

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

SN/T 5112-2019

**Determination of propylene glycol in edible animals and feeds
for import and export -- GC Method and GC-MS method**

进出口食用动物、饲料丙二醇含量测定 气相色谱法和气相色谱-质谱法

(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in GB 1.1-2009.

This standard is proposed and managed by the General Administration of Customs of the People's Republic of China.

This standard was drafted. Gongbei Customs of the People's Republic of China, Lanzhou Customs of the People's Republic of China.

1 Scope

This standard specifies the determination methods of 1,2-propanediol and 1,3-propanediol in food animals and feeds.

This standard applies to the determination of 1,2-propanediol and 1,3-propanediol in the serum, urine, milk and feed of food animals.

2 Normative references

The following documents are essential for the application of this document. For dated references, only the dated version applies to this document.

file. For undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods First method gas chromatography

3 Method summary

The propylene glycol in the sample was extracted with acetonitrile, pre-treated and purified, and then determined by gas chromatography. The retention time was qualitative and the peak area external standard method Quantitative.

4 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade, and water is first-grade water as specified in GB/T 6682.

4.1

Acetonitrile (CH_3CN), chromatographically pure.

4.2

Ethanol ($\text{C}_2\text{H}_5\text{OH}$), chromatographically pure.

4.3 1,2-Propanediol ($\text{CH}_2\text{OHCHOHCH}_3$). Standard, CASN O... 57-55-6, with a purity of 99.9%.

4.4 1,3-propanediol ($\text{CH}_2\text{OHCH}_2\text{CH}_2\text{OH}$). Standard product, CASN O... 504-63-2, purity $\geq 98.0\%$.

4.5 1,2-propanediol standard stock solution (10.0 mg/mL). Weigh accurately 250 mg (accurate to 0.1 mg) of 1,2-propanediol standard to in a 25mL volumetric flask, add ethanol to dissolve and dilute to the mark, and shake well.

4.6 1,3-propanediol standard stock solution (10.0mg/mL). Weigh accurately 250mg

(accurate to 0.1mg) 1,3-propanediol standard product to in a 25mL volumetric flask, add ethanol to dissolve and dilute to the mark, and shake well.

4.7 1,2-propanediol and 1,3-propanediol standard use solution (1,000mg/mL).

1,2-propanediol and 1,3-propanediol which were accurately removed respectively the standard stock solution is from 5,000mL to 50mL volumetric flask.

4.8 1,2-propanediol and 1,3-propanediol standard working solution. 1,000 mg/mL mixed standard solution was gradually diluted with ethanol to obtain the concentrations of 1,2-propanediol and 1,3-propanediol were 1,000 µg/mL, 2000 µg/mL, 5.0 µg/mL, 10.0 µg/mL, 20.0 µg/mL, 50 µg/mL, 100µg/mL standard working solution series. 4.9 Anhydrous magnesium sulfate.

4.10

Purification tube. 2mL centrifuge tube with stopper, containing 50mg PSA powder, 100mgC18 powder and 100mgSO4 powder.

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4.11

Microporous membrane. 0.45 µm, organic phase. 4.12 Carrier gas. high purity nitrogen (99.99%).

4.13

Auxiliary gas. hydrogen (99.99%), dry air (99.99%).

5 Instruments and equipment

5.1

Gas chromatograph (GC). equipped with hydrogen flame ionization detector (FID).

5.2

Analytical balance. Sensitivity is 0.0001g and 0.0001g, respectively.

5.3 Sample crusher

5.4 Nitrogen blowing concentration station.

5.5 Vortex the mixer.

5.6 Ultrasonic water bath.

5.7 High-speed centrifuge. Rotation speed ≥ 1000 r/min.

5.8 Scale test tube. 5mL.

5.9 Colorimetric tube with stopper. 10mL.

5.10 Centrifuge tube with stopper. 15mL.

6.1.1 Serum matrix

Weigh 2g (accurate to 0.01g) sample into a 10mL colorimetric tube, slowly add acetonitrile to the nearest scale under vortex, and cover with ultrasonic extraction 5min, take it out to room temperature, make up to volume with acetonitrile, and mix well. Transfer this extract to a 15mL stoppered centrifuge tube at 10,000r/min.

Centrifuge for 3 min. Accurately transfer 5.0mL of the supernatant to a 5mL graduated test tube, and concentrate it to about 0.5mL at 50 ° C in a nitrogen blower.

Reconstitute with ethanol, make up to volume, mix well, pass 0.45 µm organic phase filter membrane, and bottle until ready for use.

6.1.2 Urine matrix

Weigh 2g (accurate to 0.01g) sample into a 10mL colorimetric tube, slowly add acetonitrile to the nearest scale under vortex, and cover with ultrasonic extraction 5min, take it out to room temperature, make up to volume with acetonitrile, and mix well. Add about 1.5g of anhydrous magnesium sulfate to the colorimetric tube, cover with a vortex, and the extract was transferred to a 15 mL stoppered centrifuge tube, centrifuged at 1,000 r/min for 3 min, and the supernatant was passed through an organic phase filter membrane of 0.45 µm, and bottled for use on the machine.

6.1.3 Milk base

Weigh 2g (accurate to 0.01g) sample into a 10mL colorimetric tube, slowly add acetonitrile to the nearest scale under vortex, and cover with ultrasonic extraction 5min, take it out to room temperature, make up to volume with acetonitrile, and mix well. Transfer 1.0 mL of the supernatant to the purification tube (4.9), cover with a vortex, and Centrifuge at 10,000 r/min for 3 min, take the supernatant and pass it through an organic phase filter membrane of 0.45 µm, and bottle it for loading onto the machine.

6.1.4 Feed substrate

The sample was pulverized with a pulverizer, 1g (accurate to 0.0001g) of feed was weighed into a 15mL centrifuge tube with a stopper, and 10.0mL of B was accurately added.

Nitrile, cap and shake to mix well, extract 15 min by ultrasonic, take it out to room temperature, and centrifuge at 10,000 r/min for 3 min. Pipette supernatant in a 1000mL to 10mL colorimetric tube, make up to volume with ethanol and mix well. Take the diluted solution through a 0.45µm organic phase filter and bottle it for use on the machine.

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