



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

GB 5009.277-2016

**National food safety standard - Determination of sodium
hydrogen diacetate in foods**

食品安全国家标准 食品中双乙酸钠的测定

(English Translation)

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Foreword

This Standard replaces GB/T 23383-2009, Determination of sodium hydrogen diacetate in foods - High performance liquid chromatography method.

Compared with GB/T 23383-2009, the major technical changes of this Standard are as follows.

-- it changes the name of the standard into "National food safety standard -

Determination of sodium hydrogen diacetate in foods";

-- it changes the applicable scope of the standard.

National food safety standard -

Determination of sodium hydrogen diacetate in foods

1 Scope

This Standard specifies the liquid chromatography for the determination of sodium hydrogen diacetate in foods.

This Standard applies to the determination of sodium hydrogen diacetate in dried tofu, dried tofu products, unprocessed grains, tapioca pearls, pastries, premade meat products, cooked meat products, cooked aquatic products (edible directly), solid compound seasonings and puffed foods. This Standard does not apply to the

determination of seasonings, liquid compound seasonings and food added with acetic acid.

2 Principle

Sodium hydrogen diacetate in sample is acidized transforming into acetic acid; it is extracted on an ultrasonic water bath or distilled by steam; then it is collected before adjustment of pH, determined through high performance liquid chromatography and quantitated using the external method.

3 Reagents and materials

Unless specified otherwise, all reagents used in this Method are analytically pure; the water used for chromatography is grade one water as specified in GB/T 6682 and the water used for others is grade three water as specified in GB/T 6682.

3.2.1 Phosphoric acid solution (1 mol/L). add 53.5 mL of phosphoric acid into 500 mL

of water; after mixing up, add water to make up to 1,000 mL.

3.2.2 Diammonium hydrogen phosphate (1.5 g/L). weigh 1.5 g of diammonium hydrogen phosphate; add water to dissolve and make up to 1,000 mL.

3.3 Standard substance

Sodium hydrogen diacetate standard substance $[(\text{CH}_3\text{COO})_2\text{HNa}]$, CAS no.. 126-96-5].
purity $\geq 99\%$.

3.4.1 Standard stock solution (10 mg/mL). Accurately weigh 1 g (accurate to 0.000 1

g) of sodium hydrogen diacetate standard substance; use water to make up to 100 mL.
The standard stock solution is stored in a refrigerator at $0^\circ\text{C} \sim 4^\circ\text{C}$; the storage life is

3.4.2 Standard working solution. accurately absorb 5.0 mL of standard stock solution

to pour into a volumetric flask of 50 mL; use water to dilute to scale; prepare a standard working solution of concentration 1.0 mg/mL. The standard working solution is stored

in a refrigerator at 0°C ~ 4°C; the storage life is 1 month.

4.1 High performance liquid chromatograph (HPLC). equipped with an ultraviolet detector or a diode array detector.

5.1 Sample preparation

In case of solid sample, take 500 g before using a blender to mix up for use; or in case of liquid sample, shake up for use.

5.2.1 The distillation method

Accurately weigh 25 g (accurate to 0.01 g) of sample; place in a distillation flask of 500 mL; add 100 mL of water; use 50 mL of water to rinse the container; transfer to the distillation flask; add 20 mL of phosphoric acid solution (1 mol/L); 2 drops ~ 3 drops of silicone oil; carry out steam distillation; place a volumetric flask of 250 mL in the water bath as the absorption liquid receiver; take out when about 240 mL is distilled; place aside for 30 min at room temperature; use phosphoric acid solution of 1 mol/L to adjust the pH to be about 3; add water to make up; shake up; after filtering through aqueous phase micropore filter membranes of 0.45 µm, carry out liquid chromatographic determination.

5.2.2 The direct extraction method (apply only to steamed buns and steamed twisted rolls)

Accurately weigh 5 g (accurate to 0.01 g) of sample to place into a beaker of 100 mL; add 20 mL of water; add 0.5 mL of phosphoric acid solution of 1 mol/L; mix up; after ultrasonic extraction for 10 min, use phosphoric acid solution (1 mol/L) to adjust the pH to about 3; transfer sample to a volumetric flask of 50 mL; use water to make up to scale; shake up. Transfer all sample into a stoppered plastic centrifugal tube of 50 mL; carry out centrifugation for 10 min at not lower than 4,000 r/min; take liquid supernatant; after filtering through aqueous phase micropore filter membranes of 0.45 µm, carry out liquid chromatographic determination.

5.3 Apparatus reference conditions

a) chromatographic column. C18 column, column length 250 mm, internal diameter

4.6 mm and grain diameter 5 μm , or equivalent;

b) column temperature. 25°C;

c) moving phase. use phosphoric acid solution (1 mol/L) to adjust the pH of diammonium hydrogen phosphate solution (1.5 g/L) to 2.7 ~ 3.5 (prepare immediately prior to use) and filter using aqueous phase micropore filter membranes of 0.45 μm ;

d) flow rate. 1.0 mL/min;

e) wavelength. 214 nm;

f) volume of sample. 20 μL ;

g) reference conditions for the cleaning of chromatographic column. after test is completed, use methanol 10% to clean for 1 h; then use methanol 100% to clean for 1 h.

5.4.1 The distillation method

Accurately absorb 1.0 mL, 2.5 mL, 5.0 mL, 7.5 mL, 10.0 mL and 12.5 mL of sodium hydrogen diacetate standard stock solution to pour into distillation flasks of 500 mL; other operations are as in 5.2.1. The final concentrations of sodium hydrogen diacetate standard solution are respectively 0.04 mg/mL, 0.1 mg/mL, 0.2 mg/mL, 0.3 mg/mL, 0.4 mg/mL and 0.5 mg/mL. Inject them into the liquid chromatograph after filtering through aqueous phase micropore filter membranes of 0.45 μm ; measure the corresponding peak areas; plot the standard curve using the concentrations of standard working solutions as the abscissa and the peak areas as the ordinate.

5.4.2 The direct extraction method

Accurately absorb 0.5 mL, 1.0 mL, 2.0 mL, 3.0 mL, 4.0 mL and 5.0 mL of standard working solution to pour into volumetric flasks of 10 mL; add 0.2 mL of phosphoric acid solution (1 mol/L) respectively; use water to make up to 10 mL. The concentrations of