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# Determination of Impurity Fluorine Ion in Water Soluble Chemicals – Ion Chromatography Method

水溶性化工产品中杂质氟离子的测定 离子色谱法

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**Standardization Administration of PRC.**

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# Determination of Impurity Fluorine Ion in Water Soluble Chemicals – Ion Chromatography Method

## 1 Scope

This Standard specifies the ion chromatographic method for the determination of impurity fluoride ion ($F^-$) content in the water-soluble chemicals.

This Standard is applicable to the determination of fluoride ion content in water-soluble chemicals with fluoride ion content ≤400mg/kg.

In this Standard, the quantitative limit of organic chemicals is 0.02mg/kg; while the quantitative limit of inorganic chemicals is 4.0mg/kg.

This Standard is not applicable to the insoluble chemicals and the chemicals with main component containing fluorine.

## 2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

GB/T 6682 Water for Laboratory Use – Specifications

## 3 Principle

After treatment, the sample forms an aqueous solution; enters into the ion exchange chromatographic column with ion exchange resin as a stationary phase; the ion exchange resin is distributed by fixed charged group and free coordination ion; when eluted with a suitable eluent, the active exchange group on the ion exchange resin undergoes the ion exchange with ions in the test solution and ions in the eluent. As the eluent continues to flow, the anion in the test solution and the exchange group continue to occur exchanging – eluting – re-exchanging – re-eluting; depending on the different retention characteristics of the test anion on the chromatography column, the separation is realized; finally, the test anion is carried by the eluent to the detector and form a chromatographic peak. The group ion is qualitative by the retention time of each

scale; perform ultrasound for 10min; stand for 60min; filter the supernatant by the filter membrane for testing.

Organic chemicals: pipette 50mL of specimen into 100mL beaker; add 150μL of sodium hydroxide (4.2) to alkalinize; then evaporate to near dryness in water bath; dilute the slag with pure water; make constant volume into 10mL volumetric flask; perform ultrasound for 10min; stand for 60min; filter the supernatant by the filter membrane for testing.

Alkali chemicals: accurately take 1.0g~3.0g (accurate to 0.001g) of specimen into 50mL beaker; dissolve into water; then transfer to 100mL volumetric flask; dilute to the scale; perform the ultrasound for 10min; stand for 60min; take the supernatant to pass the hydrogen-type cation exchange cartridge; collect the filtrate to filter by the filtered membrane for testing.

**6.2 Ion chromatographic conditions**

a) Mobile phase: select according to the separation column, so that meet the requirements of separation in 5.5; meanwhile the main component can be eluted without disturbing the subsequent separation. For example, 20mmol/L sodium hydroxide solution (4.1).

b) Flowrate: 1.0mL/min.

c) Column temperature: 30°C.

d) Sample injection volume: 25μL.

e) Detector: conductivity detector.

**6.3 Standard working curve**

Preparation of standard working solution: take 10mL of fluoride ion standard stock solution (4.3) into a 100mL volumetric flask; dilute with water to the scale; then prepare the 10μg/mL fluoride ion standard solution. Separately take 0.4mL, 0.8mL, 1.0mL, 2.0mL, 4.0mL, 5.0mL, 20.0mL, 50.0mL of fluoride ion standard solution into 100mL volumetric flasks; dilute with water to the scale; then prepare the standard working solution with fluoride ion concentration of 0.04μg/mL, 0.08μg/mL, 0.1μg/mL, 0.2μg/mL, 0.4μg/mL, 0.5μg/mL, 2.0μg/mL, 5.0μg/mL; such solution shall be prepared for current use.

Take the above standard working solutions from the low to high concentrations into the ion chromatograph; draw a standard curve with ion concentration as abscissa and peak area as the ordinate.

**6.4 Sample determination**

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