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**General rule for determination of nitrogen content by Dumas
combustion method**

杜马斯燃烧法定氮分析方法通则

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part 1: Structure and Drafting Rules of Standardization Documents". Drafting.

Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents.

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Dumas Combustion Method for Nitrogen Analysis

1 Scope

This document establishes general rules for quantitative analysis using the Dumas combustion nitrogen analyzer.

This document applies to the determination of total nitrogen content in samples (excluding gases).

2 Normative references

GB/T 4842

GB/T 4844

GB/T 6379.1

GB/T 6379.2

GB/T 14599

GB/T 14666

GB/T 23938

GB/T 27411

GB/T 27415

3 Terms and Definitions

The terms and definitions defined in GB/T 14666 and the following terms and definitions apply to this document.

3.1

Under high-temperature, oxygen-rich conditions, the sample passes through a combustion tube containing a catalyst to generate nitrogen oxides. After drying and dehydration, it enters a combustion chamber containing a reducing agent.

The reduction tube is reduced to nitrogen gas, and after drying to remove moisture, its

signal intensity is measured using a thermal conductivity detector and compared with the calibration curve to obtain the sample.

An instrument for measuring total nitrogen content.

4 Reagents and Materials

Unless otherwise stated, all reagents used are of analytical grade or higher.

4.1 Test Gas

4.1.1 Oxygen (O₂). It shall meet the requirements of GB/T 14599 pure oxygen or above.

4.1.2 Carbon dioxide (CO₂). It shall meet the requirements of 99.99% or higher mole fraction in GB/T 23938.

4.1.3 Helium (He). It shall meet the requirements of GB/T 4844 for pure helium and above.

4.1.4 Argon (Ar). It shall meet the requirements of GB/T 4842 for pure argon and above.

Note. Select one of the test gases from 4.1.2, 4.1.3, and 4.1.4 according to the instrument instructions.

4.2 Reagents

4.2.1 Diatomaceous earth. CAS No. 61790-53-2.

4.2.2 Magnesium oxide. CAS No. 1309-48-4.

4.2.3 Quartz wool. CAS No. 60676-86-0.

Note. Choose one of 4.2.1, 4.2.2, and 4.2.3 as the adsorbent for the liquid sample according to the actual situation. Diatomaceous earth and magnesium oxide need to be heated to 800°C before use.

Calcine in a resistance furnace for 2 hours, remove and cool to room temperature, then store in a reagent bottle for later use.

4.2.4 Sucrose. CAS No. 57-50-1.

4.2.5 Sodium hydroxide. CAS No. 1310-73-2, small granules.

4.2.6 Sodium tungstate. CAS No. 13472-45-2.

4.2.7 Iron(III) oxide. CAS No. 1317-61-9.

4.2.8 Ferric oxide. CAS No. 1309-37-1.

4.3 Standard reference materials/standard samples

Nitrogen-containing standard substances/standard samples [or glycine (CAS No. 56-40-6),

urea (CAS No. 57-13-6), ethylenediaminetetraacetic acid] (CAS No. 60-00-4), Aspartic acid (CAS No. 56-84-8), etc.).

4.4 Reagent Preparation

Sodium tungstate-magnesium oxide mixed reagent (1 6). Weigh 1g of sodium tungstate (4.2.6), grind it into powder, and add 6g of magnesium oxide powder.

(4.2.2) Mix thoroughly and store in a sealed bottle.

5 Instruments and Equipment

5.1 Dumas combustion nitrogen analyzer. equipped with a thermal conductivity detector and has a calibration function.

5.2 Electronic balance. graduation value 0.01mg.

6.1 Sample Preparation

For solid samples, appropriate pulverization or homogenization methods should be used depending on the product or matrix type to ensure that the minimum sample size is representative.

For liquid samples, they should be thoroughly mixed before weighing.

6.2 Maximum sample injection volume

The total nitrogen content of the maximum sample injection volume should not exceed the nitrogen content design capacity of the Dumas combustion nitrogen analyzer.

7.1 Instrument Preparation

Ensure there is sufficient working gas for continuous operation, and preheat the instrument to a stable temperature according to the instrument manual.