

Method of effective evaluation for examination of chloride ion in concrete^{*}

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Abstract: The chloride ion contained in reinforced concrete seriously corrodes the steel surface and damages concrete, resulting in inferior reinforced concrete that strength seriously compromises the entire structure's safety. Consequently, the examination of chloride ions contained in reinforced concrete becomes an important part of a complete quality control procedure. To effectively check the concentration of chloride ions in concrete, the evaluation process should be accurate and precise. Laboratory data obtained using existing evaluation methods for the examination of chloride ion are not sufficiently objective to yield reliable results with accuracy and consistency for each sample. An evaluation algorithm with capability to define indices of precision degree (E_p) and accuracy degree (E_a) is presented in this paper. The authors established a statistically reliable index of unbiased estimators and equations to critically examine the laboratory methods' precision, accuracy degrees and application value for measuring chlorine ion concentration in reinforced concrete.

Key words: Concrete quality control, Multi-laboratory, Test error, Accuracy degree, Precision degree, Natural estimator, Acceptance testing, Examination methods, Evaluation criteria

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INTRODUCTION

For some unknown reasons, perhaps caused by salt, chlorine ions usually exist in reinforced concrete consisting of cement, water, aggregate and additional materials, and steel bars. In the presence of water, the chlorine ion reacts with iron existing in concrete and steel bars to form $\text{Fe}(\text{OH})_2$ and FeCl_2 . The chemical reactions seriously corrodes the surface of steel bars and damage the concrete, causing gradual decrease of the concrete compression and increase of its porosity, thus accelerating the water percolation rate. As a result, the quality of the reinforced concrete deteriorates to affect the safety and serviceability of the structures. Thus, a precise examination of the chlorine

ion concentration in concrete is needed for better quality control. Based on the result thus obtained, field engineers can get a better understanding for structure inspection and concrete manufacturers can make necessary adjustments to improve the quality of concrete. Consequently, degrees of accuracy and precision in the examination of chlorine ion in the concrete are essential elements for the quality evaluation of any reinforced concrete structure.

The methods currently used for examination of chlorine ions in concrete are based on the AASHTO T260, JASS 5T, ACI 318 and ASTM C114 code (Kosmatka and Panarese, 1988; Mindess and Young, 1981). Water-soluble method and acid soluble method are two major methods for testing the deviation range of chlorine ion in concrete. Research institutes and field inspection units are constantly developing new methods and instruments with im-

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proved efficiency, accuracy and cost-effectiveness. Actually, evaluating the efficacy of chlorine ion examination is similar to that used in evaluating the process capability of machinery to manufacture products. Thus, the technology of engineering quality analysis for evaluating the process capability of test tools in the manufacturing process is applied for the examination of chlorine ion. Some effective methods proposed by researchers such as Kane (1986), Chan *et al.* (1988), Chou and Owen (1989), Boyles (1991; 1994), Pearn *et al.* (1992), Greenwich and Jahr-Schaffrath (1995) and Chen (1998a; 1998b), and many others provided accurate evaluation of the industrial process capability and efficacy. The indices developed using these methods provide convenient and effective tools for objectively evaluating the production capability and performance with defined degrees of precision and accuracy. In this paper, the efficiency of chlorine ion test equipments and methods can be verified using these indices to derive an accurate estimation. A criterion based on the principle of statistics to evaluate the process performance of the test tools for measuring chlorine ion concentrations in concrete is presented. This criterion provides a simplified method for critically reviewing the process performance of the test tools for measuring chlorine ion concentrations in concrete and for evaluating the precision and accuracy degrees. Actually, the proposed method is expected to detect and solve problems in the manufacturing process.

THE PERFORMANCE INDEX FOR THE EXAMINATION OF CHLORIDE ION IN CONCRETE

The process performance index for examining chlorine ion in concrete is numerical, and evaluates whether the test tools achieve their required efficacy or not. To evaluate the efficacy of test tools, blind samples must be taken as a target value T . Assuming that X is a blind sample used to evaluate any test tools, because the inspected value is unknown, the evaluation of test tools is a random experiment where X is a random variable. If X obeys the normal distribution law with mean value μ and variance value σ^2 , it is denoted as $X \sim N(\mu, \sigma^2)$. The closer the mean value μ approaches the target value T , the higher is observed accuracy and a lower σ^2 will lead to higher precise.

Let d be the maximum allowable error, the tolerance interval is $T \pm d$ in which $U(=T+d)$ and $L(=T-d)$ represent the upper and lower bound of tolerance, respectively. In accordance with ASTM C670, that the precision degree should be determined by 2σ is assumed to follow the normal distribution $X \sim N(\mu, \sigma^2)$ and about 95% lies in the interval $\mu \pm 2\sigma$, or $F_x(\sigma+2\sigma) - F_x(\sigma-2\sigma)$ nearly equals 95%. $F_x(\cdot)$ is the X cumulative function of distribution. Comparing the actual test distribution with the tolerance interval, the evaluation index of precision degree for the manufacturing process can be defined as:

$$E_p = \frac{d}{2\sigma}, \quad (1)$$

For the case of $\mu = T$, the probability of real inspected values will exceed the upper bound for $E_p = 1.5$ and the lower bound for $E_p = 1.0$. Thus the probability of erroneous estimation lies between 4.56% and 0.27%. In Eq.(1), d is a constant implying that a lower index value will result in lower variance value σ^2 , and a higher precision degree will be detected. Hence, E_p can be declared as the imprecision index for testing methods and tools. Using the index of precision degree E_p , the accuracy index of test tool can be defined as follows:

$$E_a = 1 - \frac{|\mu - T|}{d}, \quad (2)$$

The value of E_a should be less than or equal to 1 ($E_a \leq 1$). When μ approaches T , i.e., E_a approaches 1, the accuracy degree is satisfactory. When $E_a = 1$, the inspected enumeration of this examination method equals to the target value T , i.e. $\mu = U$ or $\mu = L$. If $E_a < 0$, the test value μ is located outside the tolerance interval. Thus, the manufacturing unit can improve the test tools based on the values of E_a . Table 1 shows the relationship between various E_a and μ .

When the difference between the examined value and real value is less than the tolerance value d , the test method attains the required specification. Otherwise, the test tool is unacceptable. The risk of deviated rate (p) calculated using $1 - [F(U) - F(L)]$ is proposed in this paper, in which $F(X)$ is the cumulative function of distribution with random variable X .

Table 1 The relationship between various E_a and μ

E_a	μ
$E_a = 1.0$	$\mu = T$
$E_a = 0.8$	$\mu = T \pm (0.2) d$
$E_a = 0.6$	$\mu = T \pm (0.4) d$
$E_a = 0.4$	$\mu = T \pm (0.6) d$
$E_a = 0.2$	$\mu = T \pm (0.8) d$
$E_a = 0.0$	$\mu = L$ or $\mu = U$
$E_a < 0.0$	$\mu < L$ or $\mu > U$

According to the assumption of a normal distribution, the risk of deviated rate p can be expressed as follows:

$$p = 2 - \{ \Phi(2E_p E_a) + \Phi(2E_p [2 - E_a]) \} \quad (3)$$

where: $\Phi(\cdot)$ is the cumulative function of standard normal distribution.

Obviously, when E_a equals 1, the risk of deviated rate p is the function of E_p ($p = 2 - 2\Phi(2E_p)$). This equation shows that a larger E_p will mean a lower risk of deviated rate. For example, when $E_p = 1.0$, the risk of deviated rate is about 4.56%. If the value of E_p is smaller than 1.0, the risk of deviated rate is larger than 4.56%. This indicates that the index of precision degree E_p is closely relative to the risk of deviated rate p . Table 2 lists the relationship between the index of precision degree and the upper limit of the risk of deviated rate p for various values of E_a and E_p .

Table 2 The relationship between p , E_p and E_a

E_p	$E_a=1.0$	$E_a=0.8$	$E_a=0.6$	$E_a=0.4$	$E_a=0.2$	$E_a=0.0$
0.5	0.31731	0.32693	0.35501	0.39938	0.45667	0.52275
1.0	0.04550	0.06300	0.11762	0.21254	0.35574	0.50003
1.5	0.00270	0.00836	0.03594	0.11507	0.27425	0.50000
2.0	0.00006	0.00069	0.00820	0.05480	0.21186	0.50000

The above analyses demonstrate that once the E_p value of one of the test tools is calculated, the risk of deviated rate p can be quickly estimated using the relationship of index E_p and the risk of deviated rate p .

THE ESTIMATION FOR THE PROCESS PERFORMANCE INDEX

Samples of test tools are denoted X_1, \dots, X_n . To

acquire the natural estimators of E_p and E_a , these n samples are used to calculate their μ and σ values with focus on the inspection of blind samples. The natural estimators can be defined as:

$$\hat{E}_p = b_n \times \left(\frac{d}{2S} \right), \quad (4)$$

$$\hat{E}_a = 1 - \frac{|\bar{X} - T|}{d} \quad (5)$$

Symbols n , $\bar{X} = n^{-1}(\sum_{i=1}^n X_i)$ and $S^2 = (n-1)^{-1} \times \sum_{i=1}^n (X_i - \bar{X})^2$, represent sample size, sample mean and sample variance, respectively. The mean μ and variance σ^2 can be calculated. Thus, equation of b_n (Montgomery, 1985) can be expressed as:

$$b_n = \sqrt{\frac{2}{n-1}} \times \left(\frac{\Gamma[(n-1)/2]}{\Gamma[(n-2)/2]} \right) \quad (6)$$

Obviously, b_n is a function of sample size n . For sample size greater than 26, the equation of b_n can be simplified as:

$$b_n \cong \sqrt{\frac{n-2}{n-1}} \left(1 - \frac{1}{4(n-2)} \right) + \left(\frac{1}{32(n-2)^2} \right) + \left(\frac{5}{128(n-2)^3} \right) \quad (7)$$

Table 3 is the relationship between sample size n and value of b_n .

The statistical equation $(n-1)(b_n)^2(E_p)^2 / (\hat{E}_p)^2$ is

Table 3 The comparison table of each sample size n and value of b_n

n	b_n	n	b_n	n	b_n	n	b_n
3	0.5800	9	0.9650	15	0.9810	21	0.9869
4	0.7250	10	0.9693	16	0.9823	22	0.9876
5	0.7980	11	0.9727	17	0.9835	23	0.9882
6	0.8410	12	0.9754	18	0.9845	24	0.9887
7	0.9515	13	0.9776	19	0.9854	25	0.9892
8	0.9594	14	0.9794	20	0.9862	26	0.9896

$$b_n \cong \sqrt{\frac{n-2}{n-1}} \left(1 - \frac{1}{4(n-2)} \right) + \left(\frac{1}{32(n-2)^2} \right) + \left(\frac{5}{128(n-2)^3} \right), n > 26$$

a chi-square distribution with $n-1$ degrees of freedom based on the assumption of normality. In order to obtain the cumulative distribution function and the probability density function of \hat{E}_a , two parameters λ and Y are defined in this paper as:

$$(1) \lambda = (4n)(E_p)^2(1-E_a)^2 \tag{8}$$

$$(2) Y = n(\bar{X} - T)^2 / \sigma^2 \tag{9}$$

where: Y obeys a non-central chi-square distribution with one degree of freedom and the non-centrality parameter $\delta = \lambda^{1/2}$.

The cumulative distribution function of Y can be redefined as:

$$f_Y(y) = \sum_{l=0}^{\infty} P_l(\lambda) f_{Y_l}(y), \tag{10}$$

where: $P_l(\lambda) = e^{-\lambda/2} (\lambda/2)^l / (j!)$; Y_j is chi-square distribution of χ_{1+2j}^2 .

The above analyses can be synthesized to rewrite the estimator \hat{E}_a as:

$$\hat{E}_a = 1 - \frac{\sqrt{Y}}{\sqrt{nE_p}} \tag{11}$$

Since $P(\hat{E}_a \leq x) = P(\sqrt{Y} \geq (1-x)) = 1$, then, $F_{\hat{E}_a}(x) = 1$ as $x > 1$. If $x \leq 1$, the cumulative distribution function of \hat{E}_a becomes:

$$\begin{aligned} F_{\hat{E}_a}(x) &= P(\hat{E}_a \leq x) = P\left(1 - \frac{\sqrt{Y}}{\sqrt{nE_p}} \leq x\right) \\ &= 1 - P(Y < n(E_p)^2(1-x)^2) \\ &= 1 - \sum_{l=0}^{\infty} P_l(\lambda) \int_0^{n(E_p)^2(1-x)^2} f_{Y_l}(y) dy \end{aligned} \tag{12}$$

Based on Leibnitz's rule, the cumulative distribution function of \hat{E}_a is defined as:

$$f_{\hat{E}_a}(x) = \sum_{j=0}^{\infty} P_j(\lambda) (2n)(E_p)^2(1-x) f_{Y_j}[n(E_p)^2(1-x)^2]$$

$$\begin{aligned} &= \sum_{j=0}^{\infty} P_j(\lambda) \times \left(\frac{(\sqrt{nE_p})^{2j+1} (1-x)^{2j}}{\Gamma(j+1/2) 2^{j-1/2}} \right) \\ &\times e^{-n(E_p)^2(1-x)^2/2}, \quad x \leq 1 \end{aligned} \tag{13}$$

The probability density function of \hat{E}_a can be derived as:

$$\begin{aligned} f_{\hat{E}_a}(x) &= \begin{cases} 4A_n \sum_{j=0}^{\infty} P_j(\lambda) (B_j) \frac{(\sqrt{nE_p})^{n+2j}}{a^{2j+1}} \int_0^{\infty} (1-yz/3)^{2j} z^{n-1} \\ \times \exp\left\{-\frac{n(E_p)^2}{18a^2}(a^2z^2 + (9-yz)^2)\right\} dz, & x \leq 0, \\ 4A_n \sum_{j=0}^{\infty} P_j(\lambda) (B_j) \frac{(\sqrt{nE_p})^{n+2j}}{a^{2j+1}} \int_0^{3/y} (1-yz/3)^{2j} z^{n-1} \\ \times \exp\left\{-\frac{n(E_p)^2}{18a^2}(a^2z^2 + (9-yz)^2)\right\} dz, & x > 0. \end{cases} \end{aligned} \tag{14}$$

where:

$$A_n = \frac{1}{(3\sqrt{2})^n \Gamma((n-1)/2)}; \quad B_j = \frac{1}{2^j \Gamma((2j+1)/2)}.$$

The expected value and variance of \hat{E}_p can be calculated as:

$$E(\hat{E}_p) = E_p; \tag{15}$$

$$Var(\hat{E}_p) = \left\{ (b_n)^2 \times \left(\frac{n-1}{n-3} \right)^2 - 1 \right\} (E_p)^2. \tag{16}$$

Herein, the unbiased estimator \hat{E}_p , which is a function of the complete, sufficient statistics S^2 , can be considered as the optimal estimator of E_p . Its expected value and variance can be calculated as follows:

$$\begin{aligned} E[\hat{E}_a] &= E_a - (2\sqrt{n\pi}E_p)^{-1} \{ \sqrt{2} \exp(-\delta/2) \} \\ &+ 2(1-E_a) \Phi\{-2\sqrt{n}E_p(1-E_a)\} \end{aligned} \tag{17}$$

$$Var[\hat{E}_a] = (E_a)^2 + [9n(E_p)^2]^{-1}$$

$$\begin{aligned}
 &-(2\sqrt{n\pi}E_p)^{-1}[2\sqrt{2}\exp(-\delta/2)] \\
 &+4(1-E_a)\Phi\{-2\sqrt{n}E_p(1-E_a)\}-E[\hat{E}_a]^2 \quad (18)
 \end{aligned}$$

where: $\delta = 9n\{E_p(1-E_a)\}^2$.

THE EVALUATION CRITERIA FOR THE TEST TOOL OF CHLORIDE ION

The index E_a is proved as an excellent measure to evaluate the effectiveness in the examination of chlorine ion in concrete. If the index E_a is less than the standard value V_1 , the accuracy degree of the test tool is unsatisfactory. The standard value V_1 can be specified based on the weight and measure standards published in Central National Standard or International Inspection Standard. To verify whether the accuracy degree of a testing tool up to standard, the following statistical testing hypothesis can be considered.

$$\begin{aligned}
 H_0: E_a \leq V_1 \\
 H_1: E_a > V_1 \quad (19)
 \end{aligned}$$

The estimator \hat{E}_a is used in the test statistics. The test rule (the critical region) can be defined as $C = \{\hat{E}_a | \hat{E}_a \leq V_a\}$. The critical value V_a can be determined by

$$V_a = V_1 + t_{\alpha_1}(n-1) \times \left(\frac{b_n}{\sqrt{n}\hat{E}_p} \right), \quad (20)$$

It satisfies

$$P\{\hat{E}_a \leq C_a | E_a \geq V_1\} \leq \alpha_1 \quad (21)$$

where: $t_{\alpha_1}(n-1)$ is the upper α percentile of the t distribution with $n-1$ degrees of freedom.

Similarly, the following statistical testing hypothesis can also determine the precision degree. If the precision degree is greater than V_2 , the test method is unqualified. Assuming that the minimum requirement of the precision degree of the test tool is $E_p > V_2$, the null hypothesis and alternative hypothesis can be expressed as:

$$\begin{aligned}
 H_0: E_p \leq V_2 \\
 H_1: E_p > V_2 \quad (22)
 \end{aligned}$$

The critical value V_p can be determined using the following equation

$$\begin{aligned}
 P(\hat{E}_p \geq V_p | E_p = V_2) &= \alpha_2 \\
 P\{K \leq (n-1)b_n^2(V_2/V_p)^2\} &= \alpha_2 \quad (23)
 \end{aligned}$$

where: $K = (n-1)b_n^2E_p^2/\hat{E}_p^2$ follows a chi-square distribution with $n-1$ degrees of freedom.

Hence,

$$V_p = \frac{\sqrt{n-1} \times b_n \times V_2}{\sqrt{\chi_{\alpha_2}^2(n-1)}} \quad (24)$$

where: $\chi_{\alpha_2}^2(n-1)$ is the lower α_2 percentile of K .

DECISION MAKING

To evaluate whether a chlorine ion test tool reaches the inspection standard, the parameters of process performance, e.g. V_1 , V_2 and risk level of α_1 -risk, α_2 -risk are determined. Subsequently, the value of \hat{E}_a and \hat{E}_p can be calculated from the samples.

The parameter $t_{\alpha_1}(n-1)$ can then be acquired from an appropriate statistical table. Finally, calculating the critical value of V_a can be carried out based on α -risk, \hat{E}_a , V_1 and b_n . If the estimated value of \hat{E}_a is less than the critical value of V_1 , the accuracy degree of this test tool is unsatisfactory. Similarly, the $\chi_{\alpha_2}^2(n-1)$ value can be obtained from an appropriate statistical table, and the critical value of V_p can be calculated based on α_2 -risk, \hat{E}_p , V_2 and b_n . If the estimated value of \hat{E}_p is less than V_2 , the precision degree of the test tool is insufficient. Furthermore, the risk of deviated rate (p) can be used to estimate the error ratio and reasonability of this examination method. In order to clearly understand the whole evaluation procedure, the criteria for the performance of the examination method are established according to the following steps:

Step 1: Determine V_1 , V_2 , α_1 and α_2 based on the

desired quality condition and significance level.

Step 2: Calculate \hat{E}_a and \hat{E}_p from the experimental sample.

Step 3: Select $t_{\alpha_1(n-1)}$ and $\chi^2_{\alpha_2(n-1)}$ from an appropriate statistical table, and compute the critical value V_a and V_p by V_1, V_2, α_1 and α_2 .

Step 4: If \hat{E}_a and \hat{E}_p are greater than the critical values V_a and V_p , the accuracy degree and precision degree satisfy the quality requirement, the manufacturing quality of specified chlorine ion test tool can be accepted.

Step 5: If $E_a \leq V_1$ or $E_p \leq V_2$ which shows the accuracy degree or precision degree is unsatisfactory respectively, it concludes the test tool that fails to meet the desired quality, the risk of deviated rate (p) and reasonability for advanced discussion, and improvement measures.

EXAMPLE

Analyses of experimental data collected by various laboratories are presented to illustrate the procedure and to verify the validity of the method as proposed in this paper. Table 4 lists the accuracy degree of multi-laboratory test results.

Table 4 Accuracy degree of multi-laboratory test results

Chloride percentage density by multi-laboratories	Standard deviation	The acceptance of two testing values
0.0176	0.0030	0.0085
0.0268	0.0031	0.0088
0.0313	0.0032	0.0091
0.0592	0.0037	0.0105
0.1339	0.0048	0.0136
0.2618	0.0069	0.0195

The process performance index for the specified chlorine ion test tool employs a numerical index to assess the manufacturing quality of the test tool to decide whether it matches the required accuracy degree and precision degree. Herein, The chlorine ion density of the test sample is 0.0268 kg/m^3 , and defined as the target value T . In order to satisfy the multi-laboratory accuracy degree and the specification of ASTM C670 code, the maximum allowable error 0.0062 kg/m^3 , the upper tolerance limit ($U=T+d$)

0.033 kg/m^3 , and the lowest tolerance limit ($L=T-d$) 0.0206 kg/m^3 must be met. The examined values of sixty-six chlorine ion test tools are shown in Table 5.

Table 5 The examined values of newly developed test method of chlorine ion in concrete (Unit: kg/m^3)

0.0261	0.0232	0.0269	0.0258	0.0265	0.0271
0.0269	0.0288	0.0232	0.0293	0.0288	0.0279
0.0261	0.0291	0.0275	0.0261	0.0278	0.0282
0.0286	0.0261	0.0264	0.0269	0.0283	0.0283
0.0279	0.0273	0.0278	0.0262	0.0299	0.0282
0.0285	0.0287	0.0281	0.0271	0.0277	0.0267

The procedure for evaluating the performance of test tools is applied:

Step 1: Determine the significance levels of α_1 and α_2 both are equal to 0.05. Quality control unit defines these values. The process performance requirement of the test tool are $V_1=0.75$ and $V_2=1.50$. The null hypothesis and alternative hypothesis of accuracy degree and precision degree of the test tool can be expressed as follows:

Accuracy degree:

$$\begin{cases} H_0 : E_a \leq 0.75 \text{ (unsatisfactory)} \\ H_1 : E_a > 0.75 \text{ (satisfactory)} \end{cases}$$

Precision degree

$$\begin{cases} H_0 : E_p \leq 1.50 \text{ (unsatisfactory)} \\ H_1 : E_p > 1.50 \text{ (satisfactory)} \end{cases}$$

Step 2: Calculate \hat{E}_a ($=0.9140$) and \hat{E}_p ($=2.0797$).

Step 3: Determine V_a and V_p by $V_1, V_2, \alpha_1, \alpha_2, t_{\alpha_1(n-1)}$ and $\chi^2_{\alpha_2(n-1)}$ using appropriate statistics tables.

$$\begin{aligned} V_a &= V_1 + t_{\alpha_1(n-1)} \times \left(\frac{b_n}{\sqrt{n\hat{E}_p}} \right) \\ &= 0.75 + 1.69 \frac{0.9784}{\sqrt{36} \times 2.0797} = 0.8284 \\ V_p &= \frac{\sqrt{n-1} \times b_n \times V_2}{\sqrt{\chi^2_{\alpha_2(n-1)}}} = \frac{\sqrt{35} \times 0.9784 \times 1.50}{\sqrt{20.569}} = 1.9145 \end{aligned}$$

Step 4: $\hat{E}_a = 0.9140 > V_a = 0.8284$,

$\hat{E}_p = 2.0797 > V_p = 1.9145$.

For the above example, both of \hat{E}_a and \hat{E}_p are greater than the critical value V_a and V_p so that the accuracy degree and precision degree of the test tool satisfy the requirement.

CONCLUSION

The quality of concrete greatly affects the safety of a reinforced concrete structure. In order to inspect chlorine ion in concrete with convenience and accuracy, the inspection industries constantly develop and manufacture new test tools but falls behind in developing an effective way to evaluate the process performance of new tools. A simplified, effective and reliable evaluation procedure is presented in this paper to resolve this dilemma and enable the manufacturing industry to objectively examine accurately the quality of test tools. In addition, this evaluation procedure provides the manufacturing industry an analytical approach to improve the concrete production process and quality control capability.

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