

# Verification of Measurement Accuracy of Test Method for Chloride Ion Content in Hardened Concrete by Drilling Powder

Koji Kawamata<sup>1</sup>, Daijiro Hayakawa<sup>2</sup>,  
Hidekazu Tanaka<sup>3</sup>, Masaaki Mashimo<sup>4</sup>, Hiroshi Yashima<sup>5</sup>

<sup>1</sup> Chuken Consultant Co., Ltd.  
6-28, Rokuban-Cho, Chiyoda-Ku, Tokyo, 102-8465, JAPAN  
(+81)-3-5211-4852; Fax(+81)-3-3221-4605; Email: kkawamata@ccc.soc.co.jp

<sup>2</sup> Hachiyo Consultant Co., Ltd.  
2722, Hagisono, Chigasaki-Shi, Kanagawa, 253-0071, JAPAN  
(+81)-467-87-3451; Fax(+81)-467-57-0618; Email: d-hayakawa@hachiyo.co.jp

<sup>3</sup> Taiheiyo Consultant Co., Ltd.  
2-27-8, Higashi-Nihonbashi, Chuo-Ku, Tokyo, 103-004, JAPAN  
(+81)-3-5820-5603; Fax(+81)-3-5820-5608; Email: Hidekazu\_Tanaka@taiheiyo-c.co.jp

<sup>4</sup> Denka Renotec Co., Ltd.  
3-5-1, Asahimachi, Machida-Shi, Tokyo, 194-8560, JAPAN  
(+81)-42-721-3661; Fax(+81)-42-721-3698; Email: masaaki-mashimo@denka.co.jp

<sup>5</sup> Fuji Consultant Co., Ltd.  
64, Nakamachi, Muroran-Shi, Hokkaido, 050-0087, JAPAN  
(+81)-143-43-7073; Fax(+81)-143-41-1090; Email: h-yashima@f-consul.co.jp

## Abstract

In JIS A 1154 “Methods of test for chloride ion content in hardened concrete”, it is stipulated that hardened mortar, concrete cylinders, cores, slices in the depth direction and drilling powder should be used for the measurement of chloride ion content in hardened concrete. When drilling powder is used, a large scatter of the measurements is pointed out due to a small sampling quantity and different constitution rates between cement paste and aggregates in each drilling. In this research, concrete test specimens were made of a different coarse aggregate having a maximum size, a different chloride ion content and a different chloride penetration depth. The influence of drill diameters and drilling positions on the measurements of chloride ion content is discussed in this paper in order to recommend the optimum sampling method for drilling powder.

**Key words** : salt damage, chloride ion, hardened concrete, drilling powder, round robin test

## 1. INTRODUCTION

Japan being an island archipelago has a large number of concrete structures constructed along its long coastline, and salt damage to these structures due to airborne chlorides based on seawater is a very serious problem. Further, in mountainous regions, salt damage due to the spraying of anti-freezing material in the winter times is also seen as a problem. In order to understand the deterioration due to such salt damage, measurement of the amount of chloride ions in the concrete is extremely important. In Japan, the method of testing stipulated in JIS A 1154<sup>1)</sup> is being used as a general method for testing for the amount of such chloride ions. While concrete cores taken from the structures are very often used as specimens in this test, the method<sup>2)</sup> of using drilling powder is drawing much attention considering the ease and convenience at the test site. Although JSCE-G 573-2013<sup>3)</sup> stipulates a method of collecting the specimens, it also notes that “When using drilling powder as specimens, the amount collected is small and the constituent ratio of the cement paste and the aggregate differs among the drilled holes, which can also be considered to have effect on the results of the tests”. In addition, there have also been reports<sup>4)</sup> that suggest that differences in the specimen collection conditions influence the results of the tests.

This study, as joint experiments by six member-companies of our society of concrete consultant companies, was carried out with the purpose of obtaining samples for investigating the optimum drilling powder collection method. We prepared specimens simulating the actual structures, checked the effect that various factors such as the drill diameter and collection position, maximum size of the coarse aggregate (Gmax), amount of chloride ion

content, etc., had on the results of the chloride ion content experiments. In addition, we also report the results of round robin tests carried out among the different consultant companies that are members of the society.

## 2. EXPERIMENTAL

### 2.1 Experiments Outline

Firstly, concrete specimens were prepared that simulated the actual structures (Table 1) having different chloride ion contents and maximum size of the coarse aggregate. Next, drilling powder was collected from these prepared specimens based on the test factors and levels (Table 2), and these specimens were sent to the testing facilities of each of the consulting companies. In the testing facilities of each consulting company, the collected samples were subjected to the chloride ion content tests based on JIS A 1154<sup>1)</sup>. Further, regarding a part of the drilling powder samples, the ratio of the aggregate in the sample was obtained. The ratio of the aggregate was calculated by obtaining the insoluble residue in the sample. These results were collected and studied. The flow of executing the tests is shown in Figure 1.

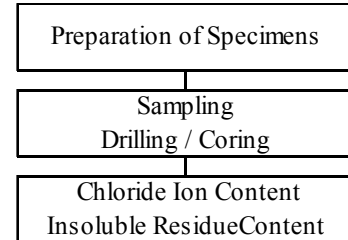


Figure 1: Experiments Outline.

### 2.2 Materials

The Materials used for this research are as follow:

- (1) **Cement;** Ordinary Portland cement(C), complying with the JIS R 5201 with density of  $3.16\text{g/cm}^3$
- (2) **Fine aggregate;** Crushed sand (S), complying with JIS A 5005, dry density;  $2.58\text{g/cm}^3$ .
- (3) **Coarse aggregates;** Crushed gravel (G1:4020), complying with JIS A 5005, dry density;  $2.47\text{g/cm}^3$  (G2:2005), complying with JIS A 5005, dry density;  $2.66\text{g/cm}^3$ , G1:G2 = 50:50 Wt%
- (4) **Admixtures;** Air-entraining admixture (Ad) with a density of  $1.04\text{g/cm}^3$ , complying with JIS A 6204.

### 2.3 Mixtures and nomenclature of the specimens

The shape of the prepared specimens was that of a square pillar of dimensions  $500\text{mm} \times 500\text{mm} \times 800\text{mm}$  as shown in Figure 2. A total of 4 specimens were prepared, with the two levels of maximum coarse aggregate size of 20mm and 40mm, and the three levels of chloride ion concentrations of 0.3, 1.2, and  $5.0\text{ kg/m}^3$ . The details of these specimens and the mixing proportions are shown in Table 1. The prepared specimens were cured inside the frames up to material age of 28 days. However, in the following report, these symbols are used for the numbers of each of these specimens.

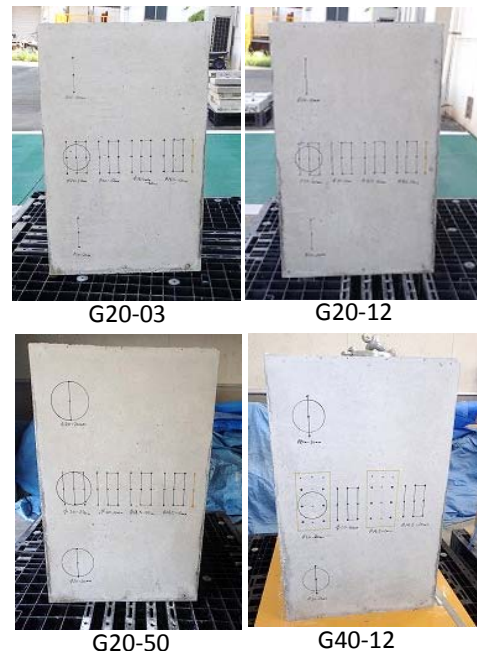


Figure 2: View of the concrete specimens.

Table 1: Mix proportion.

Case No.	G <sub>max</sub> (mm)	Cl <sup>-</sup> ( $\text{kg/m}^3$ )	W/C (%)	S/a (%)	Slump (cm)	Air (%)	(kg/m <sup>3</sup> )				
							W	C	S	G	Ad
G20-03	20	0.3	55	45	8	4.5	165	300	808	1016	0.005
G20-12		1.2					165	300	808	1016	0.005
G20-50		5.0					165	300	808	1016	0.005
G40-12	40	1.2					155	282	771	1055	0.006

### 2.4 Parameters of the experiment

The combinations of levels of the test factors and the collected samples are shown in Table 2.

In order to study the effect of the method of collection of the drilling powder samples, the drill diameter, drilling depth, and the number of drillings for the sample were set as the factors. The drill diameters were of the two levels of  $\phi 20\text{mm}$  and  $\phi 14.5\text{mm}$ . The drilling depth was of the two levels of 10 mm and 20 mm, and the number drillings for the sample was of the three levels of 1 drilling, 3 drillings, and 6 drillings.

Next, in order to study the effect of the base concrete material, we set the chloride ion concentration, and the maximum size of the coarse aggregate (Gmax) of the base concrete material, and the position of collecting the drilling powder samples as the factors. The chloride ion concentration was of the three levels of 0.3, 1.2, and 5 kg/m<sup>3</sup>. Gmax was of the two levels of 20mm and 40mm, and the sample collecting position was of the three levels of top, middle, and bottom portions.

The test results of these drilling powder samples were studied comparatively taking as the reference values the test results with the concrete core samples that were collected at the same time.

**Table 2: Factors and levels.**

Location	Drill Diameter [Core] (mm)	Drilling Depth [Core] (mm)	Number of Drillings	Gmax20mm			Gmax40mm
				Cl <sup>-</sup> 0.3kg/m <sup>3</sup>	Cl <sup>-</sup> 1.2kg/m <sup>3</sup>	Cl <sup>-</sup> 5.0kg/m <sup>3</sup>	Cl <sup>-</sup> 1.2kg/m <sup>3</sup>
Middle	20	20	1	-	-	●	-
			3	●	●	●	●
			6	●	●	●	●
		10	3	●	●	●	●
			6	●	●	●	●
			6	●	●	●	●
	14.5	20	1	-	-	●	-
			3	●	●	●	●
			6	●	●	●	●
		10	3	●	●	●	●
			6	●	●	●	●
			6	●	●	●	●
	[Gmax20=75]	[Gmax20=100]	1	●	●	●	●
Upper	20	20	3	●	●	●	●
	[75]or[100]	[100]or[120]	1	—	—	●	●
Lower	20	20	3	●	●	●	●
	[75]or[100]	[100]or[120]	1	—	—	●	●

## 2.5 Round robin test

Round robin tests were carried out among the different testing facilities of all the participating consulting companies. In the tests, the concrete cores collected at the Middle with the four types of chloride ion concentrations were taken as the common test samples.

## 3. RESULTS AND DISCUSSIONS

### 3.1 Diameter of drill bit

The effect of the drill bit diameter is shown in Figure 3. From this, firstly, irrespective of the drill bit diameters, the test results with drilling powder exhibited high values compared to the test results of concrete cores taken as the reference.

Next, considering the effect of the drill bit diameter, no clear trend was found in the results of the three types of chloride ion concentrations for the Gmax of 20mm. However, in the results for the Gmax of 40mm, the results of  $\phi$ 20mm and  $\phi$ 14.5mm clearly differed, and the values of  $\phi$ 14.5mm become high.

From these results, while it is not necessary to pay attention to the drill bit diameter with the normal aggregate dimensions (Gmax 20mm), it can be understood that it is desirable to make even the drill bit diameter large when the maximum dimension of the coarse aggregate is large.

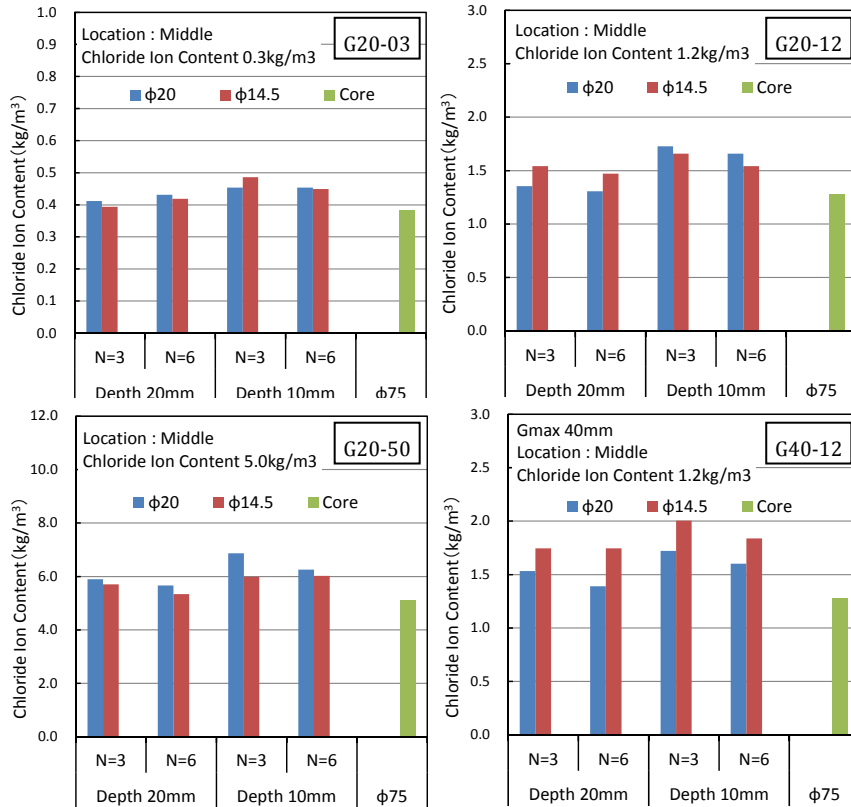


Figure 3: Effect of drill bit diameter.

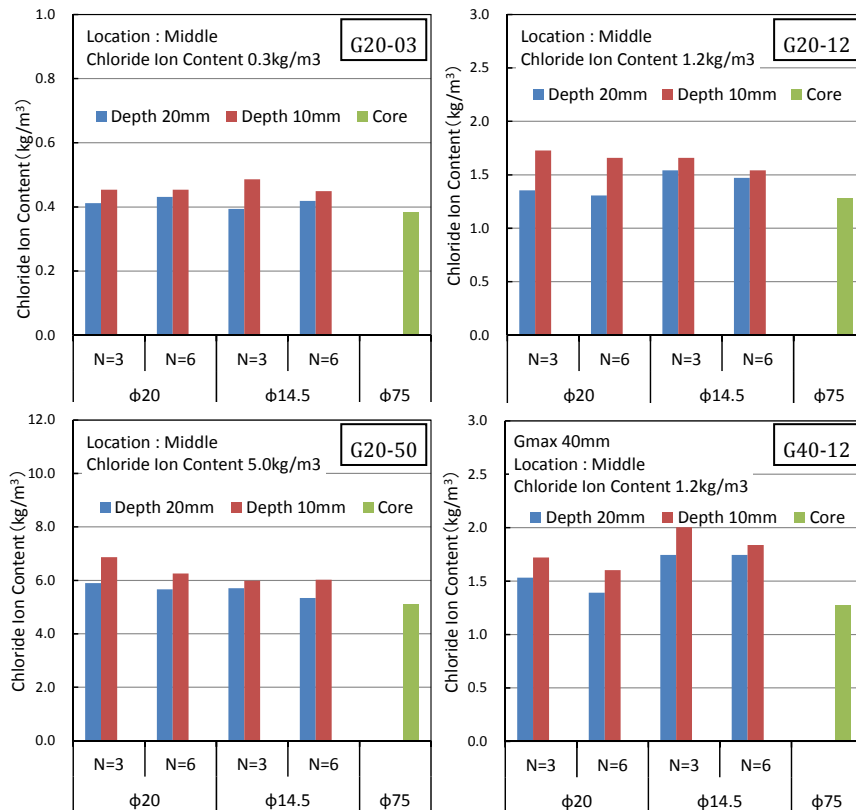
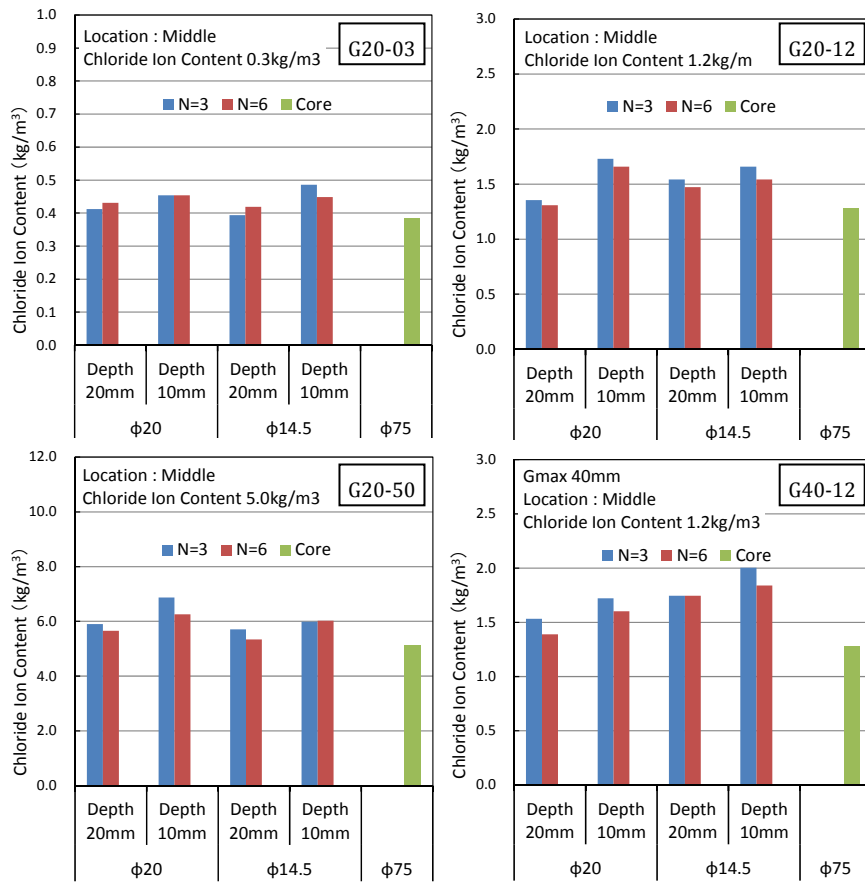
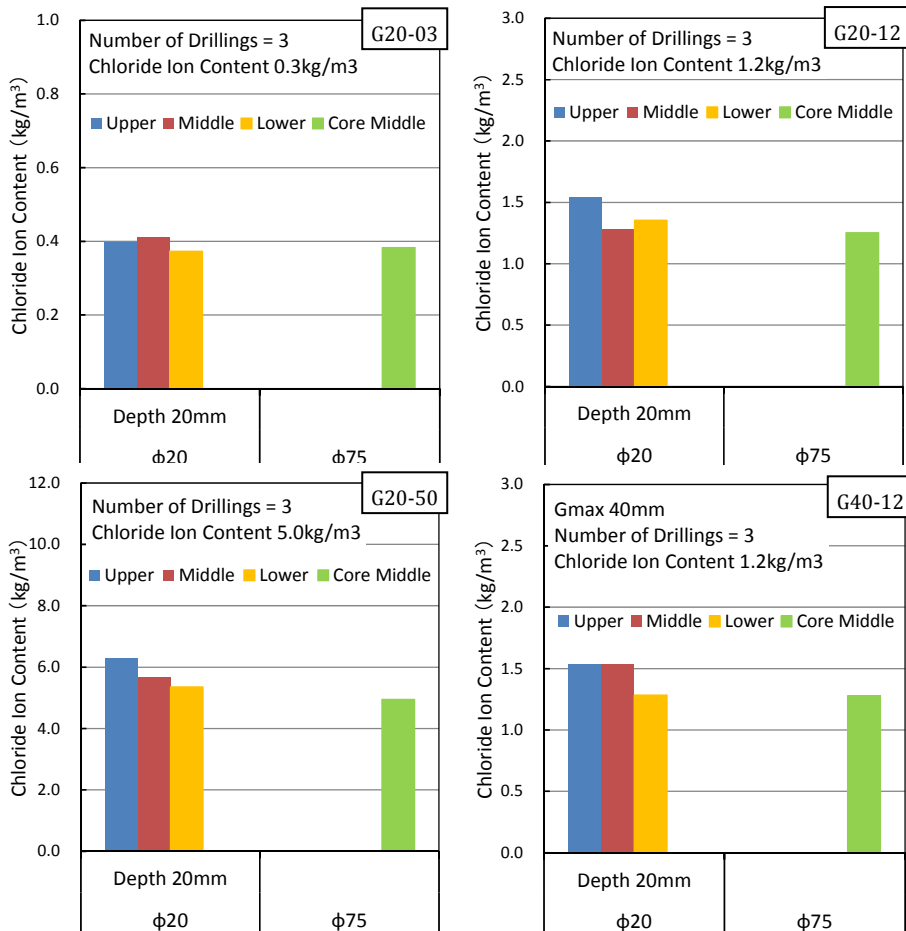


Figure 4: Effect of drill bit depth.



**Figure 5: Effect of number of drillings.**



**Figure 6: Effect of sampling location.**

### 3.2 Depth of drillings

The effect of the depth of drilling is shown in Figure 4. Firstly, irrespective of the depth of drilling, the results of drilling powder became higher than the reference value, and at any level, the drilling depth of 10mm has the trend of increasing the chloride ion concentration than the drilling depth of 20mm. In addition, the drilling depth of 20mm is closer to the reference value. However, when the number of drillings becomes large, the difference in the chloride ion concentration due to differences in the drilling depth becomes small.

### 3.3 Number of drillings

The effect of the number of drillings is shown in Figure 5. From this, when we compare the chloride ion concentration at 3 drillings and 6 drillings, broadly the results of 3 drillings and 6 drillings were either equivalent or the 6 drillings showed a smaller value. The reason for the value being close to the concrete core which is a reference value is the case that 6 drillings were taken irrespective of the drill bit diameter and the drilling depth.

Further, when compared for each specimen, as the number of drillings increases, there is a trend that the range of fluctuations in the obtained result becomes smaller.

From the above results, although there is no problem in practice with 3 drillings, it can be understood that as the number of drillings is made larger, the fluctuations become smaller.

### 3.4 Location of drillings

The effect of the drilling powder collection position is shown in Figure 6. However, since it was found from the results so far that a different trend is found in the surface layer portion, we compared with a drilling depth of 20mm. From this result, when the chloride ion concentrations in the top layer portion and the middle layer portion are compared for the same specimen, they are either broadly equivalent or the top layer portion is high. The chloride ion concentration of the lower layer is relatively low. The values of the concrete cores which become the reference values showed roughly close values in the lower layer portion.

Figure 7 shows the results of measuring the chloride ion concentrations penetrating in the vertical and horizontal directions for all the specimen excepting the specimen of the chloride ion concentration of 0.3kg/m<sup>3</sup>. Further, the aggregate ratio obtained from the Insoluble Residue is shown in the figure. From these results, there were no large differences in the chloride ion concentrations in the depth direction from the placing surface.

However, since cement paste is relatively more in the concrete surface layer side which becomes the form side surface compared to the deep portion, and since the chloride ion concentration becomes a high value, it can be understood that it is necessary to give considerations to drilling powder collection.

### 3.5 Round robin test

Table 3 shows the results of the Round Robin Test. From this, as the chloride ion concentration becomes smaller, there is the trend that the coefficient of variation

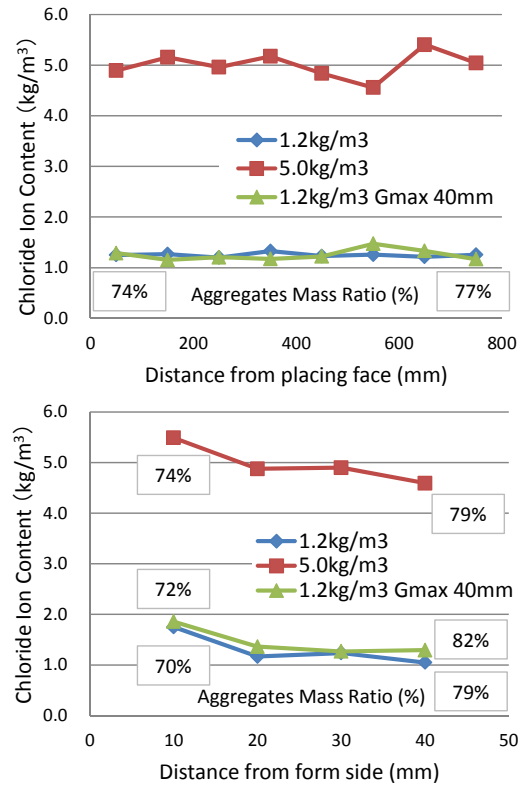


Figure 7: Effect of sampling distance.

Table 3: Results of round robin test.

Chloride Ion Content	0.3kg/m <sup>3</sup>	1.2kg/m <sup>3</sup>	5.0kg/m <sup>3</sup>	1.2kg/m <sup>3</sup> Gmax 40mm
A	0.380	1.257	4.999	1.236
B	0.364	1.186	4.898	1.229
C	0.394	1.305	5.013	1.236
D	0.371	1.236	4.944	1.213
E	0.394	1.259	4.876	1.259
F	0.394	1.282	4.990	1.282
Average	0.383	1.254	4.953	1.243
Standard Deviation	0.012	0.037	0.052	0.022
Coefficient of Variation	3.17	2.98	1.05	1.78

becomes large. However, even in the case of the chloride ion concentration of  $0.3\text{kg/m}^3$  having the largest coefficient of variation, since that coefficient of variation is about 3%, it was confirmed that there was no problem in the results among the testing locations of the six participating companies.

#### 4. CONCLUSIONS

The following conclusions are drawn based on the findings of this study:

- (1) The test results with drilling powder exhibit a large value compared to the test results of the concrete core which is taken as the reference, irrespective of the drill diameter, the drilling depth, and the number of drillings.
- (2) Although it is not necessary to consider the effect of the drill diameter for normal aggregate sizes ( $G_{\text{max}} 20 \text{ mm}$ ), when the maximum size of the coarse aggregate is large, it is desirable to make the drill bit diameter large.
- (3) Since the surface layer of concrete has a high cement paste ratio compared to the deep portion, it is necessary to give considerations to the fact that even the chloride ion concentration becomes large.
- (4) Although there are no problems in practice if 3 drillings are used, as the number of drillings is increased the fluctuations also become small.
- (5) It was confirmed that there was no problem in the results among the testing facilities of the 6 participating companies this time.

#### ACKNOWLEDGEMENT

We express our gratitude to Mr. Konosuke Takahara and Shohei Kimura of Mitsubishi Materials Corporation, Mr. Kazuya Tamugi of Taiheiyo Consultant Co., Ltd., and Mr. Shinichi Hamahira of Chuken Co., Ltd., who had eagerly participated in the progress of the test and in the analysis of the results during the working of the concrete consultant research group.

#### REFERENCES

1. JIS Standards "Method of test for chloride ion content in hardened concrete (JIS A 1154)", Japanese Industrial Standard (JIS), 2012.
2. Yuasa, N., Kasai, Y., and Matsui, I., "A Proposal of Rapid Field Testing Method for Chloride Ion Content in Concrete using Drilled Powder " Proceedings of the Japan Concrete Institute, Vol.21, Japan Concrete Institute(JCI), pp.1303-pp.1308, 1999.
3. JSCE Standards "Measurement Method for Distribution of Total Chloride Ion in Concrete Structure (JSCE-G 573-2003)", Japan Society of Civil Engineers (JSCE), 2003.
4. Birumachi, M., Yuasa, N. and Yamada, Y., "Influence of core and drill diameter on measured chloride ion content in concrete – Results in the passage of one year after exposing –" Preprint for the Annual Meeting, College of Industrial Technology, Nihon University, pp.829-pp.832, 2012.