

Analysis of Methanol in Ethanol-based Sanitizer

A variety of analytical methods have been developed, validated and put in use for analysis of methanol in ethanol-based sanitizers across the world.

In Japan, for example, methods using gas chromatography (GC) are standardized by the Japanese Industrial Standards (JIS) K 8101 : 2006, the Japan Alcohol Association Standard JAAS001 : 2012 and the Predetermined Analysis Method of the National Tax Agency Item 11. With a recent surge in demand for ethanol as a sanitizer, test methods standardized by these agencies can also be applied to the quantitation of methanol in ethanol-based sanitizers.

In this article, methanol in ethanol-based liquid and gel sanitizers was analyzed. The concentration of standards was 630 ppm to match the maximum residue level set by the U.S. Food and Drug Administration (FDA). The results obtained in this article were solely based on the comparison between the sample and standard chromatograms.

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* The actual quantitation methods vary among the three organizations. The details are to be found in each monograph listed in this article.

Preparation of Standards and Samples

Methanol standard solutions were prepared in reference to JIS K 8101 : 2006, JAAS001 : 2012 and the National Tax Agency Item 11. The details are discussed below.

To represent the liquid and gel types of samples, the following three samples were employed in this study: two ethanol-based sanitizers, one commercially available off a store shelf and the other produced by a sake brewer both to represent the liquid form of sample and an ethanol-based sanitizer gel to represent the gel form.

Table 1 shows the preparation methods for standards and samples.

Japanese Industrial Standards (JIS) and Japan Alcohol Association Standard (JAAS)

Methanol standard solutions were prepared by diluting a neat solution to 16 and 630 ppm with 99.5 % (v/v) ethanol.

For samples, the liquid sanitizers were used undiluted while the gel sanitizer was diluted 40 times with 99.5 % (v/v) ethanol prior to analysis.

Predetermined Analysis Method of National Tax Agency

Methanol standard solutions were prepared by diluting a neat solution to 630 ppm with 80 % (v/v) (aq) ethanol and to 63 ppm with 8 % (v/v) (aq) ethanol.

For samples, the liquid sanitizers were used undiluted while the gel sanitizer was diluted 10 times with water prior to analysis.

Table 1 Sample Preparation Methods

	Liquid		Gel	
	Standard	Sample	Standard	Sample
Japanese Industrial Standards JIS K8101	630 ppm MeOH (diluted with 99.5 % EtOH)	Not diluted	16 ppm MeOH (diluted with 99.5 % EtOH)	Diluted 40x with 99.5 % EtOH
Japan Alcohol Association Standard JAAS001				
National Tax Agency Predetermined Analysis Method Item 11	630 ppm MeOH (diluted with 80 % EtOH)		63 ppm MeOH (diluted with 8 % EtOH)	Diluted 10x with water

Japanese Industrial Standards

The prepared samples were analyzed according to JIS K 8101 : 2006 with a Shimadzu Nexis™ GC-2030 gas chromatograph, Table 2 lists the instrument configuration and the analytical conditions employed in this experiment.

Fig. 1 is the overlaid chromatograms of the 630 ppm standard solution and the two liquid sanitizers (i.e. one off a store shelf and the other produced by a sake brewer).

Fig. 2, on the other hand, shows chromatograms for the gel sanitizer and the 16 ppm standard solution.

Table 2 Instrument Configuration and Analytical Conditions

Model	: Nexis GC-2030 + AOC™-20i Plus
Detector	: Flame ionization detector FID-2030
Analytical Column	: SH-624 (30 m × 0.53 mm I.D., d.f. = 3 μm) *1
Column Temp.	: 40 °C (5 mins) – 5 °C/min – 90 °C (2 mins), total 17 mins
Injection Temp.	: 150 °C
Injection Mode	: Direct Injection (WBI-2030 direct injection inlet)
Carrier Gas	: He
Carrier Gas Controller	: Constant Column Flow Rate (3.00 mL/min)
Detector Temp.	: 150 °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

*1 P/N: 221-76158-30

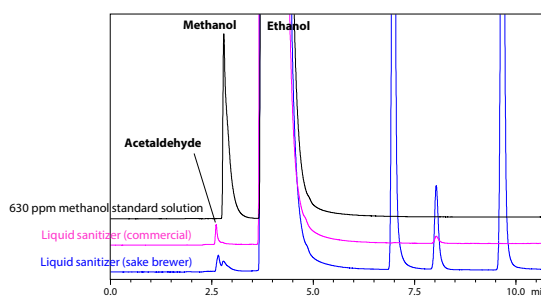


Fig. 1 Chromatograms of Liquid Sanitizers

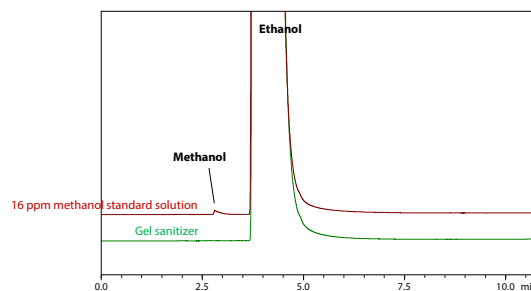


Fig. 2 Chromatograms of Gel Sanitizer

Japan Alcohol Association Standard

The prepared samples were measured according to JAAS001 : 2012 with a Shimadzu Nexis GC-2030 gas chromatograph. Table 3 lists the instrument configuration and the analytical conditions employed in this experiment.

Fig. 3 is the overlaid chromatograms of the 630 ppm standard solution and the two liquid sanitizers (i.e. one off a store shelf and the other produced by a sake brewer).

Fig. 4, on the other hand, shows chromatograms of the gel sanitizer and its standards.

Table 3 Instrument Configuration and Analytical Conditions

Model	: Nexis GC-2030 + AOC-20i Plus
Detector	: Flame ionization detector FID-2030
Analytical Column	: SH-Q-BOND (30 m × 0.53 mm I.D., d.f. = 20 μm) *1
Column Temp.	: 40 °C – 5 °C/min – 200 °C (5 mins), total 37 mins
Injection Temp.	: 200 °C
Injection Mode	: Direct injection (WBI-2030 direct injection inlet)
Carrier Gas	: He
Carrier Gas Controller	: Constant Column Flow Rate (15 mL/min)
Detector Temp.	: 200 °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	: 1.0 μL

*1 P/N: 221-75765-30

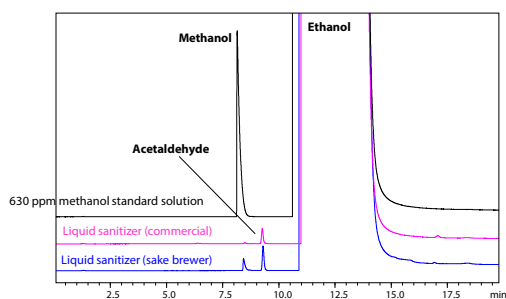


Fig. 3 Chromatograms of Liquid Sanitizers

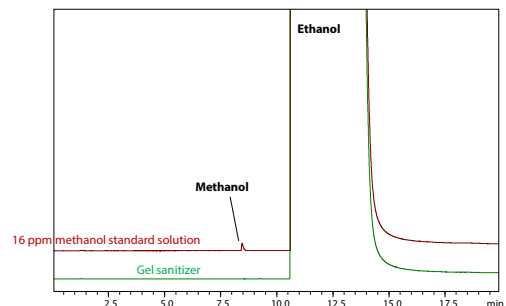


Fig. 4 Chromatogram of Gel Sanitizer

National Tax Agency Analysis Method

With a Shimadzu Nexis GC-2030 gas chromatograph, the prepared samples were analyzed according to the Predetermined Analysis Method of the National Tax Agency Item 11. Table 4 lists the instrument configuration and the analytical conditions employed in this experiment.

Fig. 5 is the overlaid chromatograms of the 630 ppm standard solution and the two liquid sanitizers (i.e. one off a store shelf and the other produced by a sake brewer).

Fig. 6, on the other hand, shows chromatograms of the gel sanitizer and its standards.

Table 4 Instrument Configuration and Analytical Conditions

Model	: Nexis GC-2030 + AOC-20i Plus
Detector	: Flame ionization detector FID-2030
Analytical Column	: SH-WAX (30 m × 0.32 mm I.D., d.f. = 0.50 μm) *1
Column Temp.	: 40 °C (5 mins) – 10 °C/min – 100 °C (2 mins), total 13 mins
Injection Temp.	: 250 °C
Injection Mode	: Split
Split Ratio	: 100
Carrier Gas	: He
Carrier Gas Controller	: Constant Column Flow Rate (1 mL/min)
Detector Temp.	: 250 °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	: 1.0 μL

*1 P/N: 221-75896-30

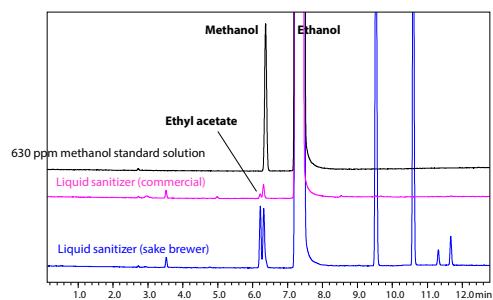


Fig. 5 Chromatograms of Liquid Sanitizers

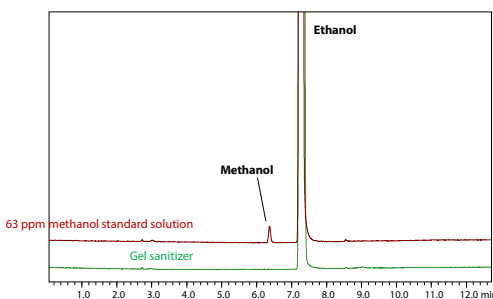


Fig. 6 Chromatogram of Gel Sanitizer

Conclusion

Methanol in ethanol-based liquid and gel sanitizers was analyzed with a Shimadzu Nexis GC-2030 gas chromatograph according the following three standardized methods: the Japanese Industrial Standards JIS K 8101 : 2006, the Japan Alcohol Association Standard JAAS001 : 2012, and the Predetermined Analysis Method of the National Tax Agency Item 11.

All samples analyzed in this experiment had methanol concentration below 630 ppm set as the maximum residue level set by the FDA.

Shimadzu Nexis GC-2030 gas chromatograph is thus considered an excellent instrument to quantitate methanol concentration in ethanol-based liquid and gel sanitizers.

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