreasing chloride content. The two chloride concentrations tested in this programme did not show similar results.

5 CONCLUSIONS

The analysis shows good accuracy and precision in measured chloride contents for cores drilled from specimens of known concentration (1.0 % and 3.0 %). Correlation between parallel cores is good.

Measured chloride contents from dry drilled dust using five parallel samples show poorer but acceptable accuracy and precision only for max. aggregate sizes below bore diameter, i.e. 11.2 mm and 16 mm. For max. diameter 32 mm, absolute deviations of single measurements up to about 40 % from the mean chloride value is observed. The same pattern is reflected when chloride profiles are measured.

Analysis of dry drilled dust therefore indicates that a bore diameter above maximum aggregate size is essential in order to obtain sufficient accuracy and precision in chloride measurements.

A bore diameter of 20 mm must be considered to be close to an upper practical limit for dry drilling. For maximum aggregate sizes above this a number of five holes must be regarded as insufficient. Further gain in accuracy and precision may only be achieved through an increase in the number of holes.

6 REFERENCES

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