

Determination of Nitrogen Content in Corn Starch and Cassava Starch with Water-cooling-Free Fully Automatic Kjeldahl Nitrogen Analyzer

Introduction

Corn starch and cassava starch are two starch varieties widely used in industrial production and daily life. Due to differences in raw material sources and processing techniques, their protein (nitrogen) contents show significant variation. Accurate determination of nitrogen content in these two starches, and subsequent conversion to protein content, is a key step in assessing starch purity, ensuring suitability for subsequent product processing, and controlling product quality. This experiment strictly followed the national standard GB 5009.5-2025 *National Food Safety Standard – Determination of Protein in Foods*, using the Kjeldahl method to precisely measure the nitrogen content in the two starch samples.

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Telephone: +86-757-83277860 Email: info@rogetscientific.com Website: www.rogetscientific.com
Address: No. 8, Zhixin Road, Dajiao Town, Nansha District, Guangzhou City, Guangdong, China

Materials and Equipment

Equipment



- Instrument: N810 Water- cooling-free Fully Automatic Kjeldahl Nitrogen Analyzer.
- Digestion equipment: NDS 20 graphite digestion furnace (20 place).
- Weighing equipment: analytical balance (0.1 mg precision).

Reagents and Materials

- Samples: solid powders of corn starch and cassava starch.
- Basic reagents: sulfuric acid (analytical grade); 400 g/L sodium hydroxide solution; 20 g/L boric acid solution.
- Titration reagent: 0.05 mol/L hydrochloric acid standard titration solution.
- Indicator and catalyst: bromocresol green- methyl red mixed indicator; mixed catalyst (composition: 3 g K_2SO_4, 0.2 g $CuSO_4$) or ready- to- use catalyst tablets.

Methods and Procedures

Sample Preparation and Blank Control

Accurately weigh 2 g of powdered starch sample (precise to 0.1 mg). Wrap the sample in weighing paper and place the entire package into a digestion tube. Then add 2 mixed

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catalyst tablets (or 3.2 g of mixed catalyst powder) and carefully add 10 mL of concentrated sulfuric acid along the inner wall of the digestion tube.

Note: To ensure consistency in the digestion system between the sample and the sample blank, add a piece of weighing paper of the same specification to the sample blank digestion tube and digest it simultaneously.

Sample Digestion Procedure

Place the digestion tubes containing samples and reagents onto the digester, covering with the waste exhaust device, or place a bent neck funnel on the mouth of the digestion tube. The digester uses a multi- steps temperature program. The specific digestion parameters are shown in Table 1:

Table 1: Digester parameter settings

Step	Target Temp	Digestion Time
1	250℃	40min
2	420℃	120min

Instrument Preheating and Analysis

After the digestion program is completed, allow the tubes to cool naturally to room temperature. Before formal testing, check that the reagents in the bottles are sufficient, then perform the instrument preheating procedure three times. After preheating, transfer the digestion tubes to the instrument for testing. The operating parameters of the Kjeldahl analyzer are detailed in Table 2:

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Table 2: N810 Kjeldahl analyzer test parameter settings

Parameter	Value	Parameter	Value
Boric acid	30mL	Distillation time	5min
Dilution water	30mL	Protein factor	/
Alkali	40mL	Concentration of titration acid	0.0500mol/L

Results and Discussion

After digestion, distillation, and titration procedures described above, the detailed experimental data obtained are shown in Table 3:

Table 3: Nitrogen content of corn starch and cassava starch

Sample Name	Weight (g)	Blank volume (mL)	Titration volume (mL)	Nitrogen content (g/100g)	Average (g/100g)
Corn starch	2.008	0.0752	1.7042	0.0568	0.0582
	2.0008		1.7794	0.0597	
	2.0082		1.7394	0.0581	
Cassava starch	2.0013		8.5264	0.2958	0.2936
	2.0014		8.3926	0.2911	
	2.0081		8.4984	0.2938	

As shown in the table above, three parallel measurements were performed for each starch sample to ensure data rigour.

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Conclusion

The results determined using the N810 water- free fully automatic Kjeldahl analyzer showed that the average nitrogen contents of the corn starch and cassava starch samples were 0.0590 g/100g and 0.2936 g/100g, respectively. Using the conventional protein conversion factor (usually 6.25) for food analysis, the crude protein content of this batch of corn starch is approximately 0.37%, while that of cassava starch is approximately 1.84%. These data quantitatively illustrate the macroscopic differences in impurity distribution and purity between the two starch samples.

References and Standards

- GB 5009.5-2025 *National Food Safety Standard – Determination of Protein in Foods*
- GB/T 22427.10-2008 *Starch and derived products – Determination of nitrogen content*
- GB/T 33862-2017 *Full (semi) automatic Kjeldahl analyzer*
- ISO 3188:1978 *Starches and derived products — Determination of nitrogen content by the Kjeldahl method — Titrimetric method*
- AOAC 920.87 *Protein (Total) in Flour*
- AOAC 2001.11 *Protein (Crude) in Animal Feed, Forage (Plant Tissue), Grain, and Oilseeds*

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