

**Alcohol Determination of Sanitizer Gel
in accordance with USP<611>**

United States Pharmacopeia (USP) General Chapters <611> ALCOHOL DETERMINATION stipulates two analytical methods for quantitating ethanol: one with distillation and the other by gas chromatography. The latter (i.e. USP <611> Method II) further gives an option of either using a packed column (Method II a) or a capillary column (Method II b).

This article introduces a quantitative analysis of ethanol in alcohol-based sanitizer gel according to USP <611> Method II b.

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Preparation of Standard Solution and Sample Solution

5 mL each of 2 % (v/v) ethanol*1 and 2 % (v/v) acetonitrile*1 (internal standard), both in water, were pipetted into a 25 mL volumetric flask, made up to volume with water and vortex to prepare a 0.4 % (v/v) standard solution.

As a sample solution, a commercially available sanitizer gel (ca. 80 % (v/v)) was first diluted with water to ca. 2 % (v/v) ethanol. To further bring down the concentration to ca. 0.4 % (v/v), 5 mL of each of the prepared ca. 2 % (v/v) sample and 2 % (v/v) acetonitrile were aliquoted into 25 mL volumetric flask and mixture was made up to volume with water.

*1 USP<611> specifies the use of USP Alcohol Determination-Alcohol RS (2 % (v/v) ethanol) and USP Alcohol Determination-Acetonitrile RS (2 % (v/v) acetonitrile) to prepare the standard solution.

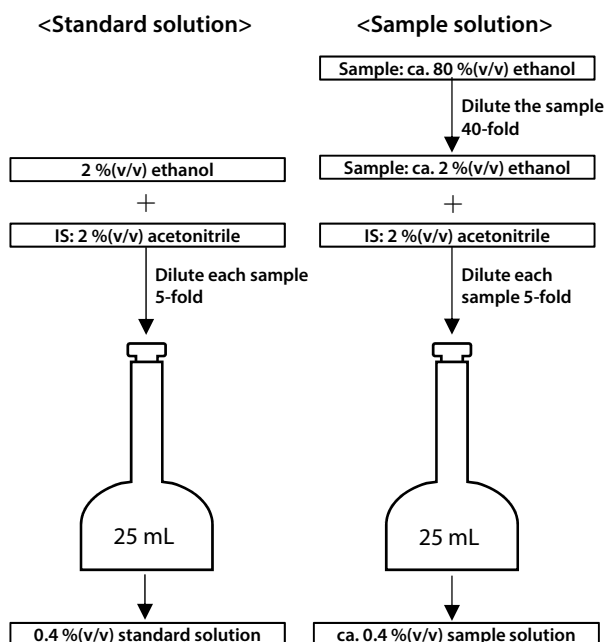


Fig. 1 Sample Preparation Method

Analysis Conditions

Using the gas chromatograph Nexis™ GC-2030, ethanol in the standard solution and the sample solution were quantitated according to USP<611>ALCOHOL DETERMINATION Method IIb. The instrument configuration and analysis conditions for the this experiment are listed below in Table 1.

Table 1 Instrument Configuration and Analysis Conditions

Model	: Nexis GC-2030 + AOC-20i Plus
Detector	: FID-2030 flame ionization detector
Column	: SH-Rtx™-624 (30 m × 0.53 mm I.D., d.f.= 3 μm)
Column Temperature	: 50 °C (5 min) – 10 °C/min – 200 °C (4 min) Total 24 mins
Injection Temperature	: 210 °C
Injection Mode	: Split
Split Ratio	: 1: 5
Carrier Gas Controller	: Linear velocity (He)
Linear Velocity	: 34 cm/sec
Detector Temperature	: 280 °C
FID H ₂ Flow Rate	: 32 mL/min
FID Make up Flow Rate	: 24 mL/min (He)
FID Air Flow Rate	: 200 mL/min
Injection Volume	: 0.2 μL
Syringe	: Elastic Syringe, AOC (P/N: 221-49548)*2

*2 When samples in aqueous solution are analyzed with a standard syringe for AOC-20i Plus, the plunger motion may become dull during analysis, which affects repeatability. Using an elastic syringe for AOC (P/N: 221-49548) equipped with a plunger made of titanium enables stable sample introduction.

In this analysis, a glass insert was specifically configured as shown in Fig.2 to meet the requirements for the system suitability test(SST) in USP<611>. 20 mg of deactivated glass wool was packed into a split glass insert at a position 20 mm from the top. Increasing the amount of wool compared to the default amount of 10 mg and placing the wool slightly (i.e. 2 mm) above the default position (i.e. 22 mm from the top) improved reproducibility.

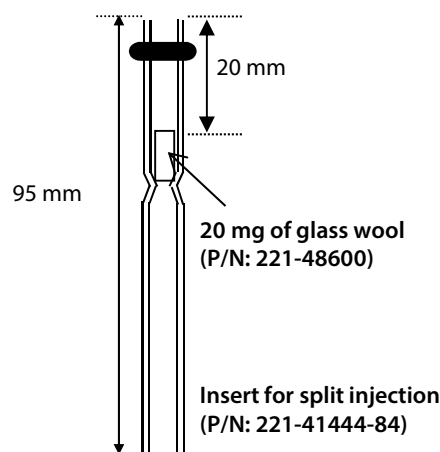


Fig. 2 Position and Quantity of Wool in the Insert

■ Chromatogram and Calibration Curve of the Standard Solution

The chromatogram and calibration curve of the standard solution are shown in Fig. 3 and 4. The SST results of the standard solution are summarized in Table 2.

The SST criteria include the following:

- The resolution factor, R, between alcohol and the internal standard be not less than 4.
- The tailing factor of the alcohol peak be not greater than 2.0.
- Six replicate injections of the standard solution show a relative standard deviation of not more than 4.0 % in the ratio of the peak of alcohol to the peak of the internal standard.

The results obtained with the standard solution satisfied all three SST criteria. The requirement for reproducibility (i.e. 4 %) was easily met with the RSD of 0.4 %.

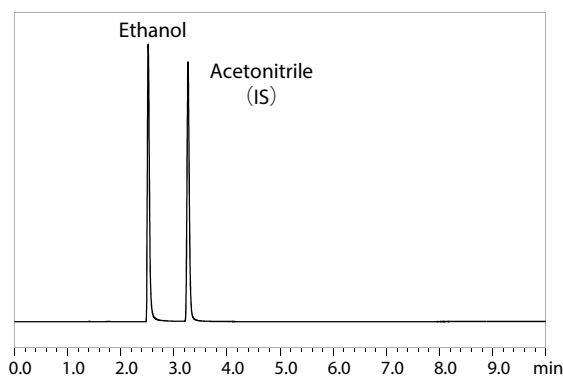


Fig. 3 Chromatograms of Standard Solution

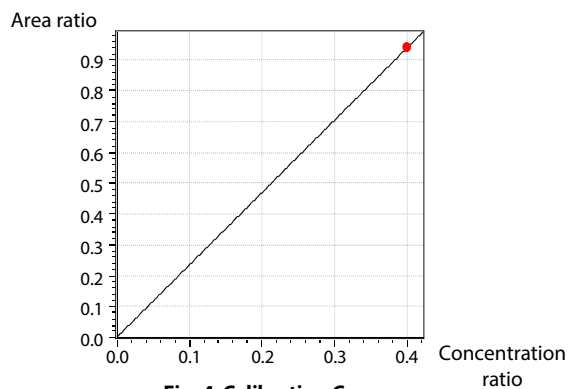


Fig. 4 Calibration Curve

Table 2 System Suitability of Standard Solution (n=6)

Compound	Peak area	Area ratio	Area ratio %RSD	Symmetry (tailing) factor	Resolution (USP)
Ethanol	627440	0.941074	0.405	1.467	---
Acetonitrile (IS)	666723	---	1.255	10.265	10.265

In Table 2, the items specified in the system suitability test are indicated in red.

Note: The values shown are reference values, not guaranteed values.

■ Chromatogram of Sample Solution and Quantitative Result for Ethanol

The chromatogram of the sample solution is shown in Fig. 5, and the quantitative results and repeatability (n=3) are listed in Table 3.

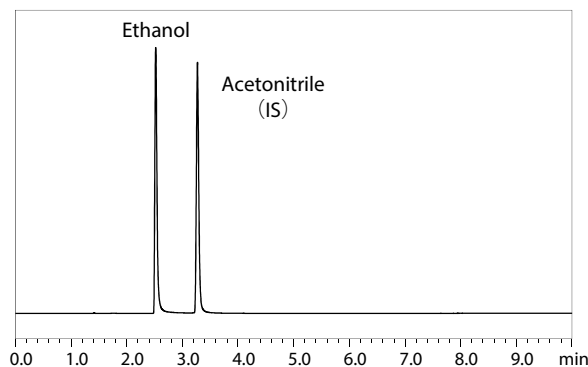


Fig. 5 Chromatograms of Sample Solution

Table 3 Ethanol Quantitative Values and Repeatability (n=3)

	Area ratio	Quantitative value (%)
Data 1	0.929279	78.997
Data 2	0.925411	78.668
Data 3	0.929298	78.998
Average	0.927996	78.888
%RSD	0.241	0.241

Note: The values shown are reference values and not intended to be guaranteed values.

■ Conclusion

Alcohol concentration in sanitizer gel was determined using a capillary column in compliance with USP <611> Method IIb.

The SST was conducted with a standard solution and satisfied with the resolution of 10.3 (cf. > 4 as a requirement) between ethanol and acetonitrile, the tailing factor of 1.5 (cf. < 2 as a requirement) and the repeatability of 0.4 % RSD (cf. 4 % limit).

The repeatability remained well even with a sample solution, proving the robustness of the gas chromatograph Nexis GC-2030.

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Note: The experiment in this article was performed based on the current version of USP-NF as of April 24, 2020.

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