

HJ 665-2013

水质 氨氮的测定

连续流动-水杨酸分光光度法

Water quality-Determination of ammonium nitrogen by
continuous flow analysis(CFA) and Salicylic acid spectrophotometry

(发布稿)

2013-10-25发布

2014-01-01实施

发布

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前 言

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2013 10 25
2014 1 1

水质 氨氮的测定 连续流动-水杨酸分光光度法

1 适用范围

		30mm	0.01mg/L	N
0.04mg/L~1.00mg/L			10mm	
0.04mg/L	N	0.16mg/L~10.0mg/L		

2 规范性引用文件

HJ 536
HJ/T 91
HJ/T 164

3 方法原理

3.1

3.2

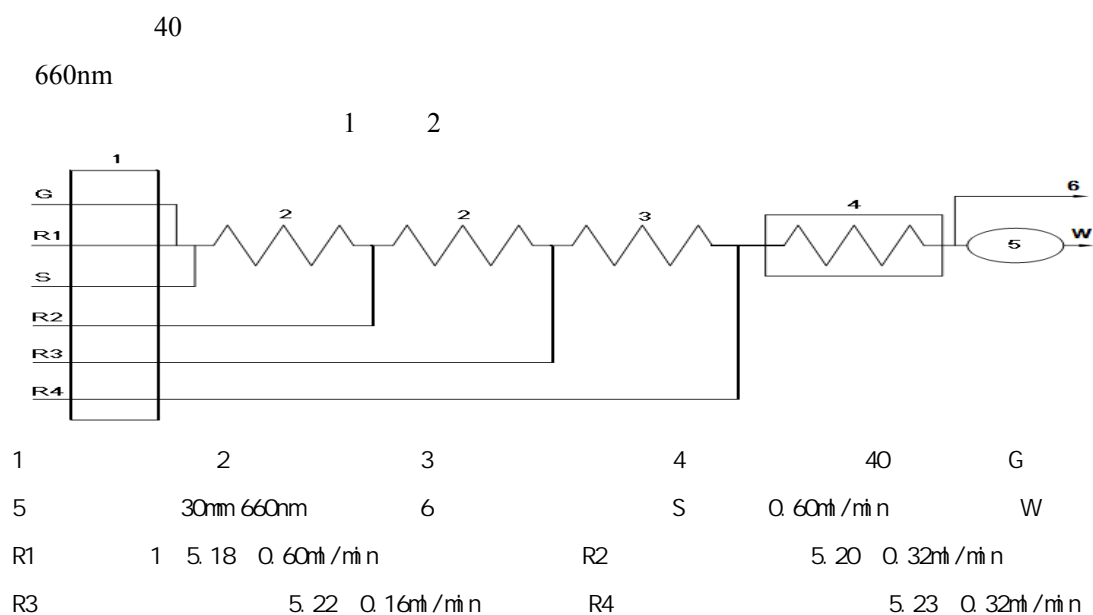


图1 直接比色法测定氨氮参考工作流程图

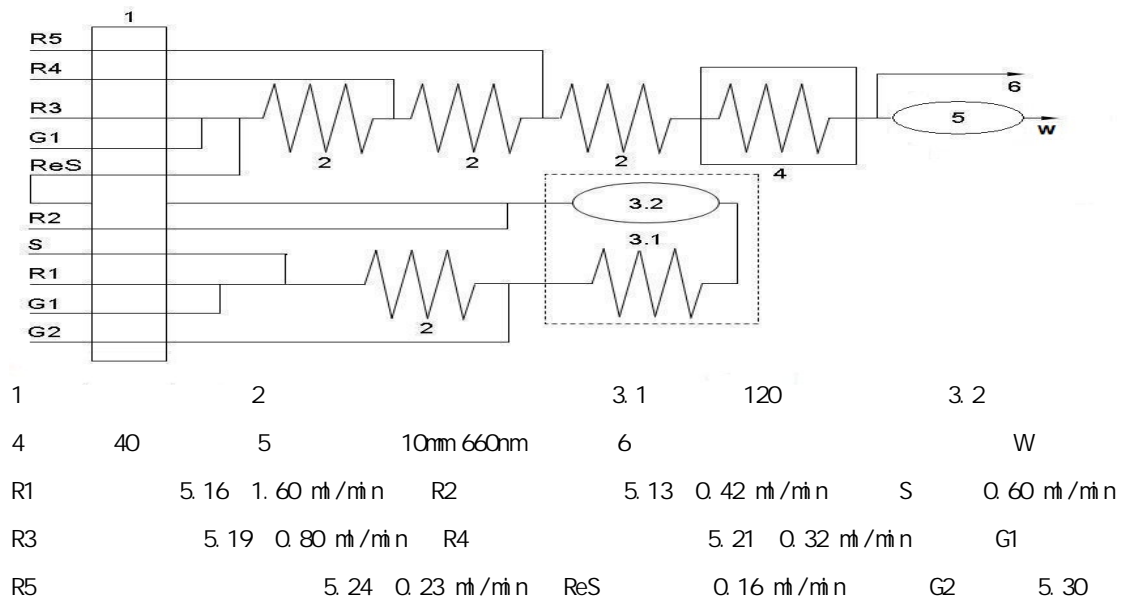


图 2 蒸馏后比色法测定氨氮参考工作流程图

4 干扰和消除

- 4.1 5.28
- 4.2 150mg/L 10mg/L 10000mg/L
- HJ 536
- 2 300mg/L
- 4.3 pH>10 pH<4 pH
- 4.4
- H₂SO₄ 5.14

5 试剂和材料

- 10MΩ·cm 25
- 5.1 ρ(HCl)=1.18g/ml
- 5.2 ρ(H₂SO₄)=1.84g/ml
- 5.3 NH₄Cl 105 ±5
- 5.4 NaOH
- 5.5 EDTA-2Na C₁₀H₁₄N₂Na₂O₈·2H₂O
- 5.6 C₄H₄O₆KNa·4H₂O
- 5.7 C₆H₅O₇Na₃·2H₂O
- 5.8 NaC₇H₅O₃
- 5.9 Na₂[Fe(CN)₅NO]·2H₂O
- 5.10 C₃Cl₂N₃O₃Na·2H₂O
- 5.11 Brij35 C₅₈H₁₁₈O₂₄

5.12		$\text{Na}_2\text{S}_2\text{O}_3$					
5.13		$c(\text{H}_2\text{SO}_4)=0.16\text{mol/L}$					
	7.5ml	5.2	800ml			1000ml	
5.14		$c(\text{H}_2\text{SO}_4)=0.5\text{mol/L}$					
	27ml	5.2	800ml			1000ml	
5.15		$\rho(\text{NaOH})=0.2\text{g/ml}$					
	200g	5.4				1000ml	
5.16							
	5g EDTA-2Na	5.5	600ml	140g	5.4		1000ml
5.17			Brij35	$\omega=30\%$			
	30 Brij35	5.11	100ml				
5.18		$\text{pH}=5.2$					
	33g	5.6	24g	5.7	800ml		1000ml
	3ml Brij35	5.17		5.1	pH	5.2 ± 0.1	
	4			pH			
5.19		$\text{pH}=5.2$					
	30g	5.7	800ml		1000ml	1ml Brij35	5.17
		5.1	pH	5.2 ± 0.1		4	
		pH					
5.20							
	25g	5.4	800ml	80g	5.8		1000ml
			4	1			
5.21							
	70g	5.8	1g		5.9	600ml	
	250ml	5.15		1000ml			4
	1						
5.22			$(\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}]\cdot 2\text{H}_2\text{O})=0.1\%$				
	1.0g		5.9	800ml		1000ml	
		4	1				
5.23			$(\text{C}_3\text{Cl}_2\text{N}_3\text{O}_3\text{Na}\cdot 2\text{H}_2\text{O})=0.2\%$				
	2.0g		5.10	800ml		1000ml	
	4	1					
5.24			$\text{C}_3\text{Cl}_2\text{N}_3\text{O}_3\text{Na}\cdot 2\text{H}_2\text{O} =3.5\%$				
	3.491g		5.10	800ml		1000ml	
	4	1					
5.25		$\rho(\text{N})=1000\text{mg/L}$					
	3.819g	5.3			1000ml		

4	6		
5.26	$\rho(N)=100\text{mg/L}$		
	10.00ml	5.25	100ml
4	7d		
5.27	$\rho(N)=10.0\text{mg/L}$		
	10.00ml	5.26	100ml
5.28	$\rho=3500\text{mg/L}$		
	3.5g	5.12	1000ml
5.29			
		NaClO	1.3%
5.30	99%		
5.31	0.45 μm		

6 仪器和设备

6.1

10mm 30mm

6.2

6.3 0.0001g

6.4 pH ± 0.02

6.5 4000r/min

6.6

7 样品

7.1

5.2 pH<2 2 ~5

7d pH

7.2

5.31

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8 分析步骤

8.1

15min

8.2~8.4

8.2 校准

8.2.1

		5.27		100ml	6
mg/L	1.00mg/L	0.00 mg/L	0.05 mg/L	0.25 mg/L	0.50 mg/L
				0.80	

		5.26		100ml	6
mg/L	10.0mg/L	0.00 mg/L	0.20 mg/L	1.00 mg/L	3.00 mg/L
				6.00	

8.2.2

8.2.1

N mg/L

8.3

7.2

注

8.4

8.3

9 结果计算与表示

9.1

N mg/L 1

$$\rho = \frac{y-a}{b} \times f \quad 1$$

——

y——

a——

b——

f——

9.2

1.00mg/L

1.00mg/L

10 精密度和准确度

10.1

6	0.10mg/L	0.50mg/L	0.90mg/L
---	----------	----------	----------

1.8%~9.2% 0.6%~2.8% 0.4%~2.6%
 5.0% 3.5% 2.2% 0.01mg/L 0.02mg/L
 0.03mg/L 0.02mg/L 0.05mg/L 0.06mg/L
 6 1.00mg/L 5.00mg/L 9.00mg/L
 0.5%~2.8% 0.2%~2.4% 0.1%~1.1%
 2.7% 2.3% 2.3% 0.05 mg/L 0.17 mg/L
 0.18mg/L 0.09 mg/L 0.35 mg/L 0.60 mg/L
 10.2
 6 0.54mg/L±0.03mg/L 0.67mg/L±0.03mg/L
 0.9%~4.3% 0.3%~3.2%
 2.8%±2.6% 1.9%±2.6% 6
 0.04mg/L~0.22mg/L 0.22 mg/L ~0.40 mg/L 0.44 mg/L ~0.84 mg/L 3
 96.0%~102% 93.6%~104% 94.6%~106%
 99.8%±5.2% 99.6%±8.2% 100%±8.4%
 6 1.33 mg/L±0.03mg/L 2.74mg/L±0.12
 mg/L 0.0%~3.8% 0.0%~3.6%
 1.9%±4.4% 1.9%±2.8%
 6 0.22 mg/L~2.36 mg/L 1.71 mg/L~5.32 mg/L
 2.24 mg/L~8.05 mg/L 3 95.0%~106%
 95.9%~107% 96.8%~103% 102%±9.2% 99.6%±8.4%
 100%±4.8%

11 质量保证和质量控制

11.1

2

11.2

γ 0.995

10

5%

11.3

10%

10

0.02mg/L~0.10mg/L

20%

0.10 mg/L ~1.0mg/L

15%

>1.0mg/L

10%

11.4

10%

10

80~120%

12 注意事项

12.1

± 5

12.2

12.3

30min

5.29

30min

15min

12.4

12.5

