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Analytical Report

Title	Volatile Organic Compounds Profile by GC-MS in "Cream de Menthe" E-liquid Flavor Concentrate
Report No.	SE-37974-19
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Specific Aim:

The goal is to determine levels of Acetoin, Diacetyl and Acetylpropionyl with reporting cutoff 5 ppm

Background of the matter

Diacetyl (2,3-butanedione, CAS 431-03-8) is a volatile liquid with intense buttery flavor occurring naturally in dairy and fermented foods. It is extensively used as a flavoring agent to impart a buttery flavor.

Acetoin (3-Hydroxy-2-butanone, CAS 513-86-0), Acetylpropionyl (2,3-Pentadione, CAS 600-14-6) are related compounds with somewhat similar custard flavor also used as flavoring agents.

These compounds find use in flavoring compositions designed to impart certain flavors to foodstuffs. They are also common products of fermentation and present in beers, wines and dairy products albeit in low (tens of ppb) levels.

Samples

Sample arrived as liquid with strong characteristic odor labeled "**Cream de Menthe 100160**". 5 ml of submitted material was placed in 40 ml headspace collection vial followed by addition of internal standard (IS). The vial was held at 50C for 4 hours before headspace sampling. Volatile compounds were collected out of headspace with the aid of 1 ml gastight syringe. 0.2 ml of collected headspace was injected at split ratio of 10.

Experimental:

- GC conditions:
 - Injector temperature: 250 C
 - Initial oven temperature: 40 C
 - Hold I 2 min
 - Ramp I 10 C/min
 - Final temperature I 220 C
 - Hold II 5 min
- MS parameters
 - Ionization and ion polarity EI+
 - Scan rate 2 scans/sec
 - Mass range 35-300 Da
 - Ion source temperature 150
 - Transfer line temperature 220C
- GC-MS analysis. Waters/Micromass Quatro GC mass spectrometer interfaced to a Thermo Electron Trace gas chromatograph was utilized for the analysis. 30M 0.32 mm ID DB-624 column was used to separate components. Carrier gas was helium at 2 ml/min.
- Data treatment.

Methylethylketone (MEK) was used as an internal standard. Table 1 lists qualifier and quantifier ions for the target compounds.

RT, min	Compound	Qualifier, m/z	Quantifier, m/z
4.25	Diacetyl	86	43
6.35	Acetylpropionyl	100	43
7.2	Acetoin	88	45

Table 1 Retention times and m/z ratios of the target compounds

Results:

TIC GC-MS chromatogram of the submitted sample is shown in Appendix I, figure 1. Displayed on figure 2 are selected ion chromatograms for ions used to quantify the target compounds. Arrows designate expected retention times (RT) for diacetyl, acetylpropionyl and acetoin elution (4.25,

6.35 and 7.2 min respectively) out of the GC column¹ Green arrow indicates a compound is not present at reporting cutoff and red arrow indicates that level exceeds the cutoff. Table 2 lists target compound levels.

Compound	Concentration, ppm
Diacetyl	N/D
Acetylpropionyl	N/D
Acetoin	16

Table 2 Sample "Cream de Menthe", target compound levels. Concentration units are ppm or N/D, not detected.

1 Presence of a peak at designated RT does not necessary signify that a target compound detected. If stated as N/D the qualifier ion is not detected.

APPENDIX I

Figure 1. Sample "Cream de Manthe" 100160" TIC chromatogram

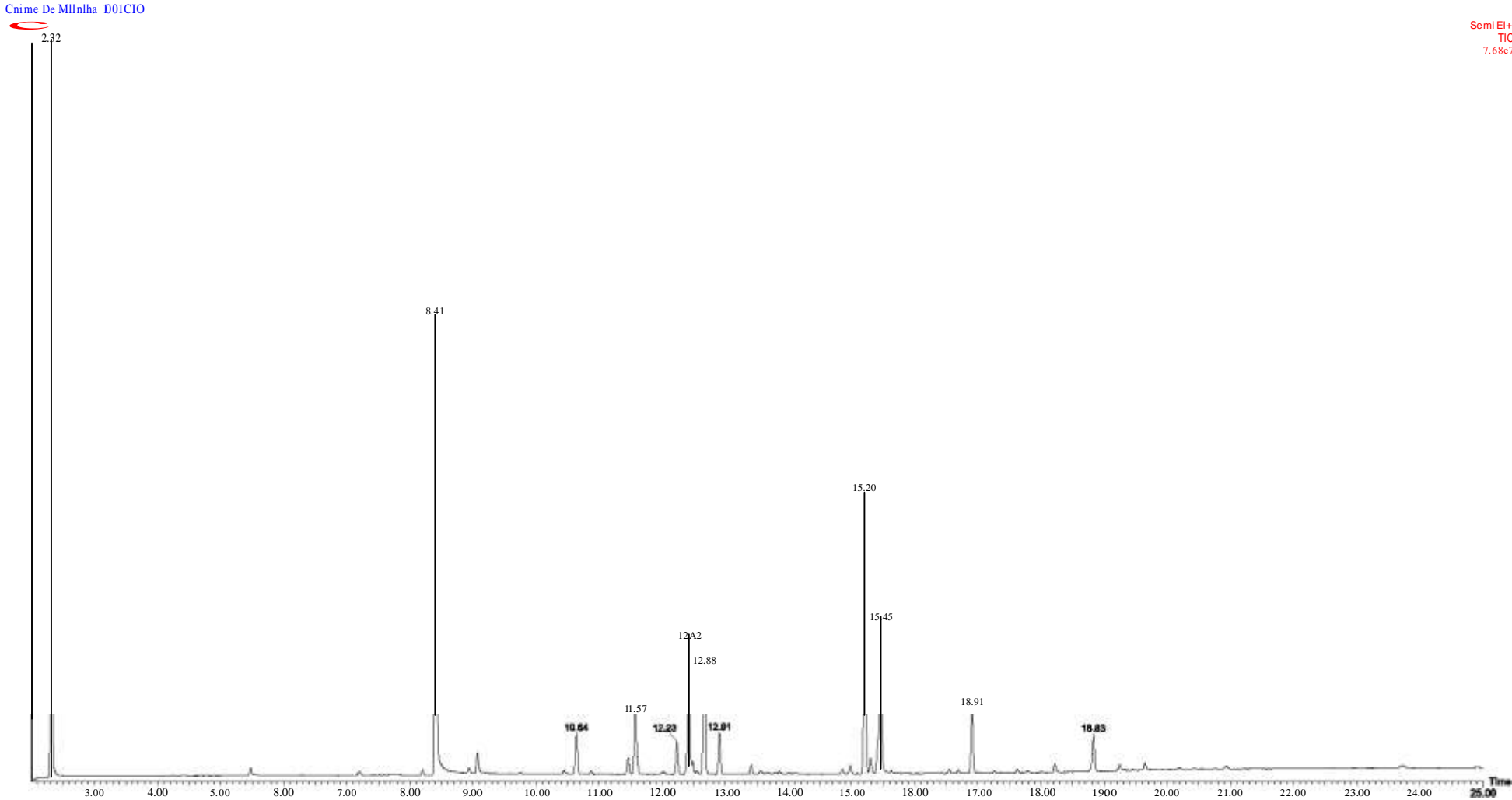


Figure 2. Sample "Cream de Menthe" 100160" Selected Ion Chromatograms

