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Gas Chromatograph Nexis™ GC-2060

Application News

The Analysis of Volatile Substances and Methanol in Spirit Drinks Using Nexis GC-2060

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User Benefits

- Nexis GC-2060 was validated for Commission Regulation (EC) No 2870/2000 for the official control of spirit drinks.
- Nexis GC-2060 provides excellent separation, high linearity, and low RSDs for reliable routine throughput.
- The Multi-Mode Injection Unit (MMI) demonstrated excellent performance to meet EC 2870/2000 criteria and significantly reduces maintenance-related downtime.

■ Introduction

The analysis of volatile substances and methanol in spirit drinks is essential for regulatory compliance, quality control and verification of product authenticity. Commission Regulation (EC) No 2870/2000 establishes Community reference methods to ensure harmonized results during official controls and in disputes; gas chromatography with flame ionization detection (GC-FID) is the specified reference technique for quantifying key volatile congeners (aldehydes, higher alcohols, ethyl acetate) and methanol.[1] This application note describes a GC-FID method for these analytes on a Shimadzu Nexis GC-2060, implemented to comply with the Regulation's requirements and suitable for routine and official-control testing across a range of spirit drink categories.

This application note also evaluates Shimadzu Multi-Mode Injection Unit (MMI) operated in split/splitless mode to verify its performance to meet the requirements of the method.

Fig. 1 Nexis™ GC-2060 with AOC-30i

■ Instrumentation and Analysis Conditions

A GC-FID system, Nexis GC-2060 (Shimadzu Corporation) was employed in this work. The details of the system and analytical conditions for this analysis method are shown in Table 1.

Table 1. System Configurations and Analysis Conditions

System Configurations	
GC system	Nexis GC-2060
Injection Port	Multi-Mode Injection Unit (MMI)
Auto sampler	AOC-30i
Column	SH-BAC Plus 1; 30 m x 0.32 mm x 1.80 µm (P/N: 227-36260-01), SH-502.2; 30 m x 0.25 mm x 1.40 µm (P/N: 227-36341-04) Both columns are connected in series.
AOC-30i Parameters	
Sample Volume	0.5 µL
Gas Chromatograph Parameters	
Injection Mode	Split mode (Split Ratio: 20)
Carrier Gas	Nitrogen
Flow Control Mode	Linear velocity (25 cm/s)
Column Temperature Program	60 °C (5 min) → 10 °C/min to 80 °C (18 min) → 30 °C/min to 200 °C (9 min)
FID Parameter	
Detector Temperature	240 °C

MMI enables rapid cooling, reducing the temperature from 230°C to 70°C in 7 minutes, compared with 32.2 minutes for conventional SPL in this system. With the compressed air cooling option, MMI can reduce the cooling time further to just 2.4 minutes. Although cooling time may vary depending on the installation environment and air supply pressure, MMI still significantly shortens maintenance downtime compared with conventional SPL.

■ Standards and Sample Preparation

Standards Preparation

Table 2 shows the list of congeners and internal standard analyzed in this work. Standard solutions A, B, C, D and E (to be used as or for the preparation of calibration solutions, checking resolution, QC Standard and internal standard spiking) were prepared in close accordance with EC 2870/2000.[1] All reagents were of analytical grade (purity ≥ 99 %), and ultrapure water was used throughout. A 40 % (v/v) ethanol solution was prepared by diluting absolute ethanol with water and served as the solvent for all standard solutions. During preparation, the mass of each component was recorded to ensure accurate quantification of unknown samples.

Calibration solutions were prepared by combining appropriate volumes of Solutions A and B to obtain final concentrations of 30, 150, 300, and 600 µL/L, with internal standard concentration of 300 µL/L. The concentrations were subsequently recalculated to µg/g for construction of the internal standard calibration curve.

QC Standard was prepared by combining 9:1 volume ratio of Solution D and E, resulting in a final concentration of 270 µL/L for each congener, with internal standard concentration of 300 µL/L.

Table 2. List of Congeners and Internal Standard (IS) Analyzed

ID #	Compound Name
1	Methanol
2	Acetaldehyde
3	Propan-1-ol
4	Butan-2-ol
5	2-Methylpropan-1-ol
6	Ethyl acetate
7	Butan-1-ol
8	Pentan-3-ol (IS)
9	Acetal
10	3-Methylbutan-1-ol
11	2-Methylbutan-1-ol

Sample Preparation

For sample preparation, a measured volume of the spirit drink sample was transferred into a sealed vessel, followed by the addition of a fixed volume of Solution E. The mixture was homogenised by inversion and, if necessary, diluted with 40 % (v/v) ethanol to ensure analyte concentrations fell within the calibration range. Prepared samples should be analysed immediately to minimise the loss of volatile components.

■ Results and Calculation

Separation & Response Factor

Chromatographic separation under the GC conditions produced clear and well-resolved peaks for all target analytes.

Solution C was employed for the evaluation of chromatographic resolution and determination of response factors. EC 2870/2000 specifies a minimum resolution greater than 1.3, except for 2-methylbutan-1-ol/ 3-methylbutan-1-ol pair. The resolution obtained from Solution C meets this requirement, as shown in Table 3, satisfying the requirement of EC 2870/2000. No significant coelutions or unresolved interferences were observed for the regulated congeners. In cases of partial overlap between 2- and 3-methylbutan-1-ol, quantification was performed in accordance with the established and validated method protocol. Response factors (RFs) for the congeners were calculated and applied for subsequent sample quantification using the following equation stated in EC 2870/2000,

$$Response\ Factor\ (RF) = \frac{A_{Analyte}}{A_{IS}} \times \frac{Conc._{IS}\ (\mu g/g)}{Conc._{Analyte}\ (\mu g/g)}$$

Where:

A = Peak Area

IS = Internal Standard

Figure 2 shows the chromatogram obtained from Solution C, while Table 3 summarizes the retention times, chromatographic resolution values between adjacent peaks, and response factors for all congeners based on Solution C.

Table 3. Retention Time, Resolution and Response Factor of Each Congener Based on Solution C

ID #	Compound Name	Retention Time (min)	Resolution	Response Factor (RF)
1	Methanol	7.100	1.3	2.117
2	Acetaldehyde	7.535	3.5	2.052
3	Propan-1-ol	10.712	18.1	1.152
4	Butan-2-ol	12.453	12.7	1.101
5	2-Methylpropan-1-ol	13.560	7.7	0.971
6	Ethyl acetate	13.861	2.1	1.742
7	Butan-1-ol	15.617	11.8	1.041
8	Pentan-3-ol (IS)	18.323	15.6	1.000
9	Acetal	19.992	8.4	1.398
10	3-Methylbutan-1-ol	21.477	4.0	0.951
11	2-Methylbutan-1-ol	21.720	1.1	0.901

Calibration Curves and LOQ

Internal standard calibration curves for individual congeners were established by linear regression analysis of the analyte-to-internal standard (IS) peak area ratio as a function of the corresponding concentration ratio. All target analytes demonstrated excellent linearity across the calibration range, with correlation coefficients (R^2) of ≥0.9994. These values meet the requirements of EC 2870/2000, which specify $R^2 \geq 0.99$. Calibration curves for all congeners are shown in Figure 3.

The limit of quantitation (LOQ) was determined practically by identifying the lowest concentration of congeners with signal-to-noise ratio (S/N) >10. The R^2 values, practical LOQs, and S/N at LOQ for each congener are summarized in Table 4.

Table 4. Summary of R^2 Values, Practical LOQ and S/N at LOQ

ID #	Compound Name	R^2	Practical LOQ (µg/g)	S/N at LOQ
1	Methanol	1.0000	11.1	16.72
2	Acetaldehyde	0.9999	4.5	12.48
3	Propan-1-ol	1.0000	5.5	13.01
4	Butan-2-ol	1.0000	5.6	15.31
5	2-Methylpropan-1-ol	1.0000	5.6	15.30
6	Ethyl acetate	0.9994	6.3	14.28
7	Butan-1-ol	0.9999	5.6	11.22
8	Pentan-3-ol (IS)	-	-	-
9	Acetal	0.9998	11.4	15.24
10	3-Methylbutan-1-ol	0.9999	5.6	10.09
11	2-Methylbutan-1-ol	1.0000	5.7	10.93

Concentration Repeatability (Low and Mid Calib. Points)

Repeatability was assessed at two concentration levels, a low calibration point near the LOQ (12 µL/L) and a mid-range calibration point (300 µL/L), using seven replicate injections per level. Figure 4 shows the overlay chromatograms for the 7 repeated injections of 12 µL/L mixture. For each analyte, within-run standard deviation and %RSD were calculated and are presented in Table 5. Observed %RSD values met the laboratory repeatability targets and were better than the repeatability expectations referenced in the Regulation for similar sample matrices; all congeners exhibited %RSD ≤ 6% at the low-level and ≤ 1% precision at the mid-level calibration standard. Split injection mode in MMI showed extremely good repeatability under routine conditions.

Table 5. Table of Standard Deviation and Concentration %RSD of Each Congener at Low- and Mid-level Calibration Points (*n*=7)

ID #	Compound Name	~12 µL/L		~300 µL/L	
		SD	%RSD	SD	%RSD
1	Methanol	0.0011	5.2	0.0021	0.4
2	Acetaldehyde	0.0003	1.7	0.0015	0.4
3	Propan-1-ol	0.0007	2.3	0.0031	0.4
4	Butan-2-ol	0.0003	0.8	0.0026	0.3
5	2-Methylpropan-1-ol	0.0006	1.6	0.0024	0.2
6	Ethyl acetate	0.0008	2.6	0.0053	0.9
7	Butan-1-ol	0.0005	1.6	0.0022	0.2
8	Pentan-3-ol (IS)				
9	Acetal	0.0007	2.7	0.0044	0.8
10	3-Methylbutan-1-ol	0.0006	1.5	0.0035	0.3
11	2-Methylbutan-1-ol	0.0014	3.4	0.0059	0.5

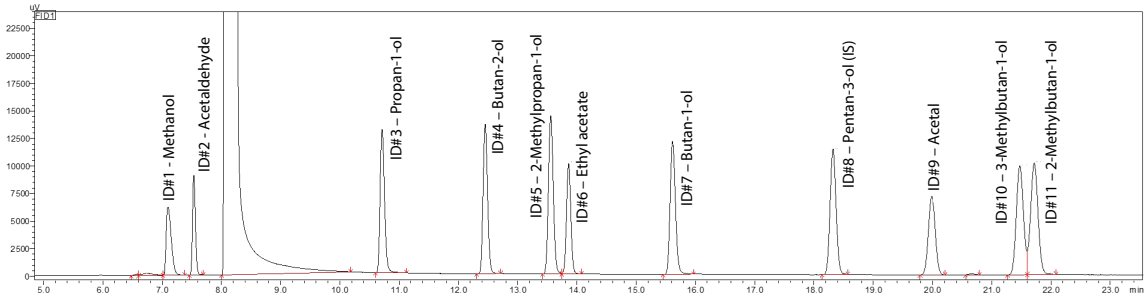

Fig. 2. Chromatogram of Standard Solution C

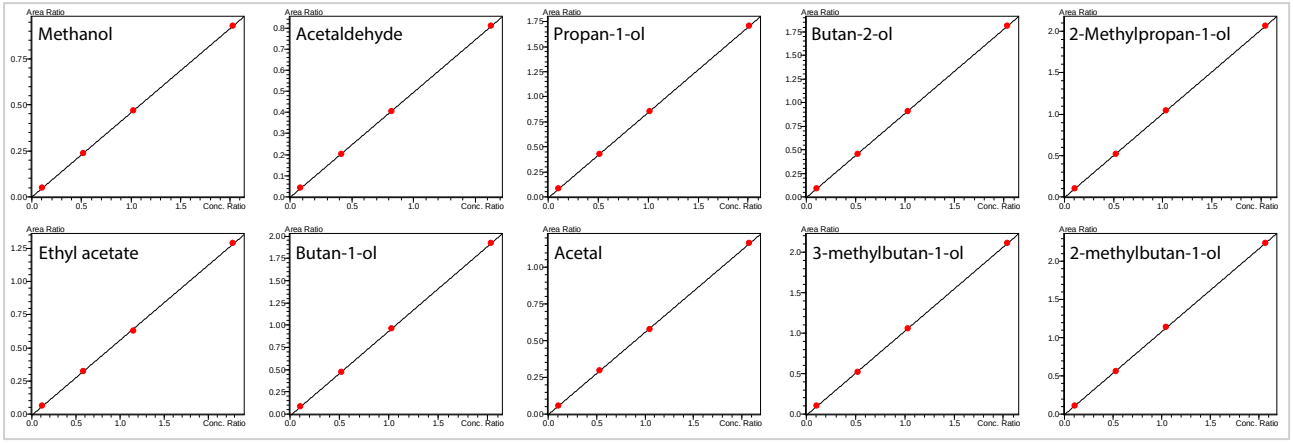

Fig. 3. Calibration Curve (Internal Standard Method) for Each Congener

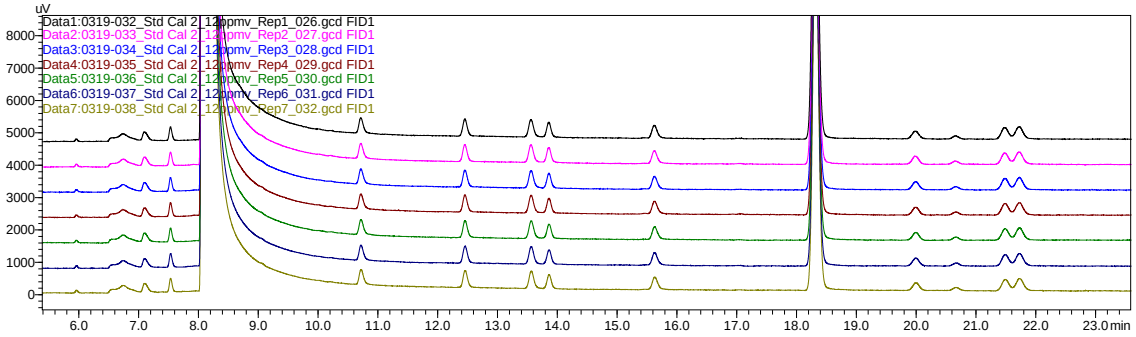

Fig. 4. Overlay Chromatograms for the 7 Repeated Injections of 12 µL/L of Standard Mixture During the Concentration Repeatability study.

Liquor Samples Quantitation

Four different spirit drinks (brandy, whiskey, gin, and vodka) were analyzed and quantified. Concentrations of the liquor samples were calculated using the following equation stated in EC 2870/2000,

$$Conc._{Analyte}(\mu g/g) = \frac{A_{Analyte}}{A_{IS}} \times \frac{M_{IS+Sample}\ (g)}{M_{Sample}(g)} \times Conc._{IS}\ (\mu g/g) \times RF$$

Where:

A = Peak Area
M = Weight
RF = Response Factor

The calculated concentrations were expressed in µg analyte per g sample using the measured peak areas, internal standard and response factors. The results were converted to g per 100 L of absolute alcohol using the measured alcoholic strength and density, following the equation defined in EC 2870/2000,

$$Result = Conc_{Analyte}(\mu g/g) \times \rho \times \frac{10}{Strength(\%\ Vol) \times 1000}$$

Where:

ρ= density in kg/m^3

Results for all analyzed spirit drinks are provided in Table 6, with values flagged if below LOQ. Results exceeded relevant regulatory thresholds were also highlighted.

Table 6. Final Reporting Results of All Liquor Samples in Terms of Gram per 100 L of Absolute Alcohol

ID#	Compound Name	Concentration of in Spirit Drinks (g/100L)			
		Brandy	Whiskey	Gin	Vodka
1	Methanol	37.0	7.09	<LOQ	71.9
2	Acetaldehyde	18.0	10.9	N.D.	N.D.
3	Propan-1-ol	31.4	72.2	<LOQ	2.80
4	Butan-2-ol	N.D.	N.D.	N.D.	N.D.
5	2-Methylpropan-1-ol	123	74.1	N.D.	N.D.
6	Ethyl acetate	48.0	34.3	N.D.	N.D.
7	Butan-1-ol	N.D.	N.D.	N.D.	N.D.
8	Pentan-3-ol (IS)				
9	Acetal	9.8	10.6	N.D.	2.50
10	3-Methylbutan-1-ol	260	28.7	N.D.	N.D.
11	2-Methylbutan-1-ol	66.3	7.09	N.D.	N.D.

QC Standard Recoveries

QC standard was analyzed at the beginning of each analytical batch and at regular intervals thereafter (approximately one QC injection every ten samples) to monitor analytical stability. The concentration of each congener ($Conc.\ Analyte$ in $\mu g/g$) were calculated in the same manner as the unknown sample with the above equation. If the concentrations of all congeners in the QC solution were within ±10% of the expected concentrations, the analysis could proceed. As the QC results in this study met the criteria, the analytical performance was deemed acceptable, and the reported concentrations for the unknown samples were therefore considered valid. The individual QC injection percent recoveries are summarized in Table 7.

Table 7. QC injection percent recoveries at defined points throughout the analytical sequence

ID #	Compound Name	% Recovery			
		QC-1*	QC-2*	QC-3*	QC-4*
1	Methanol	99	97	98	97
2	Acetaldehyde	94	94	96	96
3	Propan-1-ol	97	98	98	98
4	Butan-2-ol	97	96	98	98
5	2-Methylpropan-1-ol	98	97	98	98
6	Ethyl acetate	90	93	92	92
7	Butan-1-ol	98	97	97	98
8	Pentan-3-ol (IS)				
9	Acetal	94	95	95	95
10	3-Methylbutan-1-ol	98	98	97	98
11	2-Methylbutan-1-ol	98	99	98	98

*Where:
QC-1: start of batch;
QC-2: after calibration curve and before repeatability injection;
QC-3: after repeatability injection;
QC-4: after repeatability injection and before unknown sample analysis

■ Conclusions

In conclusion, analysis of volatile substances and methanol in spirit drinks using the Shimadzu Nexis GC-2060 produced robust chromatographic separation, an excellent linear detector response ($R^2 \geq 0.9994$), and very good repeatability (≤ 6.0% RSD at low and ≤ 1% RSD at mid calibration points). Expected concentrations of QC standard were within ±10%, meeting the requirements of EC 2870/2000. The Shimadzu Multi-Mode Injection Unit (MMI), evaluated in split/splitless mode, delivered high injection precision and sensitivity and satisfied EC 2870/2000 criteria. Therefore, the MMI can be adopted for routine use since regular calibration, QC checks and repeatability criteria are met.

■ Reference

1. Commission Regulation (EC) No 2870/2000 of 19 December 2000 laying down Community reference methods for the analysis of spirits drinks. Official Journal of the European Communities, L 333, 20–46. https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX:32000R2870

04-AD-318-EN First Edition: Jul 2026

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