

Prohibited ingredients in vaping products by Gas Chromatography-Mass Spectrometry

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Prohibited ingredients in vaping products by GCMS

This method describes the identification of the following prohibited ingredients in vaping products by Gas Chromatography-Mass Spectrometry (GCMS): Diacetyl; 2,3-Pentanedione; Acetoin; Ethylene glycol; Benzaldehyde; Diethylene glycol and trans-Cinnamaldehyde.

Instrument set up

Chromatographic system

Table 1: Chromatographic system set up

Instrument:	GCMS			
Column:	SH-624, 0.32 mm ID, 1.8 μm DF and 30 m L.			
Oven Temperature Program:	Rate Temperature Hold Time			
	n/a	40 °C	3.00 min	
	10 °C/min 210 °C 0.00 min			
	25 °C/min 300 °C 1.40 min			
Injection Temperature	250 °C			
Injection Mode	Split			
Split Ratio	20.0			
Column Flow	2.00 mL/min			

MRM Mode: Mass Spectrometer System

Table 2: Ion source and interface temperatures

Ion Source Temperature	200 °C
Interface Temperature	250 °C

Table 3: MRM event table for method: Quantifier transitions

Peak	Start time	End time	Precursor → Product	CE
Diacetyl	3.70	5.50	86→43	6
2,3-pentanedione	5.50	6.84	100→57	6
Acetoin	6.84	7.52	45→27	12
Ethylene glycol	7.52	10.00	62→33	3
Benzaldehyde	10.00	12.59	106→105	6
Diethylene glycol	12.59	15.80	75→45	6
Trans-	17.38	18.18	131→77	27
Cinnamaldehyde				

Table 4: MRM event table for method: Qualifier transitions

Peak	Precursor →	CE	lon ratio ¹	Precursor →	CE	Ion
	Product			Product		ratio ¹
Diacetyl	86→86	0	3.0 ± 1.5	n/a	n/a	n/a
2,3-pentanedione	100→43	12	9.4 ± 2.8	n/a	n/a	n/a
Acetoin	45→29	9	106 ± 32	n/a	n/a	n/a
Ethylene glycol	62→31	6	55 ± 16	n/a	n/a	n/a

¹ The ion ratios in this table are indicative only, these should be updated using the Limit Standard or the Concentrated standard. Specifications: Ion ratio ≤ 5 (\pm 50% relative); Ion ratio > 5 (\pm 30% relative).

Peak	Precursor →	CE	Ion ratio ¹	Precursor →	CE	Ion
	Product			Product		ratio ¹
Benzaldehyde	106→77	16.5	50 ± 15	106→78	15	4.9 ±
						2.4
Diethylene glycol	45→27	12	18 ± 6	45→29	9	15 ± 4
Trans-	131→103	9	45 ± 14	131→131	0	18 ± 5
Cinnamaldehyde						

Limits of detection

Table 5: Analyte Limits of detection

Analyte	Limit of Detection (µg/mL)
Diacetyl	0.3
2,3-pentanedione	0.3
Acetoin	0.2
Ethylene glycol	0.8
Benzaldehyde	0.2
Diethylene glycol	0.8
Trans-Cinnamaldehyde	0.2

Solutions

Prohibited ingredients stock solution

Prepare individual solutions in acetonitrile of the following analytes with the concentrations described in the Table 6. These solutions can be stored at 4 °C in an amber flask for up to 40 days.

Table 6: Analyte stock solution concentrations in acetonitrile

Analyte	Concentration (µg/mL)
Diacetyl	1000
2,3-Pentanedione	1000
Acetoin	1000
Benzaldehyde	1000
trans-Cinnamaldehyde	1000
Ethylene glycol	2500
Diethylene glycol	2500

Working Standard

Prepare a mixed standard using the stock solutions and methanol as the diluent, adjusting each individual aliquot to obtain the concentrations in Table 7. These solutions should be made fresh prior to testing.

Table 7: Analyte stock solution concentrations in acetonitrile

Analyte	Concentration (µg/mL)
Diacetyl	10
2,3-Pentanedione	10
Acetoin	10
Benzaldehyde	10
trans-Cinnamaldehyde	10
Ethylene glycol	25

Analyte	Concentration (µg/mL)
Diethylene glycol	25

Quality control sample

Prepare a mixture of glycerol and propylene glycol by measuring 7 mL of glycerol and 3 mL of propylene glycol and mixing the two until a homogeneous mixture is obtained (QC sample).

Using a suitable positive displacement piston operated volumetric apparatus (POVA), transfer 80 µL of the resulting mixture to an HPLC vial and then transfer 1520 µL of methanol (QC sample blank).

Limit standard

Using a suitable positive displacement POVA, transfer 80 μ L of the QC sample to an HPLC vial and then transfer 80 μ L of the Working Standard followed by addition of 1440 μ L of methanol (QC sample – limit test solution).

Concentrated standard

Using a suitable positive displacement POVA, transfer 80 μ L of the QC sample to an HPLC vial and then transfer 1520 μ L of the Working Standard (Second identification standard).

Sample solution

Prepare a fresh 1 in 20 dilution of the sample in methanol by transferring 80 μ L of the sample to an HPLC vial followed by 1520 μ L of methanol using a suitable positive displacement POVA.

For samples that contain a large amount of acetoin which is not identified due to the ion ratios being saturated, a further dilution can be prepared to improve the ion ratio match.

Injection sequence

Inject in this order the following solutions:

Methanol	2 injections
QC Sample blank	2 injections
Limit Standard	2 injections
Concentrated standard	2 injections
Samples (up to 6 samples)	1 injection
Methanol	1 injection
QC Sample blank	1 injection
Limit Standard	1 injection
Concentrated standard	1 injection
Samples (up to 6 samples)	1 injection
Methanol	1 injection
QC Sample blank	1 injection
Limit Standard	1 injection

Concentrated standard

1 injection

End

System suitability criteria for the Limit solution

The %RSD of the area of each analyte for the Limit solution (injected at least 3 times) should be NMT 20%.

Identification criteria

Prohibited Ingredients:

Disregard any peaks with areas below the areas observed in the Limit Standard.

Disregard any peaks with retention time difference (absolute) more than 0.10 min compared with the same analyte in the Limit Standard.

Disregard any peaks where the ion ratio test fails, taking into consideration the ion ratios set up either by the Limit Standard or the Concentrated standard for identification.

A prohibited ingredient is present when the area of the peak is greater than the corresponding peak in the limit standard, and the retention time difference and ion ratio requirements are met.

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