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**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

DL/T 1984-2019

**Determination of ammonia content in fly ash of coal-fired boiler.
Spectrophotometric method**

燃煤锅炉飞灰中氨含量的测定 分光光度法

(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009 "Guidelines for Standardization Work Part 1: Standard Structure and Compilation".

Please note that certain contents of this document may involve patents. The issuing agency of this document is not responsible for identifying these patents.

This standard was proposed by the China Electricity Council.

This standard is under the jurisdiction of the Electric Power Industry Environmental Protection Standardization Technical Committee.

The main drafting organizations of this standard. State Grid Zhejiang Electric Power Co., Ltd. Electric Power Research Institute, Guodian Science and Technology Research Institute Co., Ltd., State Grid Shandong Electric Power Company Electric Power Research Institute, Jiangsu Fangtian Electric Power Technology Co., Ltd.

1 Scope

This standard specifies the spectrophotometric method for the determination of ammonia content in fly ash from coal-fired boilers.

This standard applies to the determination of ammonia content in fly ash from coal-fired boilers.

For the methods and procedures specified in this standard, the lower limit of determination is 2.3 mg/kg, and the detection limit is 0.58 mg/kg.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version is suitable Used in this document. For undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods GB/T 8170 Numerical rounding rules and the expression and determination of limit values GB/T 16157 Determination of particulate matter in exhaust from stationary sources and sampling method of gaseous pollutants DL/T 414 Technical Specification for Environmental Monitoring of Thermal Power Plant

3 Principle

Fly ash is added with water and alkalized and distilled, the distillate is absorbed by boric acid solution, and the ammonium ion in the absorption solution reacts with Nessler's reagent to form the absorbance of the complex is measured at a wavelength of 420nm, and the ammonia content in the fly ash is calculated based on the amount of fly ash and the measured amount of ammonium ion.

4 Reagents and materials

Unless otherwise specified, analytically pure chemical reagents that meet national standards are used for analysis. (Suspended section, general rules?)

4.1

Water. Meet the technical requirements of Grade 1 water in GB/T 6682.

4.2

Light magnesium oxide. Heat magnesium oxide (MgO) at 500°C for 0.5h to remove carbonate.

4.3

Nessler's reagent (mercury iodide-potassium iodide-sodium hydroxide (HgI₂-KI-NaOH) solution). Weigh 16.0g sodium hydroxide (NaOH), Dissolve in 50mL water and cool to room temperature. Weigh 7.0g potassium iodide (KI) and 10.0g mercury iodide (HgI₂) and dissolve them in a small amount of water Then, slowly add to 50 mL of sodium hydroxide solution under stirring, and dilute to 100 mL with water. Store in a polyethylene bottle, use the rubber stopper or polyethylene cover is tightly closed and stored away from light. The validity period is 1 year.

4.4

Potassium sodium tartrate solution. 500g/L, weigh 50.0g potassium sodium tartrate

($\text{KNaC}_4\text{H}_6\text{O}_6 \cdot 4\text{H}_2\text{O}$), dissolve in water, heat Boil and dilute to 100 mL after fully cooling.

4.5

Sodium hydroxide solution. 1mol/L, weigh 4 g of sodium hydroxide (NaOH), dissolve in water, and dilute to 100 mL.

4.6

Boric acid solution. 20 g/L, weigh 20 g of boric acid (H_3BO_3), dissolve in water, and dilute to 1L.

4.7 Ammonium ion standard solution

4.7.1

Ammonium ion standard stock solution. 1000mg/L, weigh 2.9654g premium grade pure ammonium chloride (NH_4Cl , 105°C~110°C Dry for 2h), dissolve in water, dilute with water in a 1000mL volumetric flask, and store in polypropylene or high-density polyethylene bottles. The solution Store at 2°C~5°C, valid for 1 month.

4.7.2

Ammonium ion standard working solution. 10mg/L, take 5.00mL ammonium ion standard stock solution (4.7.1) in 500mL volume in the bottle, make the volume constant with water.

5 Apparatus and equipment

5.1

Visible spectrophotometer. equipped with 20mm cuvette.

5.2

Ammonia nitrogen distillation device. consists of a temperature-regulating heater, a 500mL Kjeldahl flask, a nitrogen ball, a condenser, a tube and an Erlenmeyer flask, the end of the condenser tube can be connected with a dropper of appropriate length so that the tip of the outlet is immersed below the surface of the absorption liquid. 500mL can also be used the distillation flask replaces the Kjeldahl flask.

1 Thermostat heater 2 Kjeldahl flask 3 Nitrogen ball 4 Condenser tube 5 Erlenmeyer flask

6.1 Collection and storage of samples

The collection of flue gas fly ash should be carried out in accordance with DL/T 414; the sampling location should be in accordance with GB/T 16157.

To ensure the representativeness of the sample. The collected samples should be sealed and stored in ziplock bags and analyzed within 7 days.

6.2 Sample pre-distillation

Add 50 mL of boric acid solution (4.6) into the Erlenmeyer flask to ensure that the outlet of the condenser is below the liquid surface of the boric acid solution. Weighing Combine 20g uniform fly ash sample, accurate to 0.0001g, transfer it into Kjeldahl flask, add 250mL water, add 0.25g light oxygen Magnesium (4.2) and several glass beads, immediately connect the nitrogen ball and the condenser. Heating distillation, controlling the heating temperature to ensure Kjeldahl the liquid in the flask is in stable boiling without bumping, the distillate rate is generally controlled at about 10mL/min, and the total amount of distillate is not when it is less than 100 mL, stop distillation. Transfer all the liquid in the Erlenmeyer flask to a 250mL volumetric flask and dilute to volume with water. This solution for the stock solution of the sample to be tested, the analysis should be completed within 24 hours.

7.1 Preparation of the instrument

According to the requirements of the spectrophotometer instruction manual, turn on the power and debug the instrument to make it in normal working condition.

7.2 Establishment of calibration curve

In 8 50mL colorimetric tubes, add ammonium ion standard working solution (4.7.2) 0.00, 0.50, 1.00, 2.00, 4.00, 6.00, 8.00 and 10.00mL, add water (4.1) to dilute to the mark, mix well and add 1.0mL potassium sodium tartrate solution (4.4), mix well. Then add 1.0mL Nessler's reagent (4.3) in sequence and mix well. Place for 10 minutes, at a wavelength of 420nm, Use a 20mm cuvette with water as a reference to complete the absorbance measurement within 20 minutes.

Use the mass of ammonium ion (μg) as the abscissa and the absorbance after blank correction as the ordinate to establish a calibration curve.

7.3 Sample analysis

Take 25mL stock solution (6.3) of the sample to be tested into a 50mL colorimetric tube, add a certain amount of sodium hydroxide solution (4.5), adjust the pH value of the water sample is 7~8 (the pH value of the water sample can be tested with pH test paper), diluted with water to the 50mL mark, and then press the calibration curve the same procedure is used to measure absorbance. When the absorbance of the sample to be tested exceeds the calibration curve range, the sample to be tested should be diluted After the determination.

7.4 Blank experiment

Use 20 mL of water (4.1) instead of fly ash, and perform absorbance measurement according to the same analysis procedure as the sample.