

**INDUSTRY STANDARD OF THE  
PEOPLE'S REPUBLIC OF CHINA**

**HJ 823-2017**

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**Water quality-Determination of cyanide - Flow injection  
analysis(FIA) and spectrophotometric method**

水质 氰化物的测定 流动注射-分光光度法

**(English Translation)**

**Issued on: March 30, 2017**

**Implemented on: May 1, 2017**

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**Issued by: Ministry of Ecology and Environment**

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## Foreword

In order to implement the Environmental Protection Law of the People's Republic of China and the Law of the People's Republic of China on the Prevention and Control of Water Pollution, Protection of human health, regulate the determination of cyanide in water method, the development of this standard.

This standard specifies the flow injection-spectrophotometric method for the determination of cyanide in surface water, groundwater, domestic sewage and industrial waste water.

This standard is the first release.

This standard is organized by the Environmental Monitoring Department of the Ministry of Environmental Protection, the Secretary for Science and Technology Standards.

The main drafting unit of this standard. Jiangsu Province Environmental Monitoring Center.

The standard verification unit. Beijing Environmental Protection Monitoring Center, Wuxi City Environmental Monitoring Center Station, Huai'an City Environmental Monitoring Central Station, Yangzhou City Environmental Monitoring Center Station, Changshu City Environmental Monitoring Station and Xuzhou City Environmental Monitoring Center

Station.

The environmental protection department of this standard approved on March 30, 2017.

This standard has been implemented since May 1, 2017.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of cyanide - Flow injection - spectrophotometric method

Warning. cyanide and pyridine are highly toxic substances, the operation should be required to wear protective equipment, to avoid contact with the skin and clothes, after the detection of residue should be properly safe handling.

## 1 Scope of application

This standard specifies the flow injection-spectrophotometric method for the determination of cyanide in water.

This standard is applicable to the determination of cyanide in surface water, groundwater, domestic sewage and industrial waste water.

When the optical path length was 10 mm, the detection limit of cyanide in water was 0.001 mg/L by isonicotinic acid-barbituric acid method.

And the detection limit of cyanide in water was 0.002 mg/L, and the detection limit was 0.002 mg/L, the assay range is 0.008 mg/L 0.50 mg/L.

## 2 Normative reference documents

The contents of this standard refer to the following or their terms. For undated references, the valid version applies to this standard.

Determination of water quality cyanide and its spectrophotometric method Technical specification for surface water and wastewater monitoring Technical specification for groundwater environmental monitoring

## 3 Terms and definitions

The following terms and definitions apply to this standard.

Total cyanide total cyanide in the presence of phosphoric acid and EDTA in the presence of phosphoric acid and EDTA, the cyanide of cyanide is heated and distilled to form cyanide, including all simple Cyanide (mostly alkali metal and alkaline earth metal cyanide, ammonium cyanide) and the vast majority of complex cyanide (zinc cyanide complex Iron cyanide complex, nickel cyanide complex, copper cyanide complex, etc.), excluding cobalt cyanide complex.

### 3.2 Easy to release cyanide easily liberatable cyanide

In the presence of zinc nitrate in the presence of zinc nitrate in the presence of zinc nitrate, the cyanide of cyanide is heated and distilled to form cyanide, including all simple cyanide (Mostly cyanide of alkali and alkaline earth metals) and zinc cyanide complexes, excluding ferricyanide complexes, ferrocyanide complexes, copper Cyanide complex, nickel cyanide complex, cobalt cyanide complex.

### 4.1 Flow injection device works

In a closed line, a volume of the sample into the continuous flow of the carrier liquid, the sample and reagents in the chemical reaction mode the blocks are mixed and reacted in a specific order and proportion, and are subjected to photometric detection under a non-complete reaction condition into the flow detection cell.

#### 4.2.1 Isonicotinic acid-barbituric acid method

Under acidic conditions, the samples were hydrolyzed by high temperature and high pressure at 140 °C and UV digestion. The released hydrogen cyanide gas was oxidized by hydrogen Sodium solution absorption. The cyanide in the absorbing solution reacts with chloramine T to form cyanogen chloride, which is then hydrolyzed with isonicotinic acid to produce and then with barbituric acid to produce blue-violet compounds, measured at 600 nm wavelength absorbance. Specific workflow See Figure 1.

#### 4.2.2 Pyridine-barbituric acid method

Under acidic conditions, the samples were hydrolyzed by high temperature and high pressure at 140 °C and UV digestion. The released hydrogen cyanide gas was oxidized by hydrogen Sodium solution absorption. Under neutral conditions, the cyanide in the absorbing solution reacts with chloramine T to form cyanogen chloride, which reacts with pyridine the formation of glutaraldehyde, and finally with barbituric acid to produce condensed reddish purple compounds, measured at 570 nm wavelength absorbance. With Body work flow shown in Figure 1.

1. Peristaltic pump; Heating pool (140 °C); UV digestion device; Diffusion pool; Injection valve; Reaction ring; Heating tank (60 °C) 8. Detection cell 10mm, 600nm or 570nm; R1 Phosphoric acid solution (6.17); Sodium hydroxide solution (6.19); R3. Phosphate buffer Liquid (6.24); R4 Chloramine-T solution (6.25 or 6.26); R5. Pyridine-barbituric acid solution (6.27) or isonicotinic acid-barbituric acid solution (6.28) C. Carrier solution (sodium hydroxide solution, 6.19); Sample; Waste liquid

Figure 1 flow injection - spectrophotometric determination of cyanide reference work flow chart

## 5 Interference and elimination

The presence of active chlorine and other oxidizing substances in the sample interference determination, can be added before the distillation of sodium sulfite ( $\text{Na}_2\text{SO}_3$ ) solution in addition to interference; the presence of nitrite ion interference in the sample, can be used before the distillation of sulfamic acid ( $\text{NH}_2\text{SO}_3\text{H}$ ) to eliminate interference;

Sulfide interference is present in the sample, and carbonate ( $\text{CdCO}_3$ ) or lead carbonate ( $\text{PbCO}_3$ ) solid powder can be added before distillation Eliminate interference; Thiocyanate produces less than 1% of positive interference.

## 6 Reagents and materials

Unless otherwise stated, analytical reagents conforming to national standards are used for analysis. The experimental water is freshly prepared Ionized water or distilled water. In addition to the standard solution, other solutions and experimental water are treated with helium (6.35) or ultrasonic (7.3) degassed.

Unless otherwise stated, analytical reagents conforming to national standards are used in the analysis, and the test water is a newly prepared deionized Water or distilled water. Experimental reagents and water are required to use helium or ultrasonic degassing, the specific method. the use of 140KPa helium Over the helium air tube 1min degassing, or the use of ultrasonic oscillation 15min ~ 30min degassing.

### 6.1

Phosphoric acid.  $\rho(\text{H}_3\text{PO}_4) = 1.69 \text{ g/ml}$ .

### 6.2

Hydrochloric acid.  $\rho(\text{HCl}) = 1.19 \text{ g/ml}$ .

### 6.3

Sodium hydroxide ( $\text{NaOH}$ ). excellent grade pure.

### 6.4

Potassium hydroxide ( $\text{KOH}$ ). excellent grade pure.

### 6.5

Sodium chloride ( $\text{NaCl}$ ). Reference level.

Dry at  $600^\circ\text{C}$  for 1h, cooler in the dryer, stand-by.

### 6.6

Silver nitrate ( $\text{AgNO}_3$ ).