GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 29.220.20

CCS K 84

GB/T 42323-2023

Water for Lead Acid Storage Battery

铅酸蓄电池用水

(IEC 62877-2:2016, Electrolyte and Water for Vented Lead Acid Accumulators - Part 2: Requirements for Water, MOD)

Issued on: March 17, 2023 **Implemented on: October 1, 2023**

Issued by: State Administration for Market Regulation;

Standardization Administration of the People's Republic of China.

Table of Contents

Water for Lead Acid Storage Battery

1 Scope

This document specifies the requirements, test methods, inspection rules, marking, packaging, transportation and storage of water for lead acid storage batteries.

This document is applicable to water for lead acid storage batteries.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 601 Chemical Reagent - Preparations of Standard Volumetric Solutions

GB/T 602 Chemical Reagent - Preparations of Standard Solutions for Impurity (ISO 6353-1:1982, NEQ)

GB/T 603 Chemical Reagent - Preparations of Reagent Solutions for Use in Test Methods (ISO 6353-1:1982, NEQ)

GB/T 9724 Chemical Reagent - General Rule for the Determination of pH (ISO 6353-1:1982, NEQ)

GB/T 9740 Chemical Reagent - General Method for the Determination of Dry Residue after Evaporation (ISO 6353-1:1982, NEQ)

GB/T 11446.1 Electronic Grade Water

GB/T 23942 Chemical Reagent - General Rules for Inductively Coupled Plasma Atomic Emission Spectrometry

3 Terms and Definitions

The following terms and definitions are applicable to this document.

3.1 water for lead acid storage battery

Water for lead acid storage battery refers to water used for lead acid battery plate preparation, electrolyte preparation, replacement (replenishment) of water lost in electrolyte due to charging

5 Test Methods

5.1 Sampling

5.1.1 In accordance with this document, carry out the test. 5 L of sample shall be taken.

5.1.2 Before sampling, use the water to be tested to repeatedly wash the container. During sampling, avoid contamination, and the sample shall be filled in a clean and airtight plastic or glass container. If the test cannot be carried out in time, the sample shall be stored in a refrigerator at 4 °C.

5.2 Appearance

In a brightly lit room, visually inspect whether the color of the water surface of the sample is colorless and transparent.

5.3 Residue Content

5.3.1 Instruments

The instruments required for the test are as follows:

a) Rotary evaporator, equipped with a 500 mL distillation bottle;

b) Constant-temperature water bath;

c) Evaporating dish: it can be made of platinum, quartz and borosilicate glass;

d) Electric oven: the temperature can be controlled at 105 °C ± 2 °C;

e) Analytical balance: with a division value of 0.1 mg.

5.3.2 Determination steps

5.3.2.1 Sample pre-concentration

Weigh-take 500 g (or measure-take 500 mL) of the sample; by separate times, add the sample to the distillation bottle of the rotary evaporator; on a constant-temperature water bath, evaporate it under reduced pressure (avoid evaporation to dryness). When the sample is finally evaporated to about 50 mL, stop heating.

5.3.2.2 Determination

Transfer the above-mentioned pre-concentrated sample to an evaporating dish with a constant mass at 105 °C ± 2 °C; use 5 g ~ 10 g (or 5 mL ~ 10 mL) of the pre-concentrated sample to wash the distillation bottle for 2 ~ 3 times. Combine the washing solution and the pre-concentrated sample in the evaporating dish and conduct the determination in accordance with GB/T 9740.

and conduct a blank test. After determining a certain number of samples, a standard sample shall be analyzed, so as to monitor these interferences.

5.4.2.2 The samples shall be analyzed as soon as possible. The longer the storage time, the larger the possibility of contamination. If immediate analysis is not possible, seal the sample bottle in a plastic bag and store it in a refrigerator at 4 °C.

5.4.2.3 During the arbitration of the determination of lead (Pb), antimony (Sb), arsenic (As), tin (Sn), bismuth (Bi), copper (Cu), cadmium (Cd), zinc (Zn), selenium (Se), iron (Fe), cobalt (Co), nickel (Ni), chromium (Cr) and manganese (Mn) contents, adopt the atomic absorption spectrophotometry.

5.4.3 Reagents and preparation methods for reagent solutions

5.4.3.1 Water for the blank

EW-I electronic grade water specified in GB/T 11446.1.

5.4.3.2 Nitric acid

ρ = 1.42 g/mL, prepare with MOS grade and above nitric acid.

5.4.3.3 Hydrochloric acid

1 + 1 (volume fraction), prepare with MOS grade and above hydrochloric acid.

5.4.3.4 Standard solutions

The standard substances used are respectively: lead GBW(E)080537, antimony GBW(E) 080545, arsenic GBW(E)080530, tin GBW(E)080546, bismuth GBW(E)080271, copper GBW(E)080122, cadmium GBW08612, zinc GBW08620, selenium GBW(E)080215, tellurium GBW(E)080548, iron GBW08616, cobalt GBW08613, nickel GBW08618, chromium GBW08614 and manganese GBW(E)080157 or BW series water quality standard solutions.

5.4.4 Instruments

5.4.4.1 Atomic absorption spectrophotometer

It includes four parts: hollow cathode lamp, graphite furnace (or flame atomizer), spectroscopic system and detection and recording system, etc.

5.4.4.2 Sample injector

Automatic sample injector or micro sampler, and sprayer for flame atomization.

5.4.4.3 Polyethylene volumetric flask

The volume is as follows:

Absorb-take a certain amount of sample into the graphite tube (or spray the sample into the flame), determine the absorbance, then, through the working curve, check the element content corresponding to the absorbance.

5.5 Determination of Lead (Pb), Antimony (Sb), Arsenic (As), Tin (Sn), Bismuth (Bi), Copper (Cu), Cadmium (Cd), Zinc (Zn), Selenium (Se), Iron (Fe), Cobalt (Co), Nickel (Ni), Chromium (Cr) and Manganese (Mn) Content - Inductively Coupled Plasma - Atomic Emission Spectrometry

In accordance with the method specified in GB/T 23942, conduct the detection.

5.6 Determination of Iron (Fe) Content - Spectrophotometry

5.6.1 Principle

Ferrous ions react with o-phenanthroline in aqueous solution and generate an orange-red complex, with an absorption peak at 510 nm, and the absorbance of the colored solution is proportional to the concentration of the ferrous ions. The color intensity does not change between pH 3 ~ 9 and is stable for a long time.

5.6.2 Instruments

The instruments required for the test are as follows:

a) Spectrophotometer;

b) 50 mL volumetric flask, 100 mL volumetric flask, pipette.

5.6.3 Reagents and preparation methods for reagents

The reagents and preparation methods for reagents are as follows:

a) Hydrochloric acid 1 + 9 (volume fraction), prepared with analytically pure and above grade hydrochloric acid;

b) Nitric acid: 1 + 1 (volume fraction), prepared with analytically pure and above grade nitric acid;

c) Ammonia water: analytically pure and above grade;

d) 1,10-phenanthroline (2 g/L): prepared in accordance with the stipulations of GB/T 603;

e) Hydroxylamine hydrochloride: analytically pure, 10% solution;

f) Ferrous standard solution (0.001 mg/mL): prepare 0.1 mg/mL ferrous standard solution in accordance with the stipulations of GB/T 602, measure-take 1.00 mL of ferrous standard solution (0.1 mg/mL) in a 100 mL volumetric flask, dilute to the scale and shake it well;

column, suppression column (or suppressor), electrical conductivity detector and data processing, etc. When analyzing anions in water, the separation column is filled with cation exchange resin with a low exchange capacity, and the eluent is diluted alkaline solution. When the eluent and the specimen flow through the separation column, at this moment, the cations in the specimen and the eluent smoothly pass through, and the anions A (A: Cl^-, NO_3^-, etc.) to be tested in the specimen compete with the anions in the eluent to replace the position of hydroxide (OH^-) on the resin ($RN^+ - OH^-$) in the separation column. Flow out from the bottom of the separation column. The separated ions to be tested, together with the eluent, enter the suppression column (or suppressor). In the eluent, sodium hydroxide or sodium bicarbonate, which has a high specific conductance, is transformed into carbonic acid or water with an extremely low ionization degree in the suppression column (or suppressor). The separated anions to be tested smoothly pass through the suppression column (or suppressor), and flow out in the form of acid with a high specific conductance.

The conductivity detector can record the chromatograms of chloride ion and nitrate ion within a dozen of minutes, so as to quantitatively determine the content of each anion in the specimen.

5.7.2 Precautions

The precautions are as follows:

a) The environmental conditions shall be strictly controlled, and the instrument shall be placed in an ultra-clean environment with an adjustable temperature;

b) When analyzing the specimen, after each analysis of the specimen, the water for the blank shall be immediately injected;

c) The chromatograms of anions vary with the instrument parameters and working conditions. For each determination, the standard solution shall be calibrated first to determine the position of the chromatogram of each anion;

d) The samples shall be analyzed as soon as possible. The longer the storage time, the larger the possibility of contamination. After the samples are collected, they shall be stored in a refrigerator at 4 °C;

e) During operation, gloves and masks shall be worn.

During the arbitration of the determination of nitrate (calculated by nitrogen) content, adopt the ion chromatography.

5.7.3 Reagents and preparation methods for reagents

5.7.3.1 Water for the blank

EW-I electronic grade water specified in GB/T 11446.1.

5.7.3.2 Standard stock solutions

The standard stock solutions and the preparation methods are as follows:

a) Chloride standard stock solution (1 mL of solution contains 0.1 mg of Cl^-); accurately weigh-take 0.1650 g of sodium chloride standard substance that has been burned at 500 °C ~ 600 °C to a constant mass, dissolve it in the water for blank and reach a constant volume in a 1 L volumetric flask, dilute to the scale;

b) Nitrate standard stock solution (1 mL of solution contains 0.1 mg of N): accurately weigh-take 0.7786 g of potassium nitrate standard substance (premium grade of purity) that has been burned at 120 °C ~ 130 °C to a constant mass, dissolve it in the water for blank and reach a constant volume in a 1 L volumetric flask, dilute to the scale.

5.7.3.3 Eluent

Weigh-take 6.30 g of sodium bicarbonate (premium grade of purity), dissolve it in for water for blank, then, add 21.20 g of sodium carbonate (premium grade of purity), mix it well, and use the water for blank to dilute it to 1 L. Thus, a stock solution for rinsing that contains 0.075 mol/L of sodium bicarbonate and 0.200 mol/L of sodium carbonate is prepared. Before use, use the water for blank to dilute it by 100 times.

5.7.3.4 Regeneration solution (for suppression column only)

Dissolve 280 mL of concentrated sulfuric acid (MOS grade of purity) in an appropriate amount of the water for blank, and dilute to 4 L. Thus, 1.25 mol/L sulfuric acid stock solution is prepared. Before use, use the water for blank to dilute it by 100 times.

5.7.4 Instruments

The instruments required for the test are as follows:

a) High-efficiency ion chromatograph and accessories: such as: high-efficiency anion separation column, trace analysis column, suppression column (or suppressor), guard column, electrical conductivity detector and recording system, etc.;

b) Analytical balance: with a division value of 0.1 mg;

c) Adjustable polypropylene injector;

d) Glassware with complete specifications.

5.7.5 Determination steps

5.7.5.1 Preparation of standard series of solutions

Accurately measure-take an appropriate amount of standard stock solution and successively step-by-step dilute it, so as to prepare a corresponding series of mixed standard solutions containing chloride ions and nitrate ions.

5.8.3 Determination steps

Measure-take 5 mL of sample into a Nessler colorimetric tube, cool it in an ice bath, then, add 0.4 mL of 10% potassium chloride solution and 0.1 mL of 0.1% diphenylamine sulfuric acid solution, shake it well. Slowly drop-wise add 5 mL of sulfuric acid and shake it well. Place the colorimetric tube in a water bath at 50 °C for 15 min.

Take 0.5 mL of standard potassium nitrate solution, add 4.5 mL of EW-I electronic grade water, and shake it well. After the same treatment as the above-mentioned steps, compare the color, and the color of the specimen shall not be darker than the control solution.

5.9 Determination of Potassium Permanganate Reducing Substance (calculated by O) Content

5.9.1 Principle

Under acidic conditions, substances that can reduce potassium permanganate in the test solution react with potassium permanganate to make the test solution fade. Add a standard amount of potassium permanganate to the sample, if the potassium permanganate cannot completely react, then, the solution will turn pink.

5.9.2 Reagents and preparation methods for reagents

The reagents and preparation methods for reagents are as follows:

a) 20% sulfuric acid solution: prepared in accordance with GB/T 603.

b) Water for blank: EW-I electronic grade water specified in GB/T 11446.1.

c) Potassium permanganate standard solution C (1/5 $KMnO_4$) = 0.01 mol/L: in accordance with the stipulations of GB/T 601, prepare 0.1 mol/L solution, then, use the water for blank to dilute it by 10 times.

5.9.3 Determination steps

Measure-take 200 mL of sample and inject it into a beaker. Add 1.0 mL of 20% sulfuric acid solution and mix it well.

In the above-mentioned acidified test solution, add 5.00 mL of potassium permanganate titration solution [C (1/5 $KMnO_4$) = 0.01 mol/L], mix it well, use a watch glass to cover it, heat to boil and maintain it for 5 min. The pink color of the solution must not completely disappear.

5.10 Determination of Ammonia (calculated by nitrogen) Content

5.10.1 Principle

Ammonia reacts with alkaline potassium mercury iodide to generate a pale yellow to brownish insoluble compound:

$$NH_4^+ + 2(HgI_4)^{2-} + 4OH^- \rightarrow \underset{\text{(brown-yellow precipitate)}}{HgO \cdot Hg(NH_2)I(\text{棕黄色沉淀})} + 7I^- + 3H_2O$$

5.10.2 Reagents and preparation methods for reagents

Alkaline potassium mercury iodide test solution:

---Weigh-take 10 g of potassium iodide, place it in a beaker, add 10 mL of water to dissolve it, then, slowly add saturated aqueous solution of mercuric dichloride, and stir as it is added, until the generated red precipitate no longer dissolves.

---Add 30 g of potassium hydroxide, after dissolving, add 1 mL or more of saturated aqueous solution of mercuric dichloride, transfer it into a 200 mL volumetric flask, and use an appropriate amount of water to dilute it to the scale and shake it well. Transfer it into a 200 mL reagent bottle, let it stand to generate precipitate. During use, take the upper layer of clear solution.

Ammonium chloride solution [0.4 g/L (calculated by nitrogen)]: take 1.529 g of ammonium chloride, place it in a 1,000 mL volumetric flask; add an appropriate amount of the water for blank to dissolve it and dilute to the scale, and shake it well.

5.10.3 Determination steps

Take 50 mL of the sample to a Nessler colorimetric tube, then, add 2 mL of alkaline potassium mercury iodide test solution and let it stand for 15 min.

Take 5 mL of ammonium chloride solution to the Nessler colorimetric tube, add 45 mL of the water for blank and 2 mL of alkaline potassium mercury iodide test solution, and take it as a control solution. The color of the specimen shall not be darker than that of the control solution.

5.11 Determination of Specific Conductance

5.11.1 Principle

At the same temperature, use a conductivity meter to determine the resistance of the sample and the resistance of the potassium chloride solution whose specific conductance is already known.

5.11.2 Instrument

Conductivity meter: equipped with the function of automatic temperature compensation; if it does not have the function of temperature compensation, an “online” heat exchanger can be installed to control the sample being tested at 20 °C ± 1 °C.

5.11.3 Determination steps

In accordance with the instruction manual of the conductivity meter used, and the following steps, conduct the determination: preheating, zero adjustment, calibration, preheating and

Conduct sampling at the water-using terminal, then, conduct the routine inspection. If there is a disqualified item in the inspection results, re-conduct the sampling and inspection. If there is still a disqualified item, then, this batch shall be determined as disqualified.

6.3.3 Frequency of inspection

The routine inspection shall be performed at least once a year. When the water production conditions are changed, routine inspection shall also be performed.

7 Marking, Packaging, Transportation and Storage

7.1 Marking

The storage container of the water for lead acid storage batteries shall be attached with an inspection certificate in a clearly visible position. The inspection certificate shall include the following contents:

a) Product name and serial No. of the standard implemented;

b) Water production organization;

c) Date of production;

d) Weight;

e) Inspector’s signature and inspection date.

7.2 Packaging

The requirements for packaging are as follows:

a) Water for lead acid storage batteries shall be stored in an appropriate container, for example, a container made of glass, hard rubber, polyethylene, polypropylene or other plastic materials. The flexible hoses shall be made of PVC, rubber or polyethylene;

b) Since metal ions will easily be leached from metal containers, containers made of metal shall not be used;

c) Water for lead acid storage batteries shall be stored in an airtight container, because carbon dioxide (CO_2) absorbed from the air will increase the specific conductance of the water;

d) Before use, new container shall be soaked in hydrochloric acid solution (mass fraction: 20%) for 2 d ~ 3 d, then, repeatedly rinsed with the water to be filled, and filled with the water to be filled and soaked for more than 6 h.

7.3 Transportation

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------