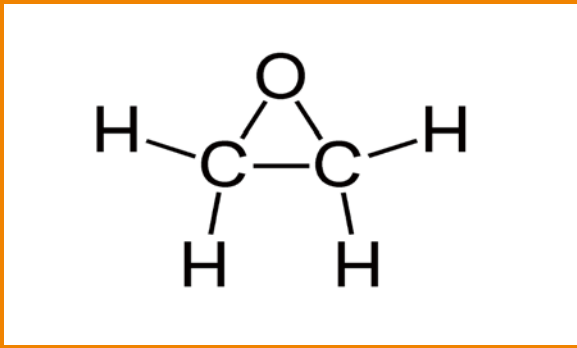
C10G-E100

SHIMADZU
Excellence in Science

Solutions for

Analysis of Ethylene Oxide and 2-Chloroethanol in Food

Chemical Structure of EtO and 2-CE



Ethylene Oxide (EtO)
(Molecular Formula – C_2H_4O)

2-Chloroethanol (2-CE)
(Molecular Formula – C_2H_5ClO)

Significance of quantitation of EtO and 2-CE at trace levels in food and other matrices

Even though Ethylene oxide (EtO / EO) is well-known to be a toxic compound with carcinogenic and mutagenic concerns, it has been widely used for fumigation in the food industry because it effectively reduces or eliminates microbiological contamination with bacteria / fungi. Once in contact with food, EtO undergoes various reactions within the matrix, further producing reaction products such as ethylene glycol, 2-Chloroethanol (2-CE) and 2-bromoethanol, which are also toxic in nature. Hence, the use of EtO for food fumigation has been phased out in many countries due to toxicological concerns. In the EU, the use of EtO for the disinfection of foodstuffs, e.g. in storage areas, is not permitted (ECHA, 2020). The EU has also proposed separate maximum residual limits (MRLs) for EtO and its primary metabolite 2-CE in different food and agriculture commodities, ranging from 0.02 to 0.1 mg/kg (Commission Regulation (EU) 2015/868).

The recent recall of food products exported to the EU due to non-compliance with EU regulations has highlighted the importance of quantitation of EtO and 2-CE residues in food.

To ensure the quality and safety of food products, the European Rapid Alert System for Food and Feed (RASFF) prohibits the sales of goods exceeding the MRL values of 0.05 mg/kg (or 50 ppb) for the sum of EtO and 2-CE.

Reference: EURL-SRM - Analytical Observations Report

EU MRLs of EtO and 2-CE for different products are

No.	Product	EU MRLs
1	Teas, cocoa and spices	0.10 mg/kg
2	Nuts, oil fruits and oilseeds	0.05 mg/kg
3	Fruits, vegetables, sugar plants, fungi and pulses	0.02 mg/kg
4	Cereals and products of animal origin	0.02 mg/kg
5	Apicultural products	0.05 mg/kg

Different methods as per EURL-SRM report

Method Reference	Components		Internal Standard	Liquid Mode		Mode
EURL-SRM Method	EtO	2-CE	Optional	QuEChERS-Method (EN 15662)	QuOil-Method (CEN/TS 17062:2019)	Liquid
Tadeo/Bononi Method	-	2-CE	-	EtO to 2-CE– Conversion Method		Liquid
-	-	2-BE*	-	EtO to 2-BE– Conversion Method		Liquid
Woodrow Method	EtO	-	-	-	-	Headspace
Ayoub Method	EtO	-	-	-	-	SPME

*2-BE: 2-Bromoethanol

Shimadzu Solutions

Shimadzu's solutions for analysis of EtO and 2-CE (Different Techniques / Instruments)

No.	Matrix Covered	Technique	Instrument	Page
1.1	Sesame Seeds	Liquid Injection (EtO and 2-CE)	GCMS-TQ8050 NX with AOC-20i/s	4
		Dynamic Headspace (EtO and 2-CE) - Solvent extraction	GCMS-TQ8050 NX with HS-20 NX	
1.2		Dynamic Headspace (EtO and 2-CE) - Direct sample in HS vial	GCMS-TQ8040 NX with HS-20 NX	5
1.3		Liquid Injection (2-CE) -Conversion Method	GCMS-TQ8040 NX with AOC-20i/s	7
		Dynamic Headspace Injection (2-CE) - Conversion Method	GCMS-TQ8040 NX with HS-20 NX	
2.1	Chili Powder	Dynamic Headspace (EtO and 2-CE) - Solvent extraction	GCMS-TQ8040 NX with AOC-30i	9
		Dynamic Headspace (EtO and 2-CE) - Solvent extraction	GCMS-TQ8050 NX with AOC-20i/s	
2.2		Dynamic Headspace (EtO and 2-CE) - Direct sample in HS vial	GCMS-TQ8050 NX with HS-20 NX	12
3.1	Noodles	Liquid Injection (EtO and 2-CE)	GCMS-TQ8050 NX with AOC-20i/s	15

1.1 Sesame Seeds Analysis

Analysis of EtO and 2-CE in Sesame Seeds

- This section deals with various approaches, extraction process and instrument techniques for the analysis of EtO and 2-CE in sesame seeds.

APPROACH	MODE OF ANALYSIS	INJECTOR	INSTRUMENT TECHNIQUE
EURL-SRM Analytical Observation Report (EtO and 2-CE)	Liquid	SPL	AOC-20i/s - GCMS-TQ8050 NX
Headspace – Method 1st (EtO and 2-CE)	Dynamic Headspace	SPL	HS-20 NX - GCMS-TQ8050 NX
Headspace – Method 2nd (2-CE)	Dynamic Headspace	SPL	HS-20 NX - GCMS-TQ8050 NX
Headspace – Method 3rd (EtO)	Dynamic Headspace	SPL	HS-20 NX - GCMS-TQ8050 NX

Trace level quantitation of Ethylene Oxide (EtO) and 2-Chloroethanol (2-CE) in sesame seeds by using various GC-MS/MS techniques with their own merits and demerits

- **Headspace** – Method 1st (EtO and 2 CE): Solvent extraction followed by analysis with the dynamic headspace mode. For simultaneous analysis of EtO and 2 CE in different matrices, this method is used most of the time.

The European Chemical Agency (ECHA) has classified EtO in category 1B, related to carcinogenicity, mutagenicity and reproductive toxicity, and in category 3, related to acute toxicity. The US National Institutes of Health (NIH) classified EtO as "known to be a human carcinogen based on sufficient evidence of carcinogenicity from studies in humans, including epidemiological studies and studies on mechanisms of carcinogens." The US Environmental Protection Agency (EPA) has concluded that EtO is carcinogenic to humans by the inhalation route of exposure. 2-CE is a prominent metabolite of EtO and is an equally hazardous compound. EU MRLs (Maximum Residue Levels as per European Commission) for EtO and 2-CE are different for different commodities. Out of many matrices, the EU MRLs for cereals, pulses and vegetables are the lowest at 0.02 mg/kg. Considering carcinogenicity and no acceptable threshold for exposure, no Acceptable Daily Intake (ADI) was established for EtO and 2-CE. Hence, it is very important to quantitate EtO and 2-CE as low as possible in food matrices.

With reference to the EURL-SRM Analytical Observation Report, Shimadzu has successfully developed and validated methods for trace-level quantification of EtO and 2-CE impurities in sesame seeds using the GCMS-TQ8050 NX with AOC-20i auto injector and AOC-20s auto sampler, and with HS-20 NX dynamic headspace sampler.

1.2 Sesame Seeds Analysis - Direct Sample Method

Analysis of EtO and 2-CE in Sesame Seeds (Direct Sample In Headspace Vial)

- In this approach, a sesame seed sample was placed directly into a headspace vial and analyzed for its EtO and 2-CE content.
- This method has been successfully validated using the GCMS-TQ8040 NX with HS-20 NX headspace sampler.

APPROACH	MODE OF ANALYSIS	INJECTOR	INSTRUMENT TECHNIQUE
Direct Placement of Sample in Headspace Vial (EtO and 2-CE)	Dynamic Headspace	SPL	HS-20 NX - GCMS-TQ8040 NX

System Description

GC-MS System	GCMS-TQ8040 NX
Headspace Sampler	HS-20 NX (Dynamic Headspace)
Capillary Column	SH-RTX-VMS (60 m X 0.25 mm ID x 1.4 um df)
Ionization Mode	Electron Ionization (EI)
Sample Information	EtO and 2-CE in sesame seeds
Sample Preparation	100 % (Sample in HS vial) + 1 uL Acetonitrile (Diluent)
Spiked Sample Preparation	100 mg Sesame Seeds + 1 uL of 1 ppm Standard
	100 mg Sesame Seeds + 1 uL of 2 ppm Standard
	100 mg Sesame Seeds + 1 uL of 3 ppm Standard

Benefits

- Easier sample preparation with no solvent extraction or any conversion or derivatization process
- Shorter sample preparation time
- Very cost effective
- Outperforms current regulatory limits

Triple Quadrupole Gas Chromatograph Mass Spectrometer

GCMS-TQ8040 NX

- Simplified maintenance allows operators to change parts quickly
- Smart Database Series enables automated method development
- Smart MRM sets method to maximize sensitivity

Product

Chromatography Parameters				
GC-MS System	:	GCMS-TQ8040 NX		
Liquid Sampler	:	-		
Headspace Sampler	:	HS-20 NX (Dynamic Headspace)		
Gas Chromatography Parameters				
Capillary Column	:	SH-VMS (60 m X 0.25 mm ID x 1.4 um df)		
Injection Mode	:	Split		
Flow Control Mode	:	Column Flow		
Carrier Gas	:	Helium		
Column Flow	:	2.00 mL/min		
Linear Velocity	:	36.0 cm/s		
Purge Flow	:	3.0 mL/min		
Split Ratio	:	10		
Diluent	:	Acetonitrile		
Temp. Program		Ramp Rate (°C/min)	Temp. (°C)	Hold Time (min)
			35.0	5.00
		30	235.0	0.33
MS Parameters				
Ionization Mode	:	EI		
Ion Source Temp.	:	250 °C		
Interface Temp.	:	240 °C		
CID Gas	:	Argon		
CID Gas pressure	:	200 kPa		

Headspace Parameters		
Method	:	Dynamic HS
Oven Temp.	:	110 °C
Sample Line Temp.	:	120 °C
Transfer Line Temp.	:	130 °C
Trap Cooling Temp.	:	-20 °C
Trap Desorb Temp.	:	280 °C
Trap Equilib. Temp.	:	-20 °C
Shaking Level	:	5
Multi Inj. Count	:	1
Pressurizing Gas Pressure (kPa)	:	192
Equilibrating Time (min)	:	15
Pressurizing Time (min)	:	0.5
Pressure Equilib. Time (min)	:	0.0
Load Time (min)	:	0.5
Load Equilib. Time (min)	:	0.0
Dry Purge Time (min)	:	0.0
Injection Time (min)	:	10
Needle Flush Time (min)	:	10
GC Cycle Time (min)	:	30
Split Ratio	:	10
Total Flow (mL)	:	25
Trap Tube	:	Tenax Ta

Representative Data

Direct Sample Method - Analysis of EtO and 2-CE (Dynamic Headspace)

EtO

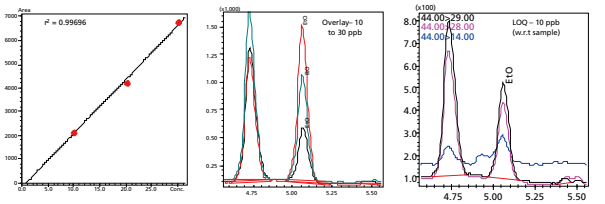


Figure 1: Calibration curve, overlay of linearity standards and chromatogram of LOQ solution

2-CE

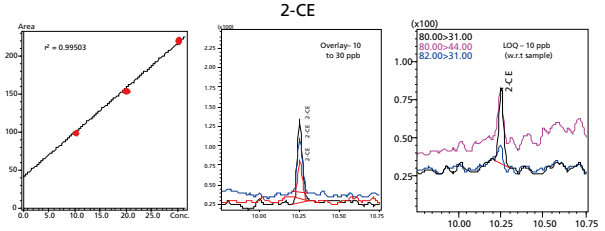


Figure 2: Calibration curve, overlay of linearity standards and chromatogram of LOQ solution

Results

Summary of results is shown in the table below.

Method =>	Dynamic Headspace	
	EtO	2-CE
Linearity levels (On column)	10,20 and 30 ppb	10,20 and 30 ppb
Linearity levels (w.r.t sample)	10,20 and 30 ppb	10,20 and 30 ppb
r^2 (n=3)	0.99696	0.99503
Level 1- conc. (On column)	10 ppb	10 ppb
Level 1- conc. (w.r.t sample)	10 ppb	10 ppb
% RSD (n=3)	2.2	10.1
S/N	26	28
Level 2- conc. (On column)	20 ppb	20 ppb
Level 2- conc. (w.r.t sample)	20 ppb	20 ppb
% RSD (n=3)	2.7	11.7
Level 3- conc. (On column)	30 ppb	30 ppb
Level 3- conc. (w.r.t sample)	30 ppb	30 ppb
% RSD (n=3)	9.7	14.1
Pre-spike 10 ppb-1st (Recovery)	97 %	101 %
Pre-spike 10 ppb-2nd (Recovery)	105 %	91 %

1.3 Sesame Seeds Analysis - Conversion Method

Analysis of EtO and 2-CE in Sesame Seeds (Conversion)

- With reference to the EURL-SRM Analytical Observation Report, the conversion method (EtO=>2-CE) for the trace-level quantification of 2-CE in sesame seeds has been successfully optimized and validated using the GCMS-TQ8040 NX with AOC-20i auto injector and AOC-20s auto sampler, and with HS-20 NX dynamic headspace sampler.

APPROACH	MODE OF ANALYSIS	INJECTOR	INSTRUMENT TECHNIQUE
EtO to 2-CE Conversion	Liquid	SPL	AOC-20i/s - GCMS-TQ8040 NX
EtO to 2-CE Conversion	Dynamic Headspace	SPL	HS-20 NX - GCMS-TQ8040 NX

System Description

GC-MS System	GCMS-TQ8040 NX
Liquid Injector and Sampler	AOC-20i and AOC-20s*
Headspace Sampler	HS-20 NX (Dynamic Headspace)
Capillary Column	SH-Stabilwax DA (60 m X 0.25 mm ID x 0.25 um df)
Ionization Mode	Electron Ionization (EI)
Sample Information	2-CE in sesame seeds (Conversion method)
Sample Preparation	20 % for Liquid / Dynamic Headspace
Reference Method	EURL-SRM Analytical Observation report

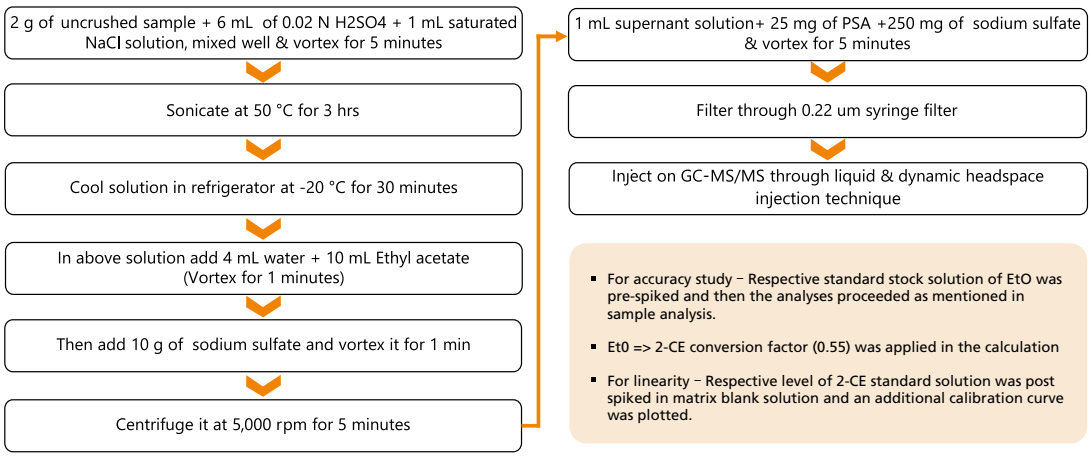
* Equivalent data can be obtained using AOC-30i with or without AOC-20s U.

Benefits

- With reference to EURL-SRM report, the conversion method for trace-level quantification of 2-CE has been optimized
  A) EtO to 2-CE conversion – Analyzed by liquid injection
  B) EtO to 2-CE conversion – Analyzed by dynamic headspace injection
- GCMS-TQ8040 NX equipment with AOC-20i auto injector and AOC-20s auto sampler, and with HS-20 NX dynamic headspace sampler is well-suited for the analysis of EtO and 2-CE

Sample Analysis

Conversion of EtO to 2-CE in sesame seeds and analyzed by liquid injection



- For accuracy study – Respective standard stock solution of EtO was pre-spiked and then the analyses proceeded as mentioned in sample analysis.
- Et0 => 2-CE conversion factor (0.55) was applied in the calculation
- For linearity – Respective level of 2-CE standard solution was post spiked in matrix blank solution and an additional calibration curve was plotted.

Chromatography Parameters				
GC-MS System	:	GCMS-TQ8040 NX		
Liquid Sampler	:	AOC-20i and AOC-20s		
Headspace Sampler	:	HS-20 NX (Dynamic Headspace)		
Gas Chromatography Parameters				
Capillary Column	:	SH-Stabilwax DA (60 m X 0.25 mm ID x 0.25 um df)		
Injection Mode	:	Split		
Flow Control Mode	:	Column Flow		
Carrier Gas	:	Helium		
Column Flow	:	1.00 mL/min		
Linear Velocity	:	25.8 cm/s		
Purge Flow	:	3.0 mL/min		
Split Ratio	:	1 (For liquid injection method)		
Injector Port Temp	:	230.0 °C		
Temp. Program		Ramp Rate (°C/min)	Temp. (°C)	Hold Time (min)
			60.0	2.00
		20	235.0	9.25
MS Parameters				
Ionization Mode	:	EI		
Ion Source Temp.	:	230 °C		
Interface Temp.	:	230 °C		
CID Gas	:	Argon		
CID Gas pressure	:	200 kPa		

Headspace Parameters		
Method	:	Dynamic HS
Oven Temp.	:	110 °C
Sample Line Temp.	:	170 °C
Transfer Line Temp.	:	180 °C
Trap Cooling Temp.	:	-10 °C
Trap Desorb Temp.	:	280 °C
Trap Equilib. Temp.	:	25°C
Shaking Level	:	5
Multi Inj. Count	:	10
Pressurizing Gas Pressure (kPa)	:	192
Equilibrating Time (min)	:	15
Pressurizing Time (min)	:	0.5
Pressure Equilib. Time (min)	:	0.1
Load Time (min)	:	0.5
Load Equilib. Time (min)	:	0.1
Dry Purge Time (min)	:	1
Injection Time (min)	:	5
Needle Flush Time (min)	:	15
GC Cycle Time (min)	:	30
Split Ratio	:	15
Total Flow (mL)	:	19
Trap Tube	:	Tenax Ta

Representative Data

Conversion Method – EtO =>2-CE (Liquid and Dynamic)

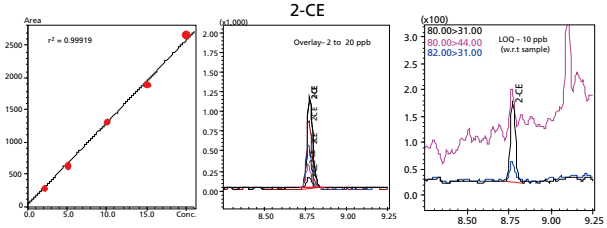


Figure 1: Calibration curve, overlay of linearity standards and chromatogram of LOQ solution for 2-CE (Liquid)

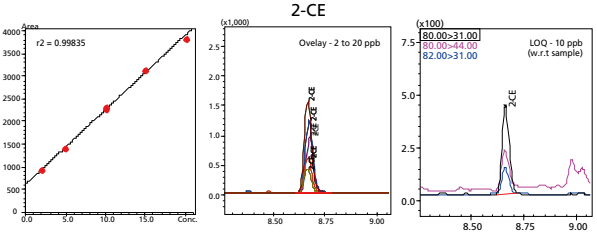


Figure 2: Calibration curve, overlay of linearity standards and chromatogram of LOQ solution for 2-CE (Dynamic)

Results

Summary of results is shown in the table below.

Method =>	Liquid	Dynamic HS
	2-CE	2-CE
Linearity levels (On column)	2,5,10,15 and 20 ppb	2,5,10,15 and 20 ppb
Linearity levels (w.r.t sample)	10,25,50,75 and100 ppb	10,25,50,75 and100 ppb
r^2 (n=3)	0.99919	0.99835
LOQ level conc. (On column)	2 ppb	2 ppb
LOQ level conc. (w.r.t sample)	10 ppb	10 ppb
% RSD (n=6)	7.6	3.8
S/N	16	44
Spiked LOQ level (On column)	2 ppb	2 ppb
Spiked LOQ level (w.r.t sample)	10 ppb	10 ppb
Avg of % recovery (n=3)	101 %	95 %

2.1 Chili Powder Analysis

Analysis of EtO and 2-CE in Chili Powder

- This section deals with various approaches, extraction process and instrument techniques for the analysis of EtO and 2-CE in a chili powder sample.

APPROACH	MODE OF ANALYSIS	INJECTOR	INSTRUMENT TECHNIQUE
EURL-SRM Analytical Observation Report (EtO and 2-CE)	Liquid	SPL	AOC-20i/s - GCMS-TQ8040 NX (AOC-30i - GCMS-TQ8050 NX)*
Headspace – Method 1st (EtO and 2-CE)	Dynamic Headspace	SPL	HS-20 NX - GCMS-TQ8050 NX

*Analysis by GCMS-TQ8050 NX is to show comparison only

System Description

GC-MS System	GCMS-TQ8040 NX
Liquid Injector	AOC-30i
GC-MS System	GCMS-TQ8050 NX
Liquid Injector and Sampler	AOC-20i and AOC-20s
Headspace Sampler	HS-20 NX (Dynamic Headspace)
Capillary Column	RTX-VMS (60 m X 0.45 mm ID x 2.55 um df)
Ionization Mode	Electron Ionization (EI)
Sample Information	EtO and 2-CE in chili powder
Reference Method	EURL-SRM Analytical Observation report

* Chromatographic conditions and sample preparation method are the same as listed in the application note.

Benefits

- The liquid injection method and developed dynamic headspace method for the analysis of EtO and 2-CE in chili powder matrix have been optimized. Both methods outperform the current regulatory limits
- GCMS-TQ8040 NX / GCMS-TQ8050 NX with auto injector and auto sampler, and with dynamic headspace sampler is a complete tool for the analysis of EtO and 2-CE

Triple Quadrupole Gas Chromatograph Mass Spectrometer

GCMS-TQ8050 NX

- Highly efficient detector and three forms of noise-reduction technologies enable ultra-high sensitivity detection
- Enhanced sensitivity enables ultra-trace level analysis
- Simplified user maintenance allows operators to change parts quickly
- Reduced maintenance frequency and lower long-term operational costs

Product

Representative Data

Solvent Extraction Method for EtO and 2-CE (Liquid)

EtO

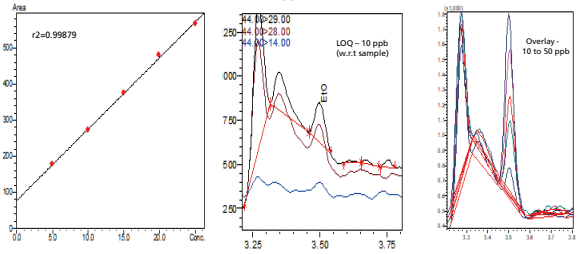


Figure 1: Calibration curve, chromatogram of LOQ solution and overlay of linearity standards for EtO

2-CE

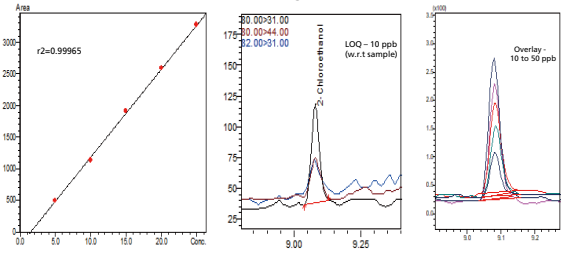


Figure 2: Calibration curve, chromatogram of LOQ solution and overlay of linearity standards for 2-CE

Results

Linearity

Summary of calibration standard is shown in Table 1.

Table 1 : Summary for linearity (n=3 for each level)

Method =>	Liquid	
	EtO	2-CE
Linearity levels (On column)	5,10,15,20 & 25 ppb	
Linearity levels (w.r.t sample)	10,20,30,40 & 50 ppb	
r^2 (n=3)	0.99879	0.99965

Precision

Summary of precision standard is shown in Table 2.

Table 2: Summary of precision standard (n=6)

Method =>	Liquid	
	EtO	2-CE
LOQ level conc. (On column)	5 ppb	5 ppb
LOQ level conc. (w.r.t sample)	10 ppb	10 ppb
% RSD (n=6)	9.3	3.9
S/N	15	79
Highest level conc. (On column)	25 ppb	25 ppb
Highest level conc. (w.r.t sample)	50 ppb	50 ppb
% RSD (n=6)	5.6	3.9

Accuracy

Summary of accuracy is shown in Table 3.

Table 3 : Summary for accuracy (n=3 for each level)

Method =>	Liquid	
	EtO	2-CE
Spiked LOQ level conc. (On column)	5 ppb	5 ppb
Spiked LOQ level conc. (w.r.t sample)	10 ppb	10 ppb
Avg of % recovery (n=3)	98 %	103 %
Spiked 2nd level conc. (On column)	10 ppb	10 ppb
Spiked 2nd level conc. (w.r.t sample)	20 ppb	20 ppb
Avg of % recovery (n=3)	96 %	118 %
Spiked 3rd level conc. (On column)	15 ppb	15 ppb
Spiked 3rd level conc. (w.r.t sample)	30 ppb	30 ppb
Avg of % recovery (n=3)	99 %	107 %

Comparison

Summary of comparison for two instruments is shown in Table 4.

Table 4: Summary for comparison

Method => Liquid	GCMS-TQ8040	GCMS-TQ8050
	EtO	EtO
LOQ level conc. (On column)	5 ppb	2.5 ppb
LOQ level conc. (w.r.t sample)	10 ppb	5 ppb
% RSD (n=6)	9.3	8.7
S/N	15	32
	2-CE	2-CE
LOQ level conc. (On column)	5 ppb	2.5 ppb
LOQ level conc. (w.r.t sample)	10 ppb	5 ppb
% RSD (n=6)	3.9	5.0
S/N	79	70

Representative Data

Solvent Extraction Method for EtO and 2-CE (Dynamic)

EtO

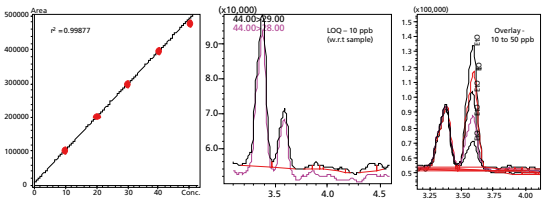


Figure 3: Calibration curve, chromatogram of LOQ solution and overlay of linearity standard for EtO

2-CE

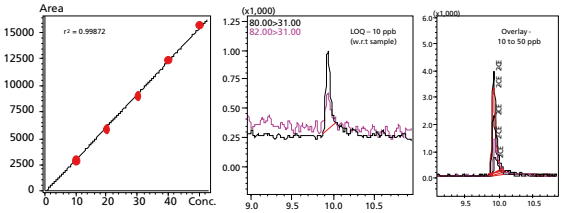


Figure 4: Calibration curve, chromatogram of LOQ solution and overlay of linearity standard for 2-CE

Results

Linearity

Summary of calibration standard is shown in Table 5.

Table 5 : Summary for linearity (n=3 for each level)

Method =>	Liquid	
	EtO	2-CE
Linearity levels (On column)	10,20,30,40 & 50 ppb	
Linearity levels (w.r.t sample)	10,20,30,40 & 50 ppb	
r^2 (n=3)	0.99877	0.99872

Precision

Summary of precision standard solutions is shown in Table 6.

Table 6 : Summary for precision (n=6)

Method =>	Liquid	
	EtO	2-CE
LOQ level conc. (On column)	10 ppb	10 ppb
LOQ level conc. (w.r.t sample)	10 ppb	10 ppb
% RSD (n=6)	2.5	9.7
S/N	15	29

Accuracy

Summary of accuracy is shown in Table 7.

Table 7 : Summary for accuracy (n=3 for each level)

Method =>	Dynamic Headspace	
	EtO	2-CE
Spiked LOQ level conc. (On column)	10 ppb	10 ppb
Spiked LOQ level conc. (w.r.t sample)	10 ppb	10 ppb
Avg of % recovery (n=3)	90 %	93 %
Spiked 2nd level conc. (On column)	30 ppb	30 ppb
Spiked 2nd level conc. (w.r.t sample)	30 ppb	30 ppb
Avg of % recovery (n=3)	87 %	114 %
Spiked 3rd level conc. (On column)	50 ppb	50 ppb
Spiked 3rd level conc. (w.r.t sample)	50 ppb	50 ppb
Avg of % recovery (n=3)	71 %	105 %

AOC-20s U AOC-30i

Auto Injector and Auto Sampler for GC and GC-MS

AOC-30i and AOC-20s U

- Achieve consistent results with high reproducibility
- Experience unwavering performance through long-term stability
- Compact design reduces space requirements

Product

2.2 Chili Powder Analysis - Direct Sample Method

Analysis of EtO and 2-CE in Chili Powder (Direct Sample In Headspace Vial)

- In this approach, a chili powder sample was placed directly into a headspace vial and analyzed for its EtO and 2-CE content.
- This method has been successfully validated using the GCMS-TQ8040 NX with HS-20 NX headspace sampler.

APPROACH	MODE OF ANALYSIS	INJECTOR	INSTRUMENT TECHNIQUE
Place sample in headspace vial (EtO and 2-CE)	Dynamic Headspace	SPL	HS-20 NX - GCMS-TQ8040 NX

System Description

GC-MS System	GCMS-TQ8040 NX
Headspace Sampler	HS-20 NX (Dynamic Headspace)
Capillary Column	SH-RTX-VMS (60 m X 0.25 mm ID x 1.4 um df)
Ionization Mode	Electron Ionization (EI)
Sample Information	EtO and 2-CE in chili powder
Sample Preparation	100 % (As such sample in HS vial) + 1 uL of Acetonitrile (Diluent)
Spiked Sample Preparation	100 mg chili powder + 1 uL of 1 ppm standard
	100 mg chili powder + 1 uL of 2 ppm standard
	100 mg chili powder + 1 uL of 3 ppm standard
	100 mg chili powder + 1 uL of 4 ppm standard
	100 mg chili powder + 1 uL of 5 ppm standard

Benefits

- Easier sample preparation with no solvent extraction or any conversion or derivatization process
- Shorter sample preparation time
- Highly cost effective
- Outperforms current regulatory limits

Chromatography Parameters				
GC-MS System	:	GCMS-TQ8040 NX		
Liquid Sampler	:	-		
Headspace Sampler	:	HS-20 NX (Dynamic Headspace)		
Gas Chromatography Parameters				
Capillary Column	:	SH-VMS (60 m X 0.25 mm ID x 1.4 um df)		
Injection Mode	:	Split		
Flow Control Mode	:	Column Flow		
Carrier Gas	:	Helium		
Column Flow	:	2.00 mL/min		
Linear Velocity	:	36.0 cm/s		
Purge Flow	:	3.0 mL/min		
Split Ratio	:	8		
Diluent	:	Acetonitrile		
Temp. Program		Ramp Rate (°C/min)	Temp. (°C)	Hold Time (min)
			35.0	5.00
		40	235.0	5.00
MS Parameters				
Ionization Mode	:	EI		
Ion Source Temp.	:	250 °C		
Interface Temp.	:	240 °C		
CID Gas	:	Argon		
CID Gas pressure	:	200 kPa		

Headspace Parameters		
Method	:	Dynamic HS
Oven Temp.	:	70 °C
Sample Line Temp.	:	110 °C
Transfer Line Temp.	:	120 °C
Trap Cooling Temp.	:	-20 °C
Trap Desorb Temp.	:	280 °C
Trap Equilib. Temp.	:	-20 °C
Shaking Level	:	5
Multi Inj. Count	:	3
Pressurizing Gas Pressure (kPa)	:	192
Equilibrating Time (min)	:	15
Pressurizing Time (min)	:	0.5
Pressure Equilib. Time (min)	:	0.1
Load Time (min)	:	0.5
Load Equilib. Time (min)	:	0.1
Dry Purge Time (min)	:	0.0
Injection Time (min)	:	10
Needle Flush Time (min)	:	10
GC Cycle Time (min)	:	28
Split Ratio	:	8
Total Flow (mL)	:	21
Trap Tube	:	Tenax Ta

Gas Chromatograph Mass Spectrometer with Headspace Sampler

GCMS-TQ8040 NX with HS-20 NX Series

- Optimal solution for volatile component analysis
- Upgraded HS-20 NX offers a powerful solution
- User-friendly option for both research and quality control

Product

Representative Data

Direct Sample Method - Analysis of EtO and 2-CE (Dynamic Headspace)

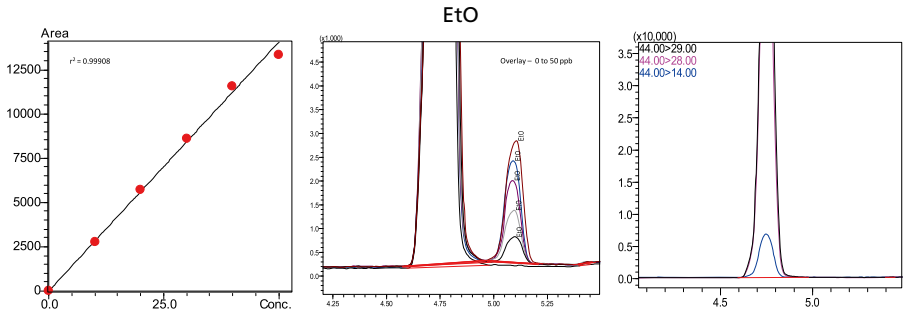


Figure 1: Calibration curve, overlay of linearity standards and chromatogram of sample solution

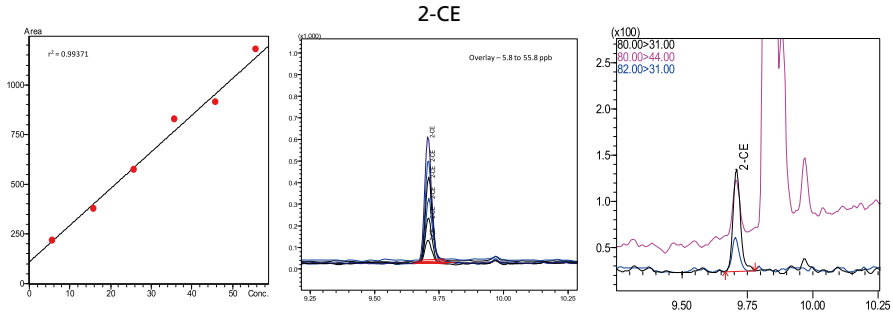


Figure 2: Calibration curve, overlay of linearity standards and chromatogram of sample solution

Results

Summary of results is shown in the table below.

Method =>	Dynamic Headspace	
	EtO	2-CE
Linearity levels (On column)	10,20,30,40 and 50 ppb	10,20,30,40 and 50 ppb
Linearity levels (w.r.t sample)	10,20,30,40 and 50 ppb	10,20,30,40 and 50 ppb
r^2 (n=3)	0.99908	0.99371
Level 1 - conc. (On column)	10 ppb	10 ppb
Level 1 - conc. (w.r.t sample)	10 ppb	10 ppb
% RSD (n=3)	12.9	3.2
S/N	42	97
Sample (Conc. observed)	-	5.8 ppb
Pre spiked 10 ppb (Conc. observed)	10.8 ppb	17.3 ppb
Avg of % recovery (n=3)	108 %	109 %

3.1 Noodles

Analysis of EtO and 2-CE in Ramen (Instant Noodles)

- This section introduces the extraction process and instrumental techniques used for the analysis of EtO and 2-CE in Ramen.

APPROACH	MODE OF ANALYSIS	INJECTOR	INSTRUMENT TECHNIQUE
EURL-SRM Analytical Observation Report (EtO and 2-CE)	Liquid	SPL	AOC-20i and AOC-20s* - GCMS-TQ8050 NX

* Equivalent data can be obtained using AOC-30i with or without AOC-20s U.

Analysis of Ethylene Oxide in Ramen (Instant Noodles) by GC-MS/MS

- GCMS-TQ8050 NX equipment with AOC-20i and AOC-20s provides stable analytical results.
- Excellent reproducibility in actual sample analysis demonstrates an RSD below 5%.
- Good recovery rates were achieved for EtO (98.9%) and 2-CE (106.3%) in a complex food matrix.

EtO is typically used as a fumigant pesticide to reduce microbial or bacterial contamination. However, it is banned in the European Union (EU) because of its carcinogenic and mutagenic properties. In 2020, Belgium was the first country to raise the alarm about the presence of EtO in imported sesame seed. Since then, EtO has been found in various food additives, including locust bean gum, which is a thickening agent or stabilizer. These ingredients are commonly used in formulations and can be found in products such as flour, cereals, ice cream, chocolate, biscuits, bread, and cheese. As these items are sold by major brands and retailers, it has led to thousands of products being affected and taken off the shelves.

With the surge in demand for EtO detection worldwide, there is an increased need for greater laboratory capacity and relevant analysis methods to ensure food safety. In this Technology Brief, Shimadzu introduces an analysis of EtO with QuEChERS and the GCMS-TQ8050 NX, which is equipped with the AOC-20i and AOC-20s for automation capabilities to meet the requirement for higher throughput and faster turnaround times.

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