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# Integrated wastewater discharge standard

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## **Foreword**

This standard is the revision of GB 8978-88 "Integrated wastewater discharge standard".

The main contents of the amendment are as follows: to put forward the year-limitation standard, AND use the year-limitation to replace the classification between the current enterprises and the newly reconstructed-expanded enterprises in the original standard. It divides into two time-sections using the date of implementation of this standard as a year-limitation. The enterprises that are built before December 31, 1997 shall follow the standard values as specified in the first time-section; AND the enterprises that are built after January 1, 1998 shall follow the standard values as specified in the second time-section.

In the scope of application of the standard, it defines that the integrated discharge standard and the industrial discharge standards are not followed at the same time. The wastewater discharge by the paper making industry, shipping, shipping industry, marine petroleum development industry, textile dyeing industry, meat product industry, synthetic ammonia industry, iron and steel industry, utilization of aviation propellants, weapon industry, phosphate fertilizer industry, caustic soda and polyvinyl chloride industry is subjected to the relevant national industrial standards. All the other enterprises that discharge wastewater are subjected to this standard. Except for the 12 industries aforementioned, the following released 17 industrial water pollutant discharge standards are included into this revision.

As compared with the original standard, in this standard, the standard values in the first time-section are basically maintained at the newly reconstructed and extended level of the original standard. In order to control the characteristic pollutants and other toxic and hazardous pollutants in the 17 industrial water pollutant discharge standards which had been included into this revision, it increases 10 control items; AND in the second time-section, it increases 40 control items as compared with the original standard, AND the maximum allowable discharge concentration of such items as COD and BOD5 are more stringent to some extent.

The standard, from the date of entry into force, replaces GB 8978-88, AND it replaces the following standards at the same time:

GBJ 48- 83 Hospital wastewater discharge standard (Trial)

GB 4645-83 Beet sugar industrial water pollutant discharge standard

GB 3546-83 Cane sugar industrial water pollutant discharge standard

GB 3547-83 Synthetic fatty acid industrial pollutant discharge standard

GB 3548-83 Synthetic detergent industrial pollutant discharge standard

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#### NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

## Integrated wastewater discharge standard

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Replacing GB 8978-88

This standard was established to implement the Environmental Protection Law of People's Republic of China, the Water Pollutant Prevention and Control Law of People's Republic of China, and the Marine Environmental Protection Law of People's Republic of China, to control water pollution, protect surface water in rivers, lakes, canals, channels, reservoirs, and the sea, protect the quality of groundwater, safeguard the people's health, ensure ecological balance, AND promote the development of the national economy and rural and urban construction.

# 1 Main content and scope of application

#### 1.1 Main content

This standard specifies the maximum allowable discharge concentration of the 69 water pollutants AND the maximum allowable discharge amount of some industries based on a fixed year-limitation, in accordance with the discharge direction of the wastewater.

## 1.2 Scope of application

This standard applies to the water pollutant discharge management of the current enterprises, the environmental impact assessment of the construction projects, AND the environmental protection facility design, completion acceptance, and the discharge management after putting into operation of the construction projects.

In accordance with the principles that the Integrated wastewater discharge standard and the national industrial discharge standards shall not apply concurrently, the paper making industry is subjected to the Paper making industrial water pollutant discharge standard (GB 3544-92); the shipping is subjected to the Shipping pollutant discharge standard (GB 3552-83); the marine industry is subjected to the Marine industrial pollutant discharge standard (GB 4286-84); the marine petroleum development industry is subjected to the Marine petroleum development oil bearing industrial wastewater discharge standard (GB 4914-85); the textile dyeing industry is subjected to the Textile dyeing industrial water pollutant discharge standard (GB 4287-92); the meat product industry is subjected to the Meat product industrial water pollutant discharge standard (GB 13457-92); the synthetic ammonia industry is subjected to the Synthetic ammonia industrial water

- **4.1.1** The wastewater which is discharged into the category III water area of GB 3838 (except for the protected areas and the swimming areas as indicated) AND which is discharged into the category II marine area of GB 3097 shall follow the class 1 standards.
- **4.1.2** The wastewater which is discharged into the category IV and V water area of GB 3838 AND which is discharged into the category III marine area of GB 3097 shall follow the class 2 standards.
- **4.1.3** The wastewater which is discharged into the urban drainage system having the secondary wastewater treatment plant shall follow the class 3 standard.
- **4.1.4** The wastewater which is discharged into urban drainage systems having no secondary wastewater treatment plant shall follow the requirements of clause 4.1.1 and 4.1.2, respectively, in accordance with the functional requirements of the discharge system water receiving area.
- **4.1.5** In the protected area as indicated in the category I and II water area and the category III water area in GB 3838 AND the category I marine area in GB 3097, it is prohibited to build new pollutant discharge outlet. The current pollutant discharge outlet shall, in accordance with the water body functional requirements, be subjected to the total pollutant amount control, in order to ensure that the receiving water body complies with the water quality standard of the specified purposes.

#### 4.2 Standard values

- **4.2.1** In this standard, the pollutants discharged are divided into two categories based on their nature and control method.
- **4.2.1.1** Category I pollutant: it is sampled at the workshop or workshop treatment facility discharge outlet regardless of the industries and wastewater discharge method, or the functional category of the receiving water body, AND its maximum allowable discharge concentration must reach to the requirements of this standard (the tailing dam water outlet of the mining industry shall not be considered as the workshop discharge outlet).
- **4.2.1.2** Category II pollutant: it is sampled at the pollutant discharge-enterprise's discharge outlet, AND its maximum allowable discharge concentration must reach to the requirements of this standard.
- **4.2.2** In this standard, it specifies the maximum allowable discharge concentration of the category I pollutant and the category II pollutant AND the maximum allowable water discharge amount of some industries, which are respectively:
- **4.2.2.1** As for the enterprises which are built (including reconstructed and extended) before December 31, 1997, the discharge of the water pollutant must follow simultaneously the requirements of Table 1, Table 4, and Table 5.

Table 2 -- Maximum allowable discharge concentration for category II pollutants (for the enterprises constructed before December 31, 1997)

Unit: mg/L

			Class 4		Olass 2
No.	Pollutants	Scope of application	Class 1 standard	Class 2 standard	Class 3 standard
1	pH	All discharge-enterprises	6 ~ 9	6 ~ 9	6 ~ 9
	Pii	Dyeing industry		180	-
2	Color (dilution ratio)	Other discharge-enterprises	50 50	80	-
		Mining, ore dressing, coal dressing	100	300	_
		Vein gold dressing	100	500	-
3	Suspended solids	Alluvial gold dressing in outlying districts	100	800	-
	(SS)	Urban secondary wastewater treatment plant	20	30	-
		Other discharge-enterprises	70	200	400
		Cane sugar processing, ramie degluing, wet fiber board industries	30	100	600
4	BOD₅	Beet sugar processing, alcohol, MSG, leather, chemical fiber starch industries	30	150	150 600
		Urban secondary wastewater treatment plant	20	30	-
		Other discharge-enterprises	30	60	300
	synthetic fatty ac dyes, fur treatment organophosphor manufacturing  MSG, alcohol, m medicine raw mapharmaceuticals fiber starch industrefining)  Urban secondar treatment plants	Beet sugar processing, coking, synthetic fatty acid, wet fiberboard, dyes, fur treatment, organophosphorus pesticide	100	200	1000
5		MSG, alcohol, medicines and medicine raw materials, biological pharmaceuticals, leather, chemical fiber starch industries	100	300	1000
		Petrochemical industry (including refining)	100	150	500
		Urban secondary wastewater treatment plants	60	120	-
		Other discharge-enterprises	100	150	500
6	Petroleum hydrocarbons	All discharge-enterprises	10	10	30
7	Vegetable and animal oils	All discharge-enterprises	20	20	100
8	Volatile phenols	All discharge-enterprises	0.5	0.5	2.0
9	Total cyanides (CN)	Motion picture film processing (ferrous cyanide)	0.5	5.0	5.0
	. ,	Other discharge-enterprises	0.5	0.5	1.0

Table 2 (continued)

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No.	Dollutonto	Soons of application	Class 1	Class 2	Class 3	
INO.	Pollutants	Scope of application	standard	standard	standard	

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Table 3 -- Maximum allowable wastewater discharge amount for some industries (for the enterprises constructed before December 31, 1997)

ina	ustries (to	r tne ente	rprises constructed be	fore December 31, 1997)		
No.		Indus	try category	Maximum allowable discharge amount OR minimum allowable		
			recycling rate			
	_		errous metals mining	Water recycling rate 75%		
		Other min	ing, ore processing and coal	Water recycling rate 90% (coal		
	Mining		processing	processing)		
1	_	Arterial	Gravity	16.0 m <sup>3</sup> /t (ore)		
		gold	Flotation	9.0 m <sup>3</sup> /t (ore)		
		dressing —	Cyanide	8.0 m <sup>3</sup> /t (ore)		
2	Calcing indust	tru / cool goo	Molten carbon	8.0 m <sup>3</sup> /t (ore) 1.2 m <sup>3</sup> /t (coke)		
3	Coking indust		• '	, ,		
3	Ferrous meta	i smeiling an	d metal processing	Water recycling rate 80% A: > 5000000 t, 1.0 m <sup>3</sup> /t (crude oil)		
				2500000 × 5000000 t, 1.2 m <sup>3</sup> /t		
	Oil refining in	ductry (not in	cluding direct discharge oil	(crude oil)		
	refineries)	dustry (110t III	cluding direct discharge oil	< 2500000 t, 1.5 m <sup>3</sup> /t (crude oil)		
	Classification	of processin	a denths:	B: > 500000 t, 1.5 m <sup>3</sup> /t (crude oil)		
	A. Fuel refi	-	g deptilis.	2500000 ~ 5000000 t, 2.0 m <sup>3</sup> /t		
4		ıbricant oil re	fineries	(crude oil)		
			rochemical oil refineries	< 2500000 t, 2.0 m <sup>3</sup> /t (crude oil)		
	(including refineries processing high sulfur crude oil shale			C: > 5000000 t, 2.0 m³/t (crude oil)		
	oil and oil add	•	3 3	2500000 ~ 5000000 t, 2.5 m <sup>3</sup> /t		
		,	(crude oil)			
				< 2500000 t, 2.5 m <sup>3</sup> /t (crude oil)		
	Chlorinating method or producing alkali		200.0 m³/t (alkali benzene)			
	Synthetic	benzene Splitting m	ethod of producing alkali			
5	detergent industry	benzene	lethod of producing alkali	70.0 m³/t (alkali benzene)		
			zene to produce synthetic			
		detergent	zono to produce symmons	10.0 m <sup>3</sup> /t (product)		
6	Synthetic fatty		V	200.0 m <sup>3</sup> /t (product)		
7			hod of producing fiber board	30.0 m <sup>3</sup> /t (board)		
_	Sugar		ar processing	10.0 m³/t (sugar cane)		
8	industry		r processing	4.0 m <sup>3</sup> /t (sugar beets)		
		Wet salt p	ig skin	60.0 m³/t (raw skin)		
9	Leather	Dry cow s	kin	100.0 m <sup>3</sup> /t (raw skin)		
	industry	Dry sheep	skin	150.0 m <sup>3</sup> /t (raw skin)		
		A1 I I	Corn as raw material	100.0 m³/t (alcohol)		
	F 4 - 4:	Alcohol	Potato as raw material	80.0 m <sup>3</sup> /t (alcohol)		
10	Fermentation and brewing	industry	Molasses as raw material	70.0 m³/t (alcohol)		
10	industry	MSG indu	stry	600.0 m <sup>3</sup> /t (MSG)		
	industry		stry (wastewater amount does e malt water)	16.0 m <sup>3</sup> /t (beer)		
11	Chromate ind		o mait water j	5.0 m <sup>3</sup> /t (product)		
12		-	er washing method)	15.0 m <sup>3</sup> /t (sulfuric acid)		
			or washing moulou)	500 m <sup>3</sup> /t (raw flax) OR 750 m <sup>3</sup> /t		
13	Ramie deglui			(dry refined flax)		
14	Chemical fiber industry		Unbleached: 150 m³/t (pulp)			

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	halide (AOX) (as CI)				
31	Chloroform	All discharge-enterprises	0.3	0.6	1.0
32	Carbon tetrachloride	All discharge-enterprises	0.03	0.06	0.5
33	Trichlorethylene	All discharge-enterprises	0.3	0.6	1.0
34	Tetrachloroethylene	All discharge-enterprises	0.1	0.2	0.5
35	Benzene	All discharge-enterprises	0.1	0.2	0.5
36	Methylbenzene	All discharge-enterprises	0.1	0.2	0.5
37	Ethylbenzene	All discharge-enterprises	0.4	0.6	1.0
38	38 o-Xylene All d	All discharge-enterprises	0.4	0.6	1.0
39	Paraxylene (p-Xylene)	All discharge-enterprises	0.4	0.6	1.0
40	m-Xylene	All discharge-enterprises	0.4	0.6	1.0

Table 4 (continued)

No.	Pollutants	Scope of application	Class 1	Class 2	Class 3
INO.	Foliularits	Scope of application	standard	standard	standard
41	Chlorobenzene	All discharge-enterprises	0.2	0.4	1.0
42	o-Dichlorobenzene	All discharge-enterprises	0.4	0.6	1.0
43	p-Dichlorobenzene	All discharge-enterprises	0.4	0.6	1.0
44	p-Nitrochlorobenzene	All discharge-enterprises	0.5	1.0	5.0
45	2,4-Dinitrochlorobenzene	All discharge-enterprises	0.5	1.0	5.0
46	Phenol	All discharge-enterprises	0.3	0.4	1.0
47	M-cresol	All discharge-enterprises	0.1	0.2	0.5
48	2,4-Chlorophenol	All discharge-enterprises	0.6	0.8	1.0
49	2,4,6-Trichlorophenol	All discharge-enterprises	0.6	0.8	1.0
50	Dibutyl (o-) phthalate	All discharge-enterprises	0.2	0.4	2.0
51	Dioctyl (o-) phthalate	All discharge-enterprises	0.3	0.6	2.0
52	Acrylonitrile	All discharge-enterprises	2.0	5.0	5.0
53	Total selenium	All discharge-enterprises	0.1	0.2	0.5
54	Excrement, intestines, fungus count	Hospitals *, veterinary hospitals, medical treatment institutions with wastewater containing pathogens	500 / L	1000 / L	5000 / L
		Wastewater from hospitals with contagious diseases and tuberculosis	100 / L	500 / L	1000 / L
55	Total excess chlorine (used for chlorine	Hospitals *, veterinary hospitals, medical treatment institutions with wastewater containing pathogens	< 0.5 **	> 3 (exposure time ≥ 1 h)	> 2 (exposure time ≥ 1 h)
Jü	disinfection in hospital wastewater)	Wastewater from hospitals with contagious diseases and tuberculosis	< 0.5 **	> 6.5 (exposure time ≥ 1.5 h)	> 5 (exposure time ≥ 1.5 h)
	Tatal annunia annuas	Synthetic fatty acid industry	20	40	-
56	Total organic carbon	Ramie degluing industry	20	60	-
	(TOC)	Other discharge-enterprises	20	30	-

Note: Other discharge-enterprises refer to all the other enterprises than those listed in these controlled items.

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	water)	
11	Chromate industry	5.0 m <sup>3</sup> /t (product)
12	Sulfuric acid industry (water washing method)	15.0 m³/t (sulfuric acid)
40	Ramie degluing industry	500 m³/t (raw flax)
13		750 m³/t (dry refined flax)

**Table 5 (continued)** 

	Table 5 (continued)					
No.	Ind	lustry category	Maximum allowable discharge amount OR minimum allowable			
		Ī	recycling rate			
		Short fiber (cotton type medium				
14	Glue fiber industry	length fiber, wool type medium	300 m <sup>3</sup> /t (fiber)			
	(pure fiber)	length fiber)				
		Long fiber	800 m <sup>3</sup> /t (fiber)			
15	Chemical fiber industry	1	Unbleached: 150 m³/t (pulp)			
	Onemical fiber inductry		Bleached: 240 m <sup>3</sup> /t (pulp)			
		Penicillin	4700 m <sup>3</sup> /t (Penicillin)			
		Streptomycin	1450 m <sup>3</sup> /t (Streptomycin)			
		Oxytetracycline	1300 m³/t (Oxytetracycline)			
		Tetracycline	1900 m³/t (Tetracycline)			
		Lincomycin	9200 m³/t (Lincomycin)			
	Pharmaceutical	Aureomycin	3000 m <sup>3</sup> /t (Aureomycin)			
		Gentamicin	20400 m³/t (Gentamicin)			
16	industry and pharmaceutical raw	Vitamin C	1200 m³/t (Vitamin C)			
	materials	Chloramphenicol	2700 m <sup>3</sup> /t (Chloramphenicol)			
	Illaterials	Sulfamethoxazole	2000 m³/t (Sulfamethoxazole)			
		Vitamin B₁	3400 m³/t (Vitamin B₁)			
		Dipyrone	180 m³/t (Dipyrone)			
		Phenacetin	750 m³/t (Phenacetin)			
		Furazolidone	2400 m³/t (Furazolidone)			
		Caffeine	1200 m <sup>3</sup> /t (Caffeine)			
		Dimethoate **	700 m <sup>3</sup> /t (product)			
		Methyl parathion (water phase method) **	300 m <sup>3</sup> /t (product)			
		Parathion (P <sub>2</sub> S <sub>5</sub> method) **	500 m <sup>3</sup> /t (product)			
		Parathion (PSCI <sub>3</sub> method) **	550 m³/t (product)			
17	Organo-phosphorous pesticide industry *	Dichlorvos (trichlorfon alkaline hydrolysis)	200 m <sup>3</sup> /t (product)			
			40 m <sup>3</sup> /t (product) (not including			
		Trichlorfon	wastewater from			
			trichloroacetaldehyde production)			
		Malathion	700 m <sup>3</sup> /t (product)			
		Nitrofen	5 m <sup>3</sup> /t (product)			
		Sodium pentachlorophenate	2 m <sup>3</sup> /t (product)			
		Pentachlorophenate	4 m <sup>3</sup> /t (product)			
		МСРА	14 m <sup>3</sup> /t (product)			
18	Herbicide industry	2,4-D	4 m³/t (product)			
		Butachlor	4.5 m <sup>3</sup> /t (product)			
		Chlorotoluron (reduced by Fe)	2 m³/t (product)			
		Chlorotoluron (reduced by Na <sub>2</sub> S)	3 m <sup>3</sup> /t (product)			

19	Thermal power industry	3.5 m <sup>3</sup> /(MWh)

#### Table 5 (continued)

No.	Industry category	Maximum allowable discharge amount OR		
	, , ,	minimum allowable recycling rate		
20	Freight train washing	5.0 m³/carriage		
21	Motion picture film development	5 m <sup>3</sup> / 1000 m (35 mm film)		
22	Petroleum bitumen industry	Recycling rate of cooling pond water: 95%		

#### Note:

## 5 Monitoring

## 5.1 Sampling points

Sampling points shall be set in accordance with the provisions for the category I and II pollutant discharge outlets in clause 4.2.1.1 and 4.2.1.2, AND it must provide the discharge outlet sign, wastewater amount metering device, and wastewater proportional sampling device at the discharge outlet.

## 5.2 Sampling frequency

The monitoring frequency of the industrial wastewater is determined in accordance with the production cycle. If the production cycle is within 8h, MAKE sampling every 2h; if the production cycle is over 8h, MAKE sampling every 4h. As for the sampling of other wastewater, the sampling number shall be not less than 2 within 24h. The maximum allowable discharge concentration is calculated in accordance with the daily mean value.

#### 5.3 Discharge amount

It is controlled by the maximum allowable discharge amount or the minimum allowable water recycling ratio, both of which are calculated in accordance with the monthly mean value.

### 5.4 Statistics

The use of raw materials, product output, etc. of the enterprises shall be based on the statutory monthly statements or the annual statements.

#### 5.5 Determination method

The determination method used in this standard is as shown in Table 6.

Table 6 -- Determination method

Ī	No.	Item	m Determination method	
ĺ	1	Total mercury	Cold atomic absorption spectrophotometry	GB 7468-87

<sup>\*</sup> Product is measured by 100% concentration.

<sup>\*\*</sup> It does not include P<sub>2</sub>S<sub>5</sub>, PSCl<sub>3</sub>, PCl<sub>3</sub> raw material production wastewater.

# Appendix A (Standard appendix)

Where the discharge-enterprise discharges two or more industrial wastewater at the same discharge outlet AND the discharge standards for the same pollutant in each industrial wastewater are different, it may use the following method to calculate the maximum allowable discharge concentration of this pollutant during the combined discharge (C combined).

$$C \text{ combined } = \frac{\sum_{i=1}^{n} C_{i} Q_{i} Y_{i}}{\sum_{i=1}^{n} Q_{i} Y_{i}}$$

#### Where:

C combined – The maximum allowable discharge concentration of a pollutant in the combined wastewater, mg/L;

C<sub>i</sub> - The maximum allowable discharge concentration of a pollutant in different industrial wastewater, mg/L;

 $Q_i$  - The maximum allowable discharge amount of different industrial wastewater,  $m^3/t$  (product)

(As for the industries which are not covered by this standard, the maximum allowable discharge amount shall be determined jointly by the local environmental protection department and the relevant departments);

 $Y_i$  – The production amount of a certain industrial product (t/d, calculated by monthly mean value).

- (1) 0.1N potassium bromate potassium bromide solution: WEIGH 2.8 g of potassium bromate and 4.0 g of potassium bromide. USE distilled water to dilute it to 1L.
- (2) 1:1 phosphoric acid: ADD distilled water two times of the phosphoric acid.
- (3) Saturated sodium chloride solution: WEIGH 40 g of sodium chloride; DISSOLVE it into 100 ml distilled water.
- (4) 20% potassium bromide solution: WEIGH 20 g of potassium bromide; DISSOLVE it into 100 ml distilled water.
- (5) 5% phenol solution: TAKE 5 ml of phenol; DISSOLVE it into 100 ml distilled water.
- (6) 5% potassium iodide solution: WEIGH 5 g of potassium iodide; DISSOLVE it into 100 ml distilled water. (PREPARE it before use; PLACE it in the dark).
- (7) 0.2% starch solution: WEIGH 1 g of soluble starch; ADD a small amount of water and STIR it uniformly; INJECT it into 500 ml boiling water; CONTINUE boiling it for 5 min; AND it may add 0.2 g of salicylic acid in summer.
- (8) Preparation of standard solution

Accurately WEIGH 0.276 g of hydroquinone (molecular weight of 110.11 g). If it is photographic grade metol (molecular weight of 344.40 g), it may weigh 0.861 g; if it is photographic grade TSS (molecular weight of 262.33 g), it may weigh 0.656 g, (OR otherwise make calculation otherwise in accordance with the molecular weight and purity of the drug used); DISSOLVE it into 25 ml of 6N HCl; TRANSFER it into a 250 ml volumetric flask; ADD distilled water to the mark; AND at this time the solution concentration is 0.0100 M.

#### 4. Steps

- (1) Drawing of standard curve
  - ① TAKE 25 ml of the standard solution; ADD distilled water to dilute it to 1000 ml; the solution concentration is 0.00025M, that is, each milliliter contains 0.25 μmol of hydroquinone (liquid A).
  - 2) TAKE 25 ml of liquid A; ADD distilled water to dilute it to 250 ml; the solution concentration is 0.000025M, that is, each milliliter contains 0.025 µmol of hydroguinone (liquid B).
  - ③ TAKE six 50 ml volumetric flasks; respectively ADD 0, 0.1, 0.2, 0.3, 0.4, and 0.5 μmol of standard diluent (liquid B) of hydroquinone (that is, 4.0, 8.0, 12.0, 16.0, and 20.0 ml of liquid B); ADD appropriate amount of

excess urea to eliminate the disturbance of the excessive NaNO<sub>2</sub> on this test, so as to achieve the purpose of elimination of interference.

Accurately TAKE appropriate amount of water sample (about 1  $\sim$  10 ml), PLACE it into a 50 ml volumetric flask; ADD about 20 ml of distilled water; ADD 2 ml of 1:1 nitric acid; ADD 3 drops of 10% NaNO<sub>2</sub>; SHAKE it thoroughly; PLACE it into the 35 °C constant temperature water bath for 15 min; ADD 2 ml of 20% urea; SHAKE it uniformly; PLACE it into the 35 °C water bath for 10 min. REPEAT the steps (5)  $\sim$  (12); MEASURE the optical density; FIND the molecules (in micrograms) contained in 50 ml from the curve.

#### 5. Calculation

The total amount C of color developer and oxide in the water sample (calculated as hydroguinone) is calculated as follows:

 $C = \mu mol \text{ amount in } 50 \text{ ml } x \text{ } 110/\text{sampling volume } (ml) \text{ } x \text{ } 1000 \text{ } (mg/L)$ 

#### 6. Precautions

- (1) This test has many steps and requires a long time, so the operation must be careful.
- (2) The glassware used must be washed with a cleaning solution.
- (3) The water bath temperature shall be accurate to 35 °C ± 1 °C, AND the reaction time for each step shall be accurately controlled.
- (4) After adding potassium bromate potassium bromide, it must use the distilled water to rinse the vessel wall, otherwise the residual potassium bromate will react with the potassium iodide to produce iodine, which increases the optical density.
- (5) In lead-free wastewater, the water sample may not be processed BUT measured directly.
- (6) If the water samples are too thick, it may be diluted firstly before being determined.

# III. Determination of elemental phosphorus

# - Molybdenum blue colorimetric method

The principle of this method: elemental phosphorus is extracted by benzene AND oxidized into molybdenum phosphate, which is reduced by the stannous chloride reduced into blue complex. Its sensitivity is higher than that of vanadium molybdophosphate colorimetric method, AND it is easy to be

1:1 sulfuric acid solution, 1:5 nitric acid solution, 20% sodium hydroxide solution.

#### Determination steps:

(I) When the elemental phosphorus content in the wastewater is more than 0.05 mg/L, the water phase is directly used for colorimetry in accordance with the following requirements:

Water sample pre-treatment:

- (A) Extraction: TRANSFER 10 ~ 100 ml of water sample into a 125 ml or 250 ml separate funnel containing 25 ml of benzene; after shaking it for 5 min, LET it be standing for stratification. TRANSFER the water phase into another separate funnel containing 15 ml of benzene; SHAKE it for 2 min; LET it be standing; DISCARD the water phase; ADD the benzene phase into the first separate funnel. ADD 15 min [translator note: it should be 15 ml] of water; SHAKE it for 1 min; LET it be standing; DISCARD the water phase; RINSE the benzene phase for 6 times.
- (B) Oxidation: ADD 10 ~ 15 ml of potassium bromate potassium bromide solution into the benzene phase and 2 ml of 1:1 sulfuric acid solution; SHAKE it for 5 min. LET it be standing for 2 min; ADD 2 ml of perchloric acid; SHAKE it 5 min; TRANSFER it into a 250 ml volumetric flask; slowly HEAT it on the electric heating plate to remove the excessive perchloric acid and bromine (do not make the sample spilled or evaporated); when the white smoke reduces, TAKE it down and COOL it; ADD small amount of water and 1 drop of phenolphthalein indicator; USE the 20% sodium hydroxide solution to neutralize it to pink; ADD 1 drop of 1:1 sulfuric acid solution to make the pink disappear; TRANSFER it into the volumetric flask; USE distilled water to dilute it to the mark (determine the dilution volume in accordance with elemental phosphorus content).

Colorimetry: TAKE appropriate amount of above diluted solution into a 50 ml colorimetric tube; ADD 2 ml of 2.5% ammonium molybdate solution and 6 drops of 2.5% stannous chloride solution. ADD water to dilute it to the mark; MIX if uniformly; PLACE it at  $20 \sim 30$  °C for  $20 \sim 30$  min. POUR it into a 3 cm cuvette. At the wavelength 690 nm of the spectrophotometer, USE the reagent blank as zero to measure the optical density.

Drawing of direct colorimetric working curve:

(A) TRANSFER appropriate amount of potassium dihydrogen phosphate standard solution (so that the content of  $PO_4^{-3} - P$  is 0, 1, 3, 5, 7.....17 µg, respectively) into a 50 ml colorimetric tube; MEASURE the optical density.

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