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Detection of trace cyanide in water by isonicotinic acid – pyrazolone – bispyrazolone polarography

© 2024. X. H. Li[1] ORCID: 0000-0002-3435-6852,
Y. Chen[1, 2] ORCID: 0009-0006-9250-3159,
[1]Liaoning Institute of Science and Technology,
176, Xianghuai Rd., Benxi, Liaoning, P. R. China, 117004,
[2]Vyatka State University,
36, Moskovskaya St., Kirov, Russia, 610000
e-mail: 184203287@qq.com

To enhance the detection sensitivity of cyanide, a method was proposed for the determination of trace cyanide in water by using isonicotinic acid – pyrazolone – bispyrazolone polarography. Under optimized experimental conditions, cyanide reacted with chloramine T, isonicotinic acid, and pyrazolone, and bispyrazolone to form a stable blue dye, which produced a sensitive polarographic wave at a peak potential of -766 mV (vs. Ag/AgCl) on the dropping mercury electrode. The results showed that there was a good linear relationship between the cyanide concentration and the second-order derivative peak current (i_p) in the range of 0.32–60 μg·L^{-1}, with an impressive detection limit of 0.08 μg·L^{-1}. Bispyrazolone could effectively inhibit the decomposition of the blue dye and increase i_p by 12%. The polarographic wave originated from the two-electron reduction of the carbonyl group on the pyrazolinone moiety of the blue dye, exhibiting an irreversible adsorptive characteristic. The method was successfully applied to the determination of cyanide in drinking water, groundwater, and surface water, with relative standard deviations of less than 4.5% and recovery rates ranging from 96.7 to 107%. The method significantly lowered the detection limit of cyanide and exhibited high precision and accuracy, making it suitable for the analysis of trace cyanide in water.

Keywords: cyanides, polarography, isonicotinic acid, pyrazolone, bispyrazolone.

УДК 543.552+543.31

Определение следов цианида в воде методом полярографии изоникотиновой кислоты – пиразолона – биспиразолона

© 2024. Сяохуэй Ли[1], доктор наук, доцент,
Я Чен[1, 2], студент,
[1]Ляонинский институт науки и технологии,
117004, КНР, провинция Ляонин, г. Бэньси, Сянхуай Роуд, д. 176,
[2]Вятский государственный университет,
610000, Россия, г. Киров, ул. Московская, д. 36,
e-mail: 184203287@qq.com

Для повышения чувствительности обнаружения цианида был предложен метод определения следов цианида в воде с помощью полярографии изоникотиновой кислоты – пиразолона – биспиразолона. В оптимизированных условиях эксперимента цианид реагировал с хлорамином Т, изоникотиновой кислотой, пиразолоном и биспиразолоном с образованием стабильного синего красителя, который генерировал чувствительную полярографическую волну с пиковым потенциалом -766 мВ (против Ag/AgCl) на ртутном капельном электроде. Результаты показали, что существует хорошая линейная зависимость между концентрацией цианида и пиковым током производной второго порядка (i_p'') в диапазоне 0,32–60 мкг/л, с пределом обнаружения 0,08 мкг/л. Биспиразолон эффективно ингибирует разложение синего красителя и увеличивает i_p'' на 12%. Полярографическая волна возникала в результате двухэлектронного восстановления карбонильной группы на пиразолиноновом мотиве синего красителя, проявляя необратимую адсорбционную характеристику. Метод был успешно применён для определения цианида в питьевой воде, грунтовых и поверхностных водах с относительными стандартными отклонениями менее 4,5% и коэффициентами восстановления от 96,7 до 107%. Метод продемонстрировал высокую точность и достоверность, значительно снизил предел обнаружения цианида, что делает его пригодным для анализа следов цианида в воде.

Ключевые слова: цианиды, полярография, изоникотиновая кислота, пиразолон, биспиразолон.

The majority of cyanides enter the environment with industrial effluents. Cyanides are fastest-acting and highly toxic substances and even their trace amounts pose potential hazards to humans and ecosystems, making them one of the priority pollutants for water quality monitoring [1]. Environment al cyanides may be water-soluble inorganic salts (NaCN or KCN) or unbound substances (CN^- or HCN); the latter being the most toxic [2].

Therefore, accurately determining trace cyanides in environmental water is of great significance for environmental protection and human health. Currently, the primary methods for detecting cyanides in water include spectrophotometry [3], ion chromatography [4], flow injection analysis [5], polarography [6], among others. Among these, polarography stands out in the field of environmental monitoring due to its simplicity of operation, rapid analysis, high sensitivity, and low equipment cost.

The isonicotinic acid – pyrazolone spectrophotometry based on the Konig reaction [7] is one of the primary methods for analyzing cyanides. However, its detection limit of 0.016 mg·L^{-1} [8] falls short of meeting the requirements for detecting trace cyanides in clean water sources such as drinking water or groundwater. Research has shown that replacing spectroscopic analysis with polarographic analysis can significantly enhance the sensitivity, thereby lowering the detection limit [9]. Guo et al. successfully applied the isonicotinic acid – pyrazolone polarography to analyze trace cyanides in food and drinking water, however, they did not delve into the analysis mechanism [10]. Moreover, the reaction products of this system are unstable, leading to poor precision. Building upon this, our experiment explored the method for determining trace cyanide in water using the isonicotinic acid – pyrazolone – bispyrazolone polarography.

Objects and methods of research

Instruments and reagents. JP–303 polarograph with a three-electrode system consisting of a dropping mercury electrode (or a hanging mercury drop electrode for cyclic voltammetry (CV) measurement), a platinum wire auxiliary electrode, and a Ag/AgCl reference electrode. Standard material for cyanogen analysis in water: 50.0 mg·L^{-1} (GBW(E)080115). Cyanide standard solution: 1.00 mg·L^{-1} and 0.100 mg·L^{-1}, prepared by diluting the standard material with 0.1% (mass fraction, the same below) NaOH solution. Chloramine T solution: 5.0 g·L^{-1}, prepared by dissolving 0.50 g of chloramine T in 100 mL water. Bispyrazolone solution: 1.0 g·L^{-1}, prepared by dissolving 0.10 g of bispyrazolone in 100 mL of N,N-dimethylformamide. Pyrazolone solution (12.5 g·L^{-1}): 1.25 g of pyrazolone dissolved in 100 mL of N,N-dimethylformamide. Isonicotinic acid solution (15.0 g·L^{-1}), 1.50 g of isonicotinic acid dissolved in 25 mL of 2.0% NaOH solution and diluted with water to 100 mL. Bispyrazolone – pyrazolone – isonicotinic acid solution: bispyrazolone solution, pyrazolone solution, and isonicotinic acid solution mixed in a volume ratio of 1:2:10. Phosphate buffer solution: 34.0 g of KH_2PO_4 and 35.5 g of Na_2HPO_4 dissolved in 1000 mL of water.

Bispyrazolone (bis(3-methyl-1-phenyl-5-pyrazolone)) (98%); pyrazolone (1-phenyl-3-methyl-5-pyrazolone) (99%); sodium hydroxide (GR); other reagents are analytical – reagent grade. The experimental water is ultrapure water (resistivity ≥ 18.2 MΩ·cm).

Experimental Method. The test samples were pre-treated according to the distillation method and interference elimination method described in HJ 484–2009 [8]. When the research object is cyanide standard solution, pretreatment is not required. Take an appropriate amount of cyanide standard solution or pretreated test sample into a 25 mL colorimetric tube, and dilute it to 10 mL with 0.1% NaOH solution. Subsequently, add 5.0 mL phosphate buffer solution and 0.2 mL chloramine T solution in sequence, mix immediately, and allow to stand for 3–5 min. After that, add 2.6 mL bispyrazolone – pyrazolone – isonicotinic acid solution, dilute the solution to 25 mL, and mix thoroughly. Next, place the colorimetric tube in a water bath at 35 °C for a reaction time of 60 min, then remove and let it stand at room temperature for another 10 min. This is the test solution to be analyzed. Transfer a portion of the test solution into a 10 mL beaker and analyze it using a polarograph. Set the initial potential to -500 mV, the scan rate to -700 mV/s, and record the second-order derivative peak current (i_p'') at the peak potential of -766 mV (Fig. 1).

Reaction Mechanism. Under neutral conditions, cyanides in water react with the hydrolysis product (HClO) of chloramine T to generate CNCl, which then reacts with isonicotinic acid and undergoes hydrolysis to form 3-carboxypentenedialdehyde. Subsequently, 3-carboxypentenedialdehyde then undergoes condensation reaction with pyrazolone to generate a blue dye. Bispyrazolone is used to eliminate residual HClO

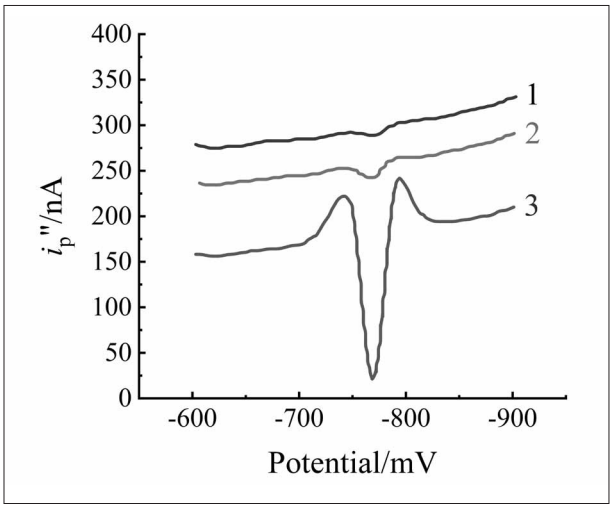

Fig. 1. Polarographic wave: 1 – reagent blank; 2 – $\rho(CN^-)$ = 0.32 μg·L^{-1}; 3 – $\rho(CN^-)$ = 10.0 μg·L^{-1}

and stabilize the blue dye. The i_p'' value of the blue dye at -766 mV (vs. Ag/AgCl) is measured by a polarograph.

Results and discussion

Determination of Analytical Conditions. The experimental conditions were investigated using a cyanide standard solution (0.100 mg·L^{-1}) of 7.00 mL as the research subject.

The effects of pH values at 6.9, 7.0, 7.1, 7.2, 7.3, and 7.4 on i_p'' were examined. The results showed that when the pH ranged from 7.0 to 7.3, i_p'' reached its maximum value and remained stable. Therefore, a pH of 7.0 was selected for the experiment, controlled by adding 5.0 mL of phosphate buffer solution.

The effects of adding chloramine T solution at volumes of 0.05, 0.10, 0.20, 0.30 mL and reaction times of 1, 2, 3, 4, 5, 6 min on i_p'' were investigated. The results indicated that when the volume of chloramine T solution ranged from 0.1 to 0.3 mL and the reaction time was 3 to 5 min, i_p'' reached its maximum and remained stable. Thus, 0.2 mL of chloramine T solution, corresponding to a concentration of 0.040 g·L^{-1}, was chosen, and the reaction time for converting cyanide to CNCl was set at 3–5 min.

The effects of adding isonicotinic acid solution at volumes of 1.0, 1.5, 2.0, 2.5, 3.0, 3.5 mL on i_p'' were examined. The results showed that when the volume of isonicotinic acid solution ranged from 1.5 to 3.0 mL, i_p'' reached its maximum and remained stable. Therefore, 2.0 mL of isonicotinic acid solution, corresponding to a concentration of 1.20 g·L^{-1}, was selected.

The effects of adding pyrazolone solution at volumes of 0.20, 0.30, 0.40, 0.50, 0.60 mL on i_p'' were investigated. The results indicated that when the volume of pyrazolone solution ranged from 0.30 to 0.50 mL, i_p'' reached its maximum and remained stable. Thus, 0.40 mL of pyrazolone solution, corresponding to a concentration of 0.20 g·L^{-1}, was chosen.

To investigate the role of bispyrazolone, the absorbance (A) of the test solution was measured simultaneously. The effects of adding bispyrazolone solution at volumes (V) of 0, 0.08, 0.12, 0.16, 0.20, 0.28, 0.40 mL on i_p'' and A were examined, and the results are shown in Figure 2. The results showed that when V was less than 0.2 mL, both i_p'' and A increased simultaneously with increasing V; when V was greater than 0.2 mL, both i_p'' and A decreased simultaneously with increasing V. Therefore, 0.20 mL of bispyrazolone solution, corresponding to a concentration of 8.0 mg·L^{-1}, was selected. Compared to V = 0 mL, i_p'' at V = 0.20 mL increased by 12%, indicating that the addition of bispyrazolone improved the sensitivity of the method.

The A~V variation indirectly reflected the effect of bispyrazolone on the concentration of the blue dye produced by the König reaction. The A~V trend was basically consistent with the i_p''~V trend, indicating that bispyrazolone enhanced the sensitivity of polarographic analysis by increasing the concentration of the blue dye.

To simplify the operation and avoid the loss of CNCl, bispyrazolone solution, pyrazolone solution, and isonicotinic acid solution were mixed in a volume ratio of 1:2:10, resulting in a total addition of 2.6 mL of the bispyrazolone – pyrazolone – isonicotinic acid solution.

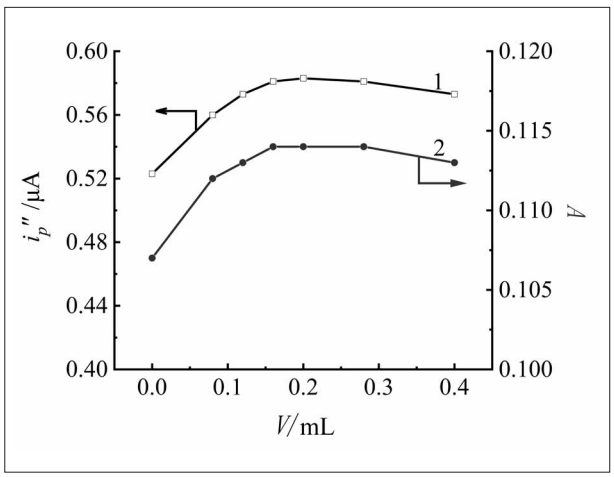

Fig. 2. Effects of the volume (V) of bispyrazolone solution on the absorbance (A) and second-order derivative peak current (i_p'')

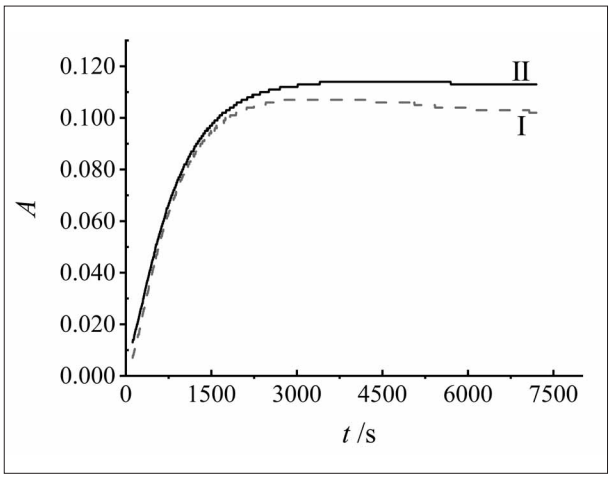

Fig. 3. *A*~*t* curves of systems: I – isonicotinic acid–pyrazolone, II – isonicotinic acid–pyrazolone–bispyrazolone

To further explore the mechanism of bispyrazolone, the time – scan function of the spectrophotometer [11] was utilized to investigate the absorbance – time (*A*~*t*) curves of System I (isonicotinic acid – pyrazolone) and System II (isonicotinic acid – pyrazolone – bispyrazolone) within 120 min. As depicted in Figure 3, the significant decrease of *A* over time after 70 min in system I, indicative of the blue dye's instability, can be attributed to the oxidation and decolorization of the dye caused by residual HClO from the chlorination process. In contrast, in System II, *A* decreases slightly only after 95 min, and the A_{max} value of System II is significantly higher than that of System I, with a longer duration of A_{max}. Thus, the introduction of bispyrazolone can effectively inhibit the decomposition of the blue dye, enabling synchronous increases in *A* and i_p'' (Fig. 2).

It has been reported that in the Konig reaction, pyrazolone not only participates in the condensation process but also eliminates the oxidative decolorization effect of HClO on blue dyes through its reaction with HClO. However, an excess of pyrazolone also lead to a reduction in CNCl [12], subsequently decreasing the sensitivity of the method. The optimal concentration of pyrazolone chosen in the experiment was 0.20 g·L^{-1}. The significant decrease in *A* value in System I when *t*>70 min indicates that HClO still remains in the system. However, in System II, with an additional 8.0 mg·L^{-1} of bispyrazolone, the *A* value is higher and more stable, suggesting that trace amounts of bispyrazolone can further reduce residual HClO while avoiding the negative impact of excessive pyrazolone on sensitivity, thus providing both stabilizing and sensitizing effects.

With the reaction time fixed at 60 min, the effect of temperature on *i*p″ was investigated within the range of 18–55 °C. The results show that *i*p″ increases with temperature when below 30 °C, reaches a maximum and remains stable within the range of 30–45 °C, and then decreases with further increases in temperature above 45 °C. This suggests that an increased temperature significantly accelerates the rate of the Konig reaction, however, excessively high temperatures can expedite the decomposition of blue dyes. Consequently, a reaction temperature of 35 °C was selected for the Konig reaction in this experiment.

The effect of scan rate (ΔE/t) on ip″ was investigated within the range of -300 to -900 mV/s (at intervals of 100 mV/s). As shown in Figure 4, i_p'' significantly increases with the increase in $|\Delta E/t|$ within the range of -300 to -700 mV/s, reaching a maximum at $\Delta E/t$ = -700 mV/s, and then slightly decreases with further increases in $|\Delta E/t|$. Therefore, a scan rate of -700 mV/s was selected.

Linear Range and Detection Limit of the Method. A good linear relationship was observed between i_p'' and cyanide (CN^-) concentrations ranging from 0.00, 0.32, 0.80, 2.00, 5.00, 10.0, 20.0, 40.0, to 60.0 μg·L^{-1}. With the cyanide concentration (ρ, μg·L^{-1}) as the horizontal axis and i_p'' (nA) as the vertical axis, the linear equation was derived as i_p''=20.96ρ+3.00, with a correlation coefficient R^2=0.9991. At a given confidence level of 95%, the limit of detection (LOD) is calculated as LOD=4.6S_b/K, where S_b is the standard deviation of i_p'' obtained from 20 measurements of reagent blanks, and *K* is the slope of the linear equation. Given S_b=0.36 nA and *K*=20.96, the LOD can be calculated as LOD=4.6·0.36/20.96=0.08 μg·L^{-1}. Following the convention of using 4 times LOD as the lower limit of quantitation (LOQ), the LOQ is determined to be 0.32 μg·L^{-1}, which is one-fiftieth of that achieved by the isonicotinic acid-pyrazolone spectrophotometric method [6]. The linear range (LR) of this method spans from 0.32 to 60 μg·L^{-1}.

Mechanism of Polarographic Analysis. A CV analysis was conducted on the reaction products. A peak appeared during the negative scan, while no peak was observed during the positive scan. Additionally, the peak current decreased with increasing scan cycles, indicating the irreversible nature of the polarographic wave. The mercury column height (*h*) exhibited a good linear relationship with i_p'' within the range of 10.6 to 33.0 cm, with a correlation coefficient

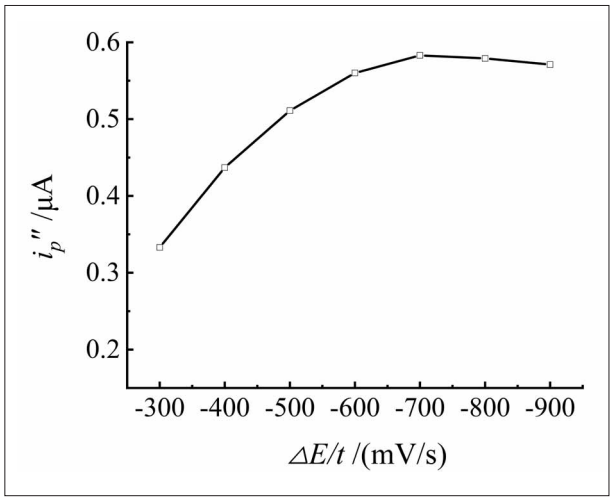

Fig. 4. Effects of scanning rate ($\Delta E/t$) on i_p

R^2=0.9972, consistent with the adsorption current formula $i_a=kh$. Upon adding small amounts of cetylpyridinium bromide, emulsifier OP, and sodium dodecyl sulfate, the polarographic wave decreased significantly or even disappeared, in line with the relationship between adsorption waves and surfactants. Within the temperature range of 26 to 30 °C, i_p'' decreased with increasing temperature, with a temperature coefficient of -0.42%, conforming to the relationship between adsorption current and temperature. In summary, the effects of h, surfactants, and temperature on i_p'' all indicate that this polarographic wave exhibits characteristics of an adsorption wave. The half-peak width of the non-derivative wave was measured as $W_{1/2}$=48.0 mV at 25°C. Using the known formula for irreversible waves $W_{1/2}=2.446RT/(anF)$ [13], we calculated an = 1.31. With the constant a=0.58, the electron transfer number n was calculated as 2.26, which approximates 2. The peak potential (E_p) of this polarographic wave shifted negatively with increasing pH. Over the pH range of 6.1 to 7.6, the relationship was given by E_p=-0.0856pH-0.182 (R^2=0.9980), yielding dE_p/dpH=-0.0856. From this, the proton transfer number m was calculated as 1.9, approximating 2. Based on the electron and proton transfer numbers, the electrode reaction mechanism of the blue dye product was was inferred. A carbonyl group on the pyrazolone moiety of the blue dye accepts two electrons and two protons, reducing to a hydroxyl group on the dropping mercury electrode, resulting in a reversible polarographic wave.

Sample Analysis. The cyanide content in mineral water, tap water, well water, and river water were analyzed, with each sample being measured in parallel for three times. The results are shown in Table 1. The results indicate that the recovery rate of the method ranges from 96.7% to 107%, and the relative standard deviation (RSD) is less than 4.5%. This method exhibits high accuracy and precision. The high precision suggests that the method has good reproducibility, while both high accuracy and good reproducibility indicate that this method is suitable for the determination of trace amounts of cyanide in water.

Method Comparison. A comparison of the detection limit and measurement range of this method with other cyanide detection methods reported in the literature is presented in Table 2. As shown in Table 2, the current national standard methods for cyanide detection (HJ 484–2009,

Table 1

Analysis result of different samples

Sample	Detected	Added	Recovery (%)	RSD (%)
	μg·L^{-1}	μg·L^{-1}		
Mineral water	0.34	0.15	107	4.5
	0.32	0.30	103	
	0.35	0.60	98.3	
Tap water	4.16	2.00	97.0	2.4
	4.12	4.00	98.8	
	4.21	8.00	102	
Well water	12.1	6.00	96.7	1.7
	12.1	12.0	97.5	
	12.2	24.0	101	
River water	25.4	12.0	102	1.4
	24.9	14.0	97.1	
	25.6	48.0	99.0	

Table 2

Comparison of different methods for cyanide detection

Method	LOD	LR	References
	μg·L^{-1}	μg·L^{-1}	
Isonicotinic acid – pyrazolone spectrophotometry	4	16~250	[8]
Isonicotinic acid – barbituric acid spectrophotometry	1	4~450	[8]
Headspace – gas chromatography (GB 5009.36-2016)	1	2~100	[14]
Headspace – gas chromatography (with NaClO)	0.2	0.4~80	[15]
Continuous flow injection spectrophotometry	0.2	2~200	[5]
Continuous flow injection amperometry	0.2	0.6~80	[16]
Ion chromatography	0.15	1~30	[17]
Capillary electrophoresis – laser induced fluorescence	0.1	0.4~13	[18]
Isonicotinic acid–pyrazolone–bispyrazolone polarography	0.08	0.32~60	This work

GB 5009.36–2016) are unable to accurately measure trace amounts of cyanide with concentrations below 1 μg·L^{-1}. By improving the standard methods or adopting advanced instrumentation, the sensitivity can be enhanced, allowing the LOD and LOQ for cyanide to be reduced below 1 μg·L^{-1}. The LOD of this method is slightly lower than that of capillary electrophoresis – laser – induced fluorescence, significantly lower than that of headspace – gas chromatography (with NaClO as the derivatizing agent), continuous flow injection spectrophotometry, continuous flow injection amperometry, and ion chromatography, but higher than that of gas chromatography – mass spectrometry. It can be seen that only the gas chromatography – mass spectrometry method based on 2-(dimethylamino)-ethylthiol-CN^- derivatives exhibits better sensitivity than this method. However, compared to it, this method offers simpler operation, lower equipment cost, and a wider linear range.

Conclusion

This work has developed a novel method for the determination of trace cyanide in water using isonicotinic acid – pyrazolone – bispyrazolone polarography. By replacing spectroscopic analysis with polarographic analysis and adding bispyrazolone to inhibit product decomposition, the detection limit for cyanide has been reduced to one-fiftieth the detection limit of isonicotinic acid–pyrazolone spectrophotometry (HJ 484-2009). This method offers advantages such as operation simplicity, high accuracy, good reproducibility, and does not require expensive equipment. In addition, the mechanism of bispyrazolone and polarographic analysis study provides a theoretical basis for further cyanide analysis research.

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