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Development of a Rapid Test Bar for Quantitative Detection of Chloride Ions in Fresh Concrete

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Abstract:
Due to the shortage of natural raw materials, many practitioners proposed to make concrete from desalinated sea sand. To reduce the risk of abuse of uncleaned sea sand, we proposed a new rapid test bar to detect chloride ions in fresh concrete quantitatively. The test bars were prepared by mixing silver chromate with cotton fiber. By virtue of the high liquid-absorbent capacity of cotton fiber, the chloride-contained liquid in fresh concrete will rapidly enter into the test bar and react with the silver chromate to finally show a certain coloring height, which corresponds to the actual chloride content. In order to optimize the preparation process, the color rendering, testing time and stability of test rods under different mixing ratios and conditions were investigated. The relationship between color height and chloride content was established, and an auxiliary card is prepared for user-friendly calculation of chloride content. The results showed that the best performance of the test bar was obtained under the following conditions: mass concentration of silver chromate 5.0g/L, soaking time 1 min, drying temperature 50℃ and content of discolored silica gel 0.5g/ root.

***Keywords:** Test bar; Chloride ion; Concrete; Cotton fiber*

1 Introduction

Since the emergence of reinforced concrete structures in 1880 and their application in industrial buildings, concrete materials have become indispensable materials in civil construction engineering because of their broad applicability and low cost [1-2]. China is currently in the peak period of infrastructure construction and maintenance, and is the area where reinforced concrete structures are most used in the world. However, the safety and durability of reinforced concrete structures cannot be ignored. Affected by the destruction of the concrete itself and the surrounding environment, a large number of early concrete structures suffer from disease and degradation under the condition of not reaching their service life, which leads to premature failure of the structure and causes enormous direct and indirect losses. Corrosion of steel bars is one of the causes of poor durability of concrete structures. Economic losses and maintenance costs caused by corrosion of steel bars are extraordinary [3-5]. According to the investigation results of corrosion by Academician Hou [6], the annual economic losses caused by corrosion in China is account for about 3.34% of the GDP. It can be seen that the durability of concrete caused by corrosion of steel bars is a significant problem that should be widely concerned by all countries in the world. In modern engineering construction, concrete will still be one of the largest building materials [7], and the durability of reinforced concrete is the fundamental guarantee to ensure the safe operation of all kinds of projects, which is a common concern and necessary research problem at home and abroad. In this context, it is very important and far-reaching to carry out the research on the measures to improve the durability of reinforced concrete to prolong the service life of reinforced concrete structures and ensure the safe operation of all kinds of engineering facilities.

At present, chloride corrosion is the main cause of steel corrosion [8]. Chloride ion has a small radius and strong permeability. The pH value here is significantly reduced, when chloride ion penetrates the passivation film on the steel bar surface and adsorbs on the steel bar surface [9]. Recent theoretical developments have revealed that [10], when the pH value is less than 11.5, the passivation film will become unstable. When the pH value is less than 9.88, the passivation film starts to be destroyed, and no new passivation film is formed. When the passivation film is damaged, it will no longer play a protective role in the steel bar, and when the O_2 and H_2O

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come into contact with the steel bar, the steel bar will rust [11]. Moreover, the Cl- also has a transport function [12]. Cl^- reacts with Fe^{2+} to form $FeCl_2$, and then $FeCl_2$ dissolves in water to form $Fe(OH)_2$ precipitation when it encounters OH^-. When $Fe(OH)_2$ diffuses into the concrete, it forms iron oxide and rust, leading to steel bars' corrosion. In this process, Cl^- is not consumed, which plays the role of "catalytic transport" and accelerates the corrosion of steel bars exponentially. Currently, many areas are facing a shortage of sand and gravel, some areas have begun to use sand resources. China is rich in sea sand resources, with a reserve of 16,000 tons of sea sand. As we all know, sea sand containing too many chloride ions have strong corrosivity [13-15]. Song Xuyan et al. [16] showed that if sea sand is directly used to prepare concrete, the high chloride ion content of sea sand will accelerate the corrosion of steel bars in concrete. Once untreated seawater is used in concrete buildings, it will inevitably cause the corrosion of steel reinforcement in the concrete and the decline of concrete durability, which will cause the destruction of buildings. 60%~70% of the sand used for concrete production in Guangzhou is desalinated sea sand or sand mixed with river sand [17]. At present, river sand resources in China are becoming scarce lacking, and inappropriate use of sea sand leads to the problem of "sea sand house" [18].

If the content of chloride ions in concrete can be controlled, the problem of steel corrosion can be alleviated, so as to improving the durability of reinforced concrete and prolong the service life of reinforced concrete structures. Therefore, detecting chloride ions in fresh concrete has become an indispensable part of modern engineering [19]. Moreover, China already has the standard stipulation of chlorine ion detection technology in concrete [20]. At present, the commonly used detection methods include silver nitrate titration, chloride ion selective electrode method and paper colorimetry. The method of silver nitrate titration depends on the change of color, which is easy to cause error [21]. Although chlorine ion selective electrode method can accurately detect the content of chlorine ion, it has the disadvantages of poor portability. Barriers for testers are high and time consuming. The use of paper colorimetry will cause visual errors. Therefore, we develop a test bar for the detection of chlorine ions in fresh concrete. Once we directly insert the test bar into the fresh concrete, the degree of discoloration indicates the amount of chloride ion in the concrete with portability, rapidity and accuracy. Furthermore, it provides a simple, accurate and rapid detection method for engineering supervision and detection. Convenient, efficient and rapid chloride ion content detection provides a guarantee of improving the project quality and reducing the project risk, thus stabilizing the quality of engineering concrete products and improving the durability of concrete.

2 Experimental section

2.1 Materials and reagents

Pure potassium chromate (analysis), silver nitrate (analysis), sodium chloride (analysis), cotton fiber, silica gel, water color silicone, poly carboxylic acid water reducing agent (Nanjing TongXin technology co., LTD.), acrylic plastic pipe (TianChang organic products co., LTD.), cement (Portland cement), sea sand (apparent density of 2480 kg/m^3) and limestone rubble (5 ～ 25 mm continuous gradation, apparent density of 2650 kg/m^3) were used in this experiment to preparation the test bars and the samples.

2.2 Instruments and equipment

Electric blast dryer (sbk-101A-3, Nanjing sibeke instrument co., LTD.), electronic balance (wtb-5003, Hangzhou wante co., LTD.), filling mold (Nanjing TongXin technology co., LTD.), mixer (Shanghai QiuZuo instrument co., LTD.) and camera (SAMSUNG-NSmini) were used in this experiment.

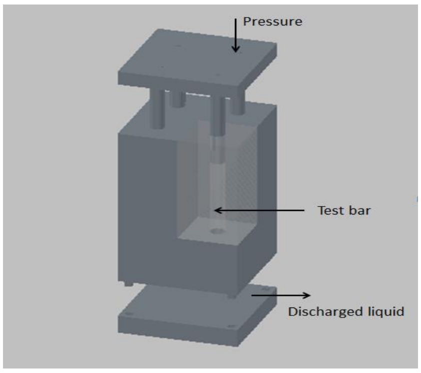

Figure 1 Application method of filling mold

2.3 Experimental principle

Cotton fiber and absorbent silica gel excel in water absorption. They can absorb the pore solution in the newly mixed concrete into the test bar. Under certain conditions, silver chloride precipitation is generated by silver chromate reacting with chloride ions. Within a certain range, the content of chlorine ions is proportional to the height of white precipitation. By comparing the height of rise with a standard height card, the mass concentration of chlorine ions can be determined, therefore, the content of chlorine ion in fresh concrete can be quantitatively detected.

2.4 Experimental methods

2.4.1 Preparation of test bar

First, we prepared silver chromate solution with a mass concentration of 5g/L and put 300 mL (of the solution) into a beaker. Then, the cotton fiber and absorbent silica gel were immersed in the solution for 1 min adding polycarboxylic acid water reducer. Third, the agitator stirred the fiber for 10 minutes at 1000 RPM, in order to make a composite base core of the cotton fiber. After 3 minutes of vibration to remove most of the water, it was placed in a blast drying box and dried at a specific temperature. The color-changing silica gel powder was applied to the upper end of the test bar with the coating amount of 0.5g/root when it was dry. The prepared test bar should be stored in a vacuum bag in a dry and closed place away from light.

2.4.2 Selection of test bar preparation parameters

Portland cement was used to prepare concrete samples in this experiment. By changing the preparation parameters such as the content of cotton fiber, the length of test bar, the drying temperature, etc., test bars with different parameters were prepared and put into the fresh concrete pore solution. The absorption time, color effect and color contrast were then observed to determine the preparation parameters of the test bar.2.4.3. The preparation of corresponding cards

The test bars were prepared at 25℃ and inserted into the concrete pore solution with chloride ion content of 0.005%, 0.01%, 0.02%, 0.03%, 0.04%, 0.05%, 0.06%, 0.07%, 0.08%, 0.09% and 0.10%, respectively. The height of the color-changing part of the test bar was detected after the color change was stabilized, and the relationship card of "color-changing height of the test bar - chloride ion concentration" was prepared according to the experimental results.

2.4.3 Test bar stability test

Temperature stability test

According to the adjusted parameters, the test bars were prepared to test the chloride ion content in C25, C30 and C40 fresh concrete at 5℃, 15℃, 25℃ and 35℃, respectively, to verify the temperature stability.

Reproducibility experiment

The adjusted parameters were divided into 6 batches to prepare the test bars. The test bars were selected from each batch to test the chloride ions in 5 different pore solutions of fresh concrete, to verify the repeatability of the results of multiple measurements.

Coexisting ion effect

Coexisting ions with different contents were added into C30 concrete pore solution with chloride ion concentration of 0.06% and tested with chloride ion test bars. The test results were compared with the standard height to verify the anti-jamming ability of the test bar.

3 Results and discussion

3.1 Influence of preparation parameters on water absorption and color contrast

Test bars at different temperatures were dried to investigate the effect of drying temperature on the color development effect of the test bar. Photos were taken to read RGB values in the color panel by Photoshop, as shown in table 1. The color rendering effect of the test bars dried at different temperatures was different. The color rendering contrast of the low-temperature drying bars was obvious but took a longer time. With the increase of temperature, the drying time of the test bar is shortened. However, the color contrast of the test bar is no longer obviously apparent. Taking color contrast and drying time into consideration, the paper was prepared by blast drying at 50℃.

During the experiment, the content of cotton fiber has a significant influence on the water absorption of the test bar, and the selection of the base core of the test bar should meet the requirements of absorbing pore solution easily and large capacity. As can be seen from Table 2, the adsorption effect differed with the addition of different amounts of cotton fibers. The adsorption rate is defined as the ratio of the density after water absorption to the density before water absorption. When the cotton fiber incorporation amount is small, it is easier to absorb the pore solution, but the total adsorption amount is small. When the content of cotton fiber is about 1.5g/cm^3, the adsorption of the pore solution is relatively easy, and the total adsorption capacity is significantly increased. When the content of cotton fiber is increased gradually, the total adsorption is increased slightly, but the adsorption of pore solution became more difficult. Considering the factors such as the difficulty of absorbing pore solution and the total adsorption capacity, the cotton fiber content was 1.5g/cm^3.

Table 1 Influence of drying temperature on color rendering effect of test bar

Dry conditions	Drying time	Pre-reaction color	Post-reaction color
30℃Drum wind drying	8h	🟫	⬜
40℃Drum wind drying	6.5h	🟫	🟧
50℃Drum wind drying	5h	🟫	🟧
60℃Drum wind drying	3.5h	🟥	🟧
70℃Drum wind drying	2h	🟫	🟫

Table 2 Influence of cotton fiber content on water absorption of test bar

Cotton fibre content	Water absorption and difficulty	The total water quantity
0.9g/cm^3	2.25 easy	2.03g/cm^3
1.2g/cm^3	2.03 easy	2.43g/cm^3
.5g/cm^3	1.95 normal	2.92g/cm^3
1.8g/cm^3	1.83 difficult	3.22g/cm^3
2.1g/cm	1.65 hard	3.30g/cm^3

During the use of the test bar, the length of the test bar has a significant influence on the measuring range of the test bar and the adsorption time of pore solution. The selection of the length of the test bar should meet the requirements of a shorter adsorption time of pore solution and wider measurement range. Twelve kinds of test bars in different lengths were prepared, and 4 rods of each type were selected for the adsorption time test and the results were averaged. With the increase of the length of the test bar, the detection ranges of chlorine ions in the fresh concrete increases, but the time to fully absorb the pore solution also increases gradually. It can be seen from table 3 and Figure 2that the adsorption time of the test bar for pore solution increases with the augment of the length of the test bar and the increment of time tardily. The specification requires that the maximum chloride ion content in concrete is 0.06%. As can be seen from Figure 3, when the length of the test bar is less than 40mm, the adsorption time of pore solution is shorter, but the measurement range is smaller. When the length of the test bar is more than 55 mm, the measurement range continues to increase, but the adsorption time of the pore solution is increased significantly. Combined with the approximate range of chlorine ion content in fresh concrete and the actual construction detection time, the length of the test bar should be selected as 50 mm. As shown in Figure 4, test bars were inserted into freshly mixed concrete, and every 3-test bar was a group. After completion of detection, the test bars were removed and the average value of the three test bars was recorded.

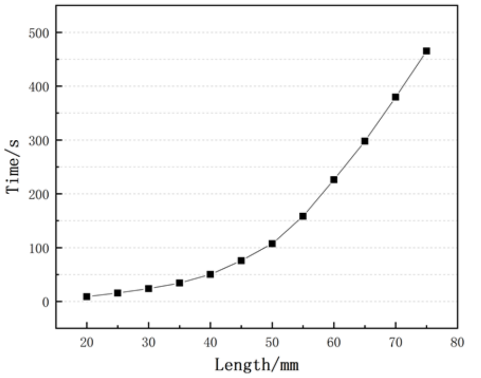

Figure 2 Relationship between the length of test bar and the service time of test bar

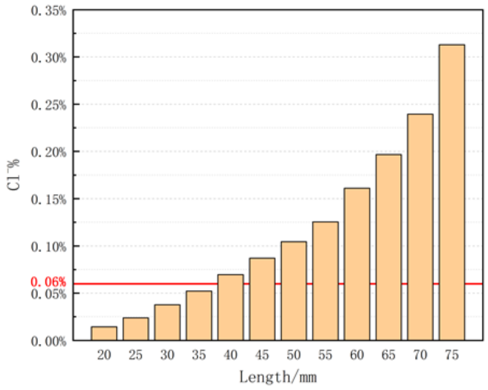

Figure 3 Relationship between the length of test bar and the range of test bar

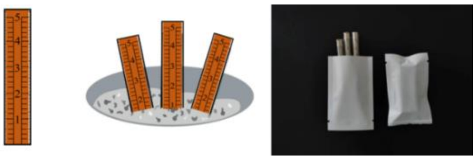

Figure 4 Test method for test bar in freshly mixed concrete

Table 3 Influence of length on the time and range of test bar

Length /mm	Time/s	Maximum range (percentage chloride weight)
20	8.91	0.0145%
25	15.73	0.0239%
30	23.78	0.0377%
35	34.18	0.0522%
40	50.09	0.0696%
45	75.78	0.0871%
50	107.35	0.1045%
55	158.23	0.1255%
60	226.18	0.1611%
65	297.65	0.1967%
70	379.47	0.2395%
75	465.26	0.3129%

3.2 Relationship between the color change height of test bar and the content of chlorine ion

Under the same condition, the test bars with adjusted parameters were placed into the concrete pore solution with different design concentrations in turn. The color change height of each test bar was recorded after 120 s of absorption. According to different chloride ion concentration and corresponding color rendering height of the test bar, the "color rendering height - chloride ion content" curve was fitted, as shown in Figure 5, Figure 6 and equations (1), (2). With the increase of chloride ion content, the color rendering height of the test bar also increased significantly. As shown in Figure 7, the corresponding card of "color development height - chlorine ion content" was drawn according to the index value of test bar, so as to realize the semi-quantitative and rapid detection of chlorine ion content in the pore solution of fresh concrete.

Table 4 Different chloride ion concentration and corresponding color developing height of test bar

Cl^-%	Length/mm
0	0
0.005	10
0.01	17
0.02	23
0.03	27
0.04	31

Cl^{-}%	Length/mm
0.05	34
0.06	37
0.07	40
0.08	43
0.09	46
0.1	49

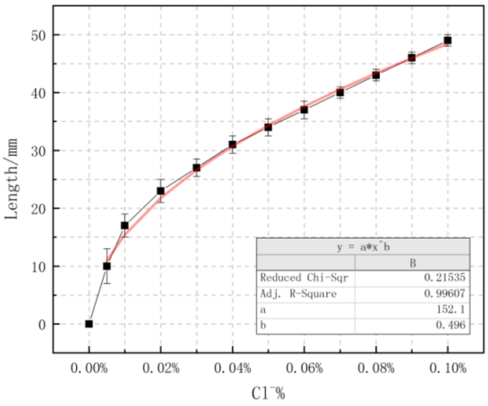

Figure 5 Fitting curves of different chloride ion concentrations and corresponding chromogenic heights

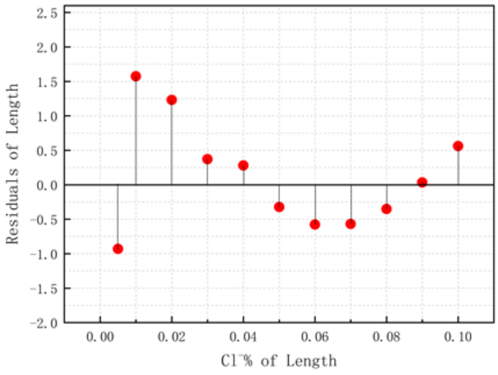

Figure 6 Residuals of color rendering length

$$Y = 152.1X^{0.496} \tag{1}$$

$$X = \sqrt[0.496]{\left(\frac{Y}{152.1}\right)} \tag{2}$$

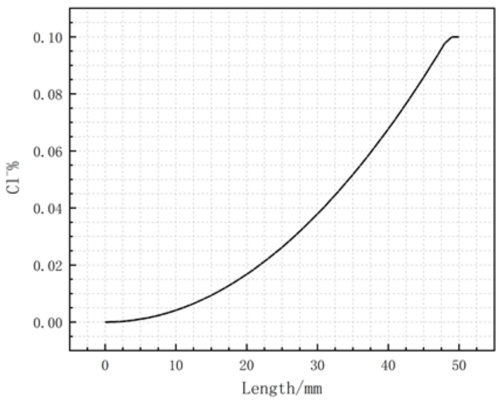

Figure 7 "Color development height - chlorine ion content" corresponding curve

Table 5 "Color development height - chlorine ion content" corresponding card

Length/mm	Cl^{-}%	Length/mm	Cl^{-}%	Length/mm	Cl^{-}%
0	0.0000	18	0.0135	35	0.0517
2	0.0002	19	0.0151	36	0.0547
3	0.0004	20	0.0167	37	0.0578
4	0.0007	21	0.0185	38	0.0610
5	0.0010	22	0.0203	39	0.0643
6	0.0015	23	0.0222	40	0.0677
7	0.0020	24	0.0242	41	0.0711
8	0.0026	25	0.0262	42	0.0747
9	0.0033	26	0.0284	43	0.0783
10	0.0041	27	0.0306	44	0.0820
11	0.0050	28	0.0330	45	0.0858
12	0.0060	29	0.0354	46	0.0897
13	0.0070	30	0.0379	47	0.0937
14	0.0082	31	0.0405	48	0.0978
15	0.0094	32	0.0432	49	0.1000
16	0.0107	33	0.0459	50	0.1000
17	0.0121	34	0.0488		

3.3 Stability analysis

3.3.1 Temperature stability

The content of chloride ion in C25, C30 and C40 fresh concrete was measured with test bars in the case of changing the ambient temperature. As can be seen from Table 6, the test of C25, C30, and C40 fresh concrete at 5℃, 15℃, 35℃ was carried out by the test bar and the deviation of the measured value at 25℃ was within ± 10% at different temperatures, which indicated this method was not sensitive to temperature and could be applied to a variety of temperature environments. The indicator number of the test bar did not change significantly with the increase of temperature, which was similar to that of the potentiometric titrator at different temperatures. This may be due to the fact that the reaction process of silver chromate combined with chloride ions in the indicator material to generate silver chloride was not susceptible to environment temperature. Moreover, temperature compensation can be provided at lower ambient temperatures due to the heat released during cement hydration. When testing bars used in a higher temperature environment, moisture of the fresh concrete evaporated faster with the increase of ambient temperature, consequently the concentration of the sodium chloride in concrete increased slightly. Likewise, the absorption volume of the test bars was unchanged and the concentration of chloride ion increased, resulting in the index of the test bars slightly higher than normal temperature environment (25℃). The evaporation of concrete moisture was negligible at relatively high temperatures due to the short time used by the test bars, thus the influence of temperature on the measurement results could be ignored. Therefore, the change of environmental temperature had little interference to the test bars in engineering applications. the evaporation of moisture could be negligible in a short time at room temperature, while it was necessary to control the mixing time of concrete to suppress the evaporation in a high temperature environment.

3.3.2 Reproducibility

Under the same environment, test bars made in six different batches were put into five kinds of fresh concrete. The data results and deviations are shown in table 7. When the chloride ion concentration is small, the variance of data is small, but the gap between each batch is still large, so the error in measuring the mixed concrete with low chloride ion concentration is large. It can be seen from table 7 that for the same sample, although some data deviate greatly, the test results are all very close. As an example, the difference between the test results of sample No. 2 with a maximum deviation of 0.00376 is only 0.0083% of the chloride ion content, indicating that the difference in results is not significant. At the same time, the reserved number of the decimal point also greatly influences the calculation of the deviation. For example, if the last two digits of a small number are accurate to the percentage, the two data records will become 0.02% and 0.02%, and the deviation will be 0.

3.3.3 Influence of coexisting ions

It can be seen from Table 5.6 that with the increase of the content of SO_4^{2-}, Mg^{2+} and CO_3^{2-} ions commonly found in concrete in fresh concrete, the number of test bars is relatively stable without major fluctuations. Therefore, SO_4^{2-}, and CO_3^{2-} ions do not affect the test results of rapid test bars. The principle of color development of the indicator material is the combination of silver chromate and chloride ions to form silver chloride. Under the same temperature environment, the solubility product of silver chromate is less than that of silver sulfate, silver carbonate and other substances, and is greater than that of silver chloride. Therefore, in the complex ion environment of the concrete itself, the indicating material in the detection test rod can "avoid" the carbonate sulfate radical plasma to preferentially react with the chloride ion to complete the detection of the chloride ion in the fresh concrete. When the traditional silver nitrate titration method is used to detect the chloride ion in concrete, the sulfate and carbonate ions contained in the method need to be removed in advance. The process is complex and the anti-interference ability is weak. Compared with the traditional method, the rapid detection test bar method has good anti-interference performance in concrete. The addition of iodine ion in fresh concrete will greatly affect the indicator of test rod. With the increase of iodine ion content in fresh concrete, the indicator of test rod will also increase significantly. This is because the indicator material is more likely to bind iodine ions and can only bind chloride ions in the solution of the inhalation hole, thus, the rapid detection test rod is not suitable for detection under the condition of containing halogen group element ions. In summary, since the halogen group element ion content is less in the concrete, the rapid detection test bar has a good resistance to ion interference.

4 Conclusion

A novel method based on concrete test bar was established to detect the concentration of chlorine ion swiftly and optimized its conditions of preparation process and formula. The influence of the preparation parameters on the test bar demonstrated that the test bars exhibited excellent performance under the best conditions (5.0 g/L silver chromate, soaking time 1 min, drying temperature 50℃). The test bar had the characteristics of small size, sensitive response, low cost, easy operation, and high stability. Moreover, the test bar could realize the rapid detection of semi-quantitative detection compared with the traditional method of silver nitrate titration. In general, it had a wide range of applications, great potential and development prospects in the construction industry.

Table 6 Test results of test bars at different ambient temperatures %

Strength grade	25℃	35℃		15℃		5℃	
	Measured value	Measured value	Deviation	Measured value	Deviation	Measured value	Deviation
C25	0.0148	0.013794	-6.8	0.015599	5.4	0.015792	6.7
C30	0.0254	0.026187	3.1	0.026543	4.5	0.027356	7.7
C40	0.0185	0.017168	-7.2	0.020147	8.9	0.019888	7.5

Table 7 Test results of different batches of test rods in concrete

Concrete number	First batch /%	Second batch /%	Third batch /%	Fourth batch /%	Fifth batch /%	Sixth batch /%	The variance
1#	0.0009	0.0014	0.0026	0.0007	0.0015	0.0033	0.00101
2#	0.0165	0.0212	0.0173	0.0239	0.0244	0.0161	0.00376
3#	0.0356	0.0335	0.0413	0.0359	0.0387	0.0364	0.00272
4#	0.0613	0.0635	0.0672	0.0624	0.0677	0.0618	0.00279
5#	0.0821	0.0867	0.0842	0.0821	0.0852	0.0841	0.00179

Table 8 Results of cross reaction of test bar to other ions

SO_4^{2-}	0.003%	0.006%	0.01%	0.02%	0.03%	0.04%
Color rendering height/mm	38	38	38	38	39	38
Mg^{2+}	0.003%	0.006%	0.01%	0.02%	0.03%	0.04%
Color rendering height/mm	38	38	38	38	39	39
I^-	0.003%	0.006%	0.01%	0.02%	0.03%	0.04%
Color rendering height/mm	39	39	41	44	46	49
CO_3^{2-}	0.003%	0.006%	0.01%	0.02%	0.03%	0.04%
Color rendering height/mm	38	38	38	38	38	38
Br^-	0.003%	0.006%	0.01%	0.02%	0.03%	0.04%
Color rendering height/mm	38	39	40	43	46	49

Conflict of Interest: The authors declare that there is no conflict of interest regarding the publication of this paper.

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