Comprehensive Design Experiment (Ⅱ) and water quality inspection

1、Purpose

(1) Comprehensively apply the theory of precipitation dissolution equilibrium, coordination balance, electrode potential, etc. of insoluble and strong electrolytes to improve the ability to analyze and solve practical problems.
(2) Train the ability to design experimental programs.
(3) Learn to use conductivity meter and determine the conductivity of various water samples.
(4) Learn the principles and methods of measuring water hardness.

2、Principle

(1) Determination of water purity by conductivity meter

Since there are inorganic salt ions in the water, it is actually an extremely dilute electrolyte solution. This dilute electrolyte solution also has the phenomenon of electrical conductivity, and the higher the ion concentration, the stronger the electrical conductivity.

The conductivity of a conductor is generally represent by the resistance R or conductance G., and the two are reciprocal, that is

$$G = 1/R$$

The SI unit of the resistance is Ohm, the symbol is Ω; the SI unit of conductance is Siemens, the symbol is S.

At a certain temperature, the resistance between the two poles and the distance between the poles is proportional to l, inversely proportional to the electrode area A, that is

$$R \propto l/A$$

or

$$R = \rho l/A$$

The proportional constant ρ is called the resistivity. The SI unit is Ω • m and the symbol is Ω • m. The inverse of the resistivity is called the conductivityκ

$$\kappa = 1/\rho$$

The conductivity unit of SI is Xi [menzi] meter^{-1}and the symbol is S m^{-1}.

The above formula was collated:

$$G = \kappa A/ l$$

$$\kappa = G\, l/A$$

According to the above formula, when l/A=1, κ=G, soκis equal to the conductance of the solution between the two poles of 1 unit length and 1 unit area.

The higher the salt content in water, the better the conductivity and the higher the conductivity; on the contrary, the higher the purity of water, the lower the conductivity. And the conductivity of various water samples followed:

water samples	tap water	distilled water	deionized water	high purity water
conductivity k/ S • m^{-1}	0.5-5×10^{-2}	10^{-3}	10^{-4}	5.5×10^{-6}

(2) Determination of water hardness

The total amount of Ca^{2+} and Mg^{2+} contained in the water is called the total hardness of water, referred to as hardness.

There are many methods for determining the hardness of water. The most common method is EDTA coordination titration. In this method, Ca^{2+} and Mg^{2+} in water were titrated with a standard solution of EDTA (indicated by H_2Y^{2-}) in an ammonia buffer solution of pH = 10.0 using chrome black T (HIn^{2-}) as an indicator. Chrome black T can form red complexes with $Ca^{2+}$and Mg^{2+}in water, but the complex is not as stable as EDTA and Ca^{2+}, Mg^{2+}complexes. Therefore, when EDTA was added into the water sample, EDTA first formed complexes with free Ca^{2+} and Mg^{2+},

Then the free Ca^{2+} and $Mg^{2+}$are taken from the indicator complex to form a more stable complex, and the indicator HIn^{2-} is released. Solution changes from red to blue, and the reaction is to reach the end. According to the amount of EDTA and concentration, it can calculate the total hardness of water.

The above reaction process can be expressed as:

$$Mg^{2+} + HIn^{2-} \rightleftharpoons MgIn^{-} + H^{+}$$
$$Ca^{2+} + HIn^{2-} \rightleftharpoons CaIn^{-} + H^{+}$$
$$Ca^{2+} + H_2Y^{2-} \rightleftharpoons CaY^{2-} + 2H^{+}$$
$$Mg^{2+} + H_2Y^{2-} \rightleftharpoons MgY^{2-} + 2H^{+}$$
$$CaIn^{-} + H_2Y^{2-} \rightleftharpoons CaY^{2-} + HIn^{2-} + H^{+}$$
$$MgIn^{-} + H_2Y^{2-} \rightleftharpoons MgY^{2-} + HIn^{2-} + H^{+}$$

The water hardness is expressed by the concentration $mmol \cdot dm^{-3}$ of Ca^{2+} and Mg^{2+} in water. The water hardness can be calculated according to the following formula:

$$\text{water hardness} = c(EDTA) \cdot V(EDTA)/V_{\text{water sample}}$$

among c(EDTA)—EDTA concentration of standard solution($mol \cdot L^{-1}$);
V(EDTA)—titration with EDTA standard solution volume(mL);
$V_{\text{water sample}}$—the volume of water to be measured(mL).

Experimental requirements

(1) According to the experimental content, the use of theoretical knowledge learned to determine the experimental program, the development of specific experimental steps.
(2) Check the solubility constants of the insoluble and strong electrolytes and the stability constants of the complexes. According to the experiment, the conversion rules of the insoluble electrolytes and complexes are explained.
(3) Grasp the use of redox reactions and neutralization precipitation treatment of chromium-containing wastewater basic method.

3、Apparatus and Materials

3.1 Apparatus：

Acidity meter (or pH test paper), Centrifuge, Platform scale, Graduated pipette (5mL), Pipette (10mL), Beaker (500mL, 250mL), Volumetric flask (1000mL, 100mL, 50mL), Graduated cylinder (100mL, 10mL), Test tube, Centrifuge tube, Dropper, Funnel, Funnel holder, Glass rod, Filter paper, Alcohol lamp.

Materials:

$AgNO_3$ (0.1 mol ·L^{-1}), NaCl(0.1 mol ·L^{-1}),

KBr (0.1 mol ·L^{-1}), KI (0.1 mol ·L^{-1}),

Na_2S (0.1 mol ·L^{-1}), $NH_3 \cdot H_2O$ (6 mol ·L^{-1}),

$Na_2S_2O_3$ (1 mol ·L^{-1} and saturation), H_2SO_4 (50%, 3 mol ·L^{-1}),

H_3PO_4 (50%), $Ca(OH)_2$ (saturation),

Chromium standard solution * (1 mol ·L^{-1}),

Diphenylcarbazide-acetone solution **,

Chromium waste liquid *** (1 g ·L^{-1}), $FeSO_4 \cdot 7H_2O$ (s).

* Weighed 0.2829g dried at 110 ℃ for 2h $K_2Cr_2O_7$, dissolved in water and transferred into a 1000 mL volumetric flask, diluted with water to the mark, shake. which was 1L containing 1mg chromium hexavalent chromium standard stock solution.

** Weigh 0.20g diphenylcarbazide, dissolved in 50 mL of acetone, diluted with water to 100 mL, shake, with a brown bottle dress, store the refrigerator, should be used now preparation.

*** With $K_2Cr_2O_7$ or CrO_3 preparation, if the industrial wastewater should be filtered, such as pretreatment.

3.2 Apparatus:

conductivity meter, 5 beakers, 50mL pipet, pipette (2.5 mL,25 mL), conical flask, acid burette, titration tube holder, measuring cylinder(10 mL,50 mL), electric furnace or electric heating plate, thermometer.

Reagents: NH_3-NH_4Cl buffer solution(pH=10), EDTA standard solution (0.005 mol ·L^{-1})， chrome black T indicator.

4、Experimental content

(1) The self-developed scheme was confirmed by the mutual transformation experiment between the complex and the poorly soluble strong electrolyte precipitate:

$$K_S\ (AgCl) > K_S\ (AgBr) > K_S\ (AgI) > K_S\ (Ag_2S)$$

$$K_t\ \{Ag(S_2O_3)_2^{3-}\} > K_t\ \{Ag(NH_3)_2^{+}\}$$

Moreover, $NH_3 \bullet H_2O$ can dissolve AgCl ↓; different concentrations of $Na_2S_2O_3$ can dissolve AgBr↓and AgI↓, but cannot dissolve Ag_2S↓.

(2) The existing industrial wastewater containing hexavalent chromium 1.0 mol•L^{-1}, please design their own experimental program. The chromium in the waste water is removed by chemical method so that the hexavalent chromium content in the water is less than 0.5 mg• L^{-1}, to achieve "Integrated Wastewater Discharge Standard" (GB8978-1996) requirements.

(3) Determination of water purity by conductivity meter

Take three small beakers, each tap water, distilled water, deionized water 50 mL in the beaker, the sample should be measured before the water sample beaker cleaning 2-3 times. Conductivity meter was used to measure their conductivity, record data.

(4) Determination of water hardness

Measure 50.00 mL water (tap water) into in the flask, add 10 mL NH_3-NH_4Cl buffer solution and a small amount of solid chromium black T indicator. In the violent shaking, with EDTA standard solution titration to the solution from purple to blue, it is the end. Record the results, the other to repeat the water sample operation once.

5、Data recording and processing

(1) Determination of water purity by conductivity meter

water sample	tap water	distilled water	deionized water
conductivity κ /μs cm^{-1}			

(2) Determination of water hardness

project	data
concentration of EDTA standard solution	c(EDTA)= mol ·L^{-1}
the volume of the EDTA standard solution used in the titration	V_1= mL
	V_2= mL
the volume of water sample	$V_{\text{water sample}}$ = mL

6、Question

(1) In precipitation and complex transformation experiments, the formation of precipitation if not centrifugal separation, filtration of liquid, directly to the mixture for the next experiment, what will be the impact?

(2) In the treatment of chromium-containing wastewater, the pH of the solution was controlled by adding acid. What is the pH of the solution? What is the pH of the solution controlled by adding alkali? Please calculate the description. In addition, what are the constituents in the resulting precipitate?

(3) What basis is the determination of water purity by conductivity meter?

(4) What is the impact on the following conditions on the determination of conductivity?

① Determination of conductivity when the platinum electrode on the electrode is not fully immersed in water samples to be tested.

② Determination of conductivity when the beaker or conductive electrode cleaning is not clean.

(5) How to wash the burette, pipette and triangular bottle in determination of water hardness?