Application solution of chloride in concrete admixtures by ion chromatography

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Foreword

Chloride ion is a harmful component in cement and cement raw materials. It has a direct impact on preheater and kiln calcination in new dry process cement production, resulting in accidents such as ring formation and plugging, affecting equipment operation rate and cement clinker quality. At the same time, when the chloride ion content in cement exceeds a certain value, it will corrode the steel bar in concrete,



reduce the strength of the steel bar, can also cause concrete damage caused by expansion, and when serious, it will cause concrete cracking and bury hidden dangers to the quality of the project, so it must be strictly controlled. The requirement for chloride ion limitation is added in the article 7.1 of GB 175-2007 *Common portland cement*. The requirement is that chloride content in cement is not greater than 0.06%. Ammonium thiocyanate volumetric method, potentiometric titration method and ion chromatography method are commonly used for the determination of chloride ions. However, because the stability of silver chloride is not good, the structure of silver (chlorine) electrode is unstable, and the environmental impact is greater, they result in poor repeatability and suitable for the detection of substances with high chloride content. Ion chromatography, as the preferred method for the detection of ionic substances, can be used to analyze multiple ions simultaneously with one injection, and has the characteristics of rapid and accurate. In this paper, ion chromatography is used to analyze and test concrete additives and chloride ion in cement.

Standard introduction

The National Standard GB8076-2008 "Concrete Admixture" stipulates the detection method of chloride ion in eight types of concrete admixtures: high performance water reducer (early strength, standard, retarding type), high efficiency water reducer (standard, retarding type), common water reducer (early strength, standard, retarding type), air-entraining water reducer, pumping admixture, early strength agent, retarder and air-entraining agent. Method for detecting chloride ion in agent. Compared with GB8076-1997, this standard deletes the test method of steel corrosion in the original standard and establishes the determination method of chloride ion content in concrete admixtures by ion chromatography. GB8076-2008 stipulates that chloride ions shall be detected in accordance with Appendix B (Ion Chromatography) or in accordance with GB 8077-2012, but Ion Chromatography is the

arbitration method.

Table 2 Uniformity index

Item	Index
Chloride ion content/%	No more than the control value of the plant
Total amount of alkali/%	No more than the control value of the plant
Solid content/%	S>25%, Should be controlled at 0.95S~1.05S;
	S≤25%, Should be controlled at 0.90S~1.10S;
Rate of water content/%	W>5%,Should be controlled at 0.90W~1.10W;
	W≤5%,Should be controlled at 0.80W~1.20W;
Density/(g/cm ³)	D>1.1,Should be controlled at D±0.03;
	D≤1.1,Should be controlled at D±0.02;
Fineness	It should be within the control range of the plant.
pH value	It should be within the control range of the plant.
Sodium sulfate content/%	No more than the control value of the plant

Note1:The manufacturer should clearly indicate the control value of product uniformity index in relevant technical data.

Note2:Other requirements for uniformity and equivalence between the same and different batches can be agreed by the supplier and the buyer.

Note3:The S,W and D in the table are the control values of solid content, rate of water content and density respectively.

Pretreatment

Weigh 1g admixture sample accurately(accurate to 0.1mg). Place it into 100mL beaker, and dissolve the sample with 50mL water and 5 drops of nitric acid. When the sample can be dissolved by water, it can be directly transferred to 100mL volumetric flask and diluted to scale; When the sample can not be dissolved by water, the sample is dissolved by ultrasonic and heating method, then filtered by rapid filter paper, the filtrate is palace into 100mL volumetric bottle, and diluted to the scale with water.

Remove the organic matter from samples and the soluble organic matter in concrete admixture can be removed by RP pretreatment column.

Chromatographic condition

• IC Type: CIC-D120

• Analysis column: SH-AC-4 (OrSH-AC-1 and other chromatographic columns with similar properties)

• Eluent: 2.4 mM Sodium Carbonate/6.0mM Sodium Bicarbonate



• Flow rate: 1.0 mL/min

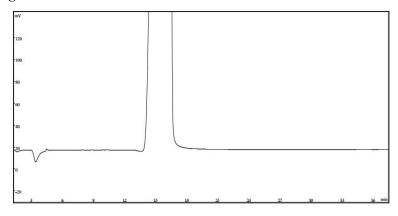
• Detection method: Suppressed conductivity detection

Standard curve drawing

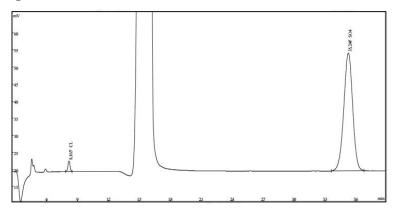
Blank tests were carried out under the above chromatographic conditions. A series of chloride ion standard solutions were separated by ion chromatograph to obtain chromatogram. The peak area or peak height of chromatographic peaks were determined. The standard curve was drawn with chloride ion concentration as abscissa and peak area or peak height as ordinate.

Application case

1.Blank test chromatogram



2. Sample test chromatogram



Conclusion

Ion chromatography can be used to analyze chloride ions in concrete admixtures accurately and quickly. Because of the advantages of ion chromatography, the content of sulfate radical can also be determined simultaneously. Apart from concrete additives, ion chromatography can also be used to analyze and determine conventional anions in cement and concrete water.

