

Analysis of E-Cigarette Liquid by 7890 GC and Intuvo 9000 Using an Agilent J&W DB-HeavyWAX GC Column

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Abstract

This Application Note demonstrates the use of the Agilent J&W DB-HeavyWAX GC column for the analysis of e-cigarette liquid using an Agilent 7890 and Agilent Intuvo 9000 gas chromatograph. E-cigarette liquids can contain many fruit flavor compounds that require a higher final temperature or longer run time to elute off the column. The DB-HeavyWAX has a maximum isothermal temperature of 280 °C and programmed temperature of 290 °C, with increased thermal stability, allowing for the efficient analysis of later eluting and higher boiling compounds.

Introduction

As the use of e-cigarettes, also known as vaping, has become more popular so has the analysis of the compounds in the e-cigarette liquid. E-cigarette liquid becomes an aerosol when heated by a battery heating element¹. The two main compounds in the liquid mixtures are propylene glycol and glycerol². In addition to the main components, nicotine and flavor compounds may be present, the most popular being mint or fruit flavors. Some of the fruit flavor compounds have higher boiling compounds and require a longer final hold time at 250 °C with traditional WAX type columns to elute off the GC column. Without this longer final hold, they could potentially carryover into subsequent injections and present interference problems.

The DB-HeavyWAX has an increased temperature limit compared to traditional WAX type columns, increasing the isothermal temperature from 250 °C to 280 °C. This increased temperature limit permits the analysis of higher boiling compounds without risking the integrity of the column phase³. Using the increased temperature limit of the DB-HeavyWAX, the risk of carryover and interferences can be decreased and injection-to-injection reproducibility increased.

As the popularity of e-cigarettes grows, so does the need for a high-throughput analytical technique such as that offered by the Intuvo 9000 GC. The planar column design allows for efficient heating and ramping rates up to 250 °C/min, enabling rapid GC analysis. Combining the DB-HeavyWAX with the Intuvo 9000 GC allows for a faster and more reproducible analysis of components in e-cigarette liquid.

Materials and methods

An Agilent 7890 GC equipped with a split/splitless inlet, an Agilent Intuvo 9000 GC/FID equipped with a split/splitless inlet, and an Agilent 7693 Sampler with control software MassHunter were used for GC/MSD experiments.

Sample preparation

Commercially available samples of e-cigarette liquid were purchased and prepared 10:1 in isopropyl alcohol (Sigma) and injected in split mode.

Instrument conditions

7890 GC conditions	
Column	J&W DB-HeavyWAX, 60 m × 0.25 mm, 0.25 µm (p/n 122-7162)
Carrier	Helium, constant flow, 2.0 mL/min
Oven	60 °C (2.0 minutes), ramp 10 °C/min to 250 °C (15.0 minutes) 60 °C (2.0 minutes), ramp 10 °C/min to 280 °C (15.0 minutes)
Inlet	Split mode, 250 °C, split ratio 50:1
Inlet liner	Ultra Inert, split, low pressure drop, glass wool (p/n 5190-2295)
GC/FID	7890B GC equipped with 5977 MSD
Sampler	Agilent 7693 autosampler
Intuvo 9000 GC conditions	
Column	Agilent J&W DB-HeavyWAX Intuvo GC column module, 30 m × 0.25 mm, 0.25 µm (p/n 122-7132-INT)
Carrier	Helium, constant flow, 2 mL/min
Oven	60 °C (2.0 minutes), ramp 10 °C/min to 250 °C (15.0 minutes) 60 °C (2.0 minutes), ramp 250 °C/min to 280 °C (5.0 minutes)
Inlet	Split mode, 250 °C, split ratio 50:1
Inlet liner	Ultra Inert, split, low pressure drop, glass wool (p/n 5190-2295)
Guard Chip	Guard Chip, Intuvo split/splitless (p/n G4587-60565) 250 °C Isothermal
Main bus	250 °C
GC/FID	Intuvo 9000 GC equipped with 5977 MSD
Sampler	7693 autosampler
Flowpath supplies	
Vials	2 mL, screw top, amber, write-on spot, certified, (p/n 5182-0716, 100/pk)
Vial caps	9 mm blue screw cap, PTFE/RS (p/n 5185-5820, 500/pk)
Septum	Bleed and temperature optimized (BTO), 11 mm septa (p/n 5183-4757, 50/pk)
Gold seal (7980)	Ultra Inert gold seals (p/n 5190-6145, 10/pk)
Guard Chip	Guard Chip, Intuvo split/splitless (p/n G4587-60565)
Flow Chips	Intuvo Inlet Chip (p/n G4581-60031) Flow Chip, Intuvo, D1 (p/n G4581-60032) Detector tail, Intuvo, FID or TCD (p/n G4583-60331)
Inlet/FID (7890)	85:15 Vespel: graphite ferrules (p/n 5062-3508, 10/pk)
Inlet/FID (Intuvo)	Polyimide Intuvo gasket (p/n 5190-9072)

Results and discussion

In Figure 1, a sample of Flavor A flavored e-cigarette liquid, containing 10 mg of nicotine, was analyzed on a 7890 GC/FID. Figure 2 demonstrates the analysis of an e-cigarette liquid titled

Flavor B containing 2.5 mg of nicotine. The components in the e-liquids were confirmed by analyzing by mass spectrometry, and identified by library searching against NIST14 EI Mass Spectral Database.

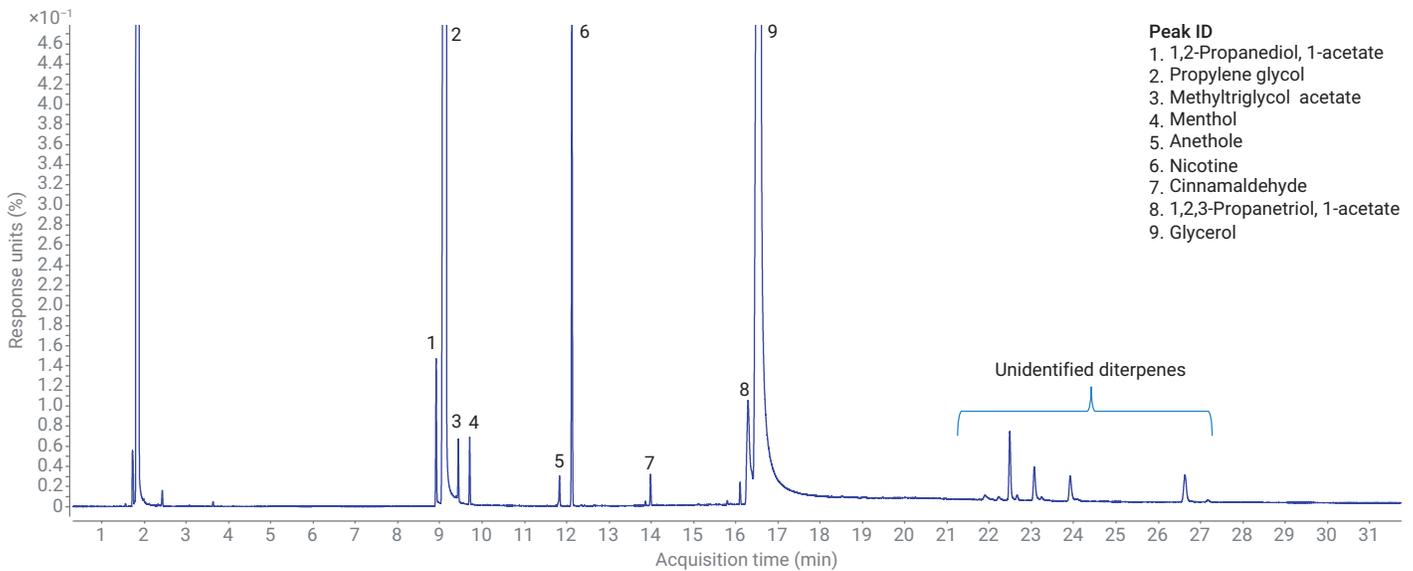


Figure 1. A sample of Flavor A e-cigarette liquid analyzed on a DB-HeavyWAX column using a 7890 GC with a final temperature of 250 °C.

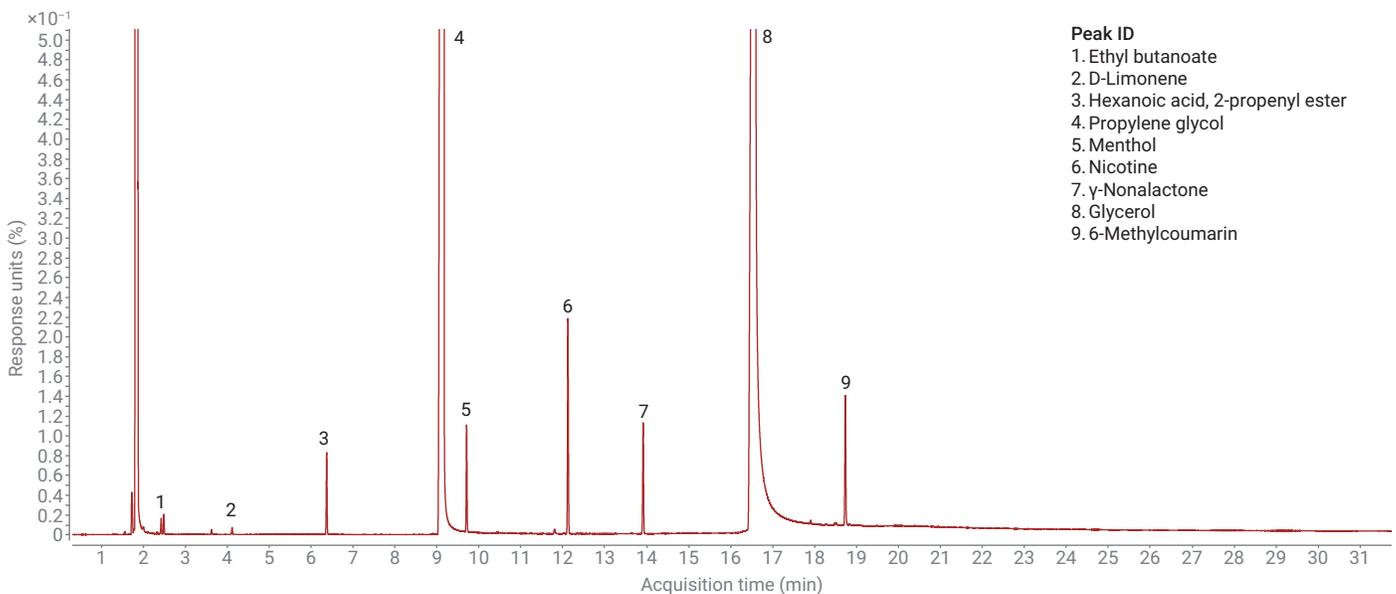


Figure 2. A sample of Flavor B e-cigarette liquid analyzed on a DB-HeavyWAX column using a 7890 GC with a final temperature of 250 °C.

The DB-HeavyWAX has an increased temperature limit of 280 °C isothermal and 290 °C programmed, and maintains low levels of bleed even at a final temperature of 280 °C, as shown in Figure 3. The decreased column bleed is due to the improved thermal stability of the DB-HeavyWAX, and it allows for greater sensitivity of later eluting compounds at higher temperatures.

Equivalency to an Intuvo 9000 GC

An identical method was applied on an Intuvo 9000 GC and a 7890 GC, except for the Guard Chip, which was not present on the 7890 GC. The Intuvo 9000 uses a planar column design and click and run connections. The same sample of e-cigarette liquid was analyzed on a DB-HeavyWAX designed for a 7890 GC with a traditional seven-inch

column, and a DB-HeavyWAX designed for an Intuvo 9000 with planar column design. Identical temperature program, pressure settings, column phase, and column dimensions were used on the Intuvo 9000 and the 7890 GC. Figure 4 demonstrates that, under identical conditions, the two chromatograms are almost indistinguishable regarding time and peak height.

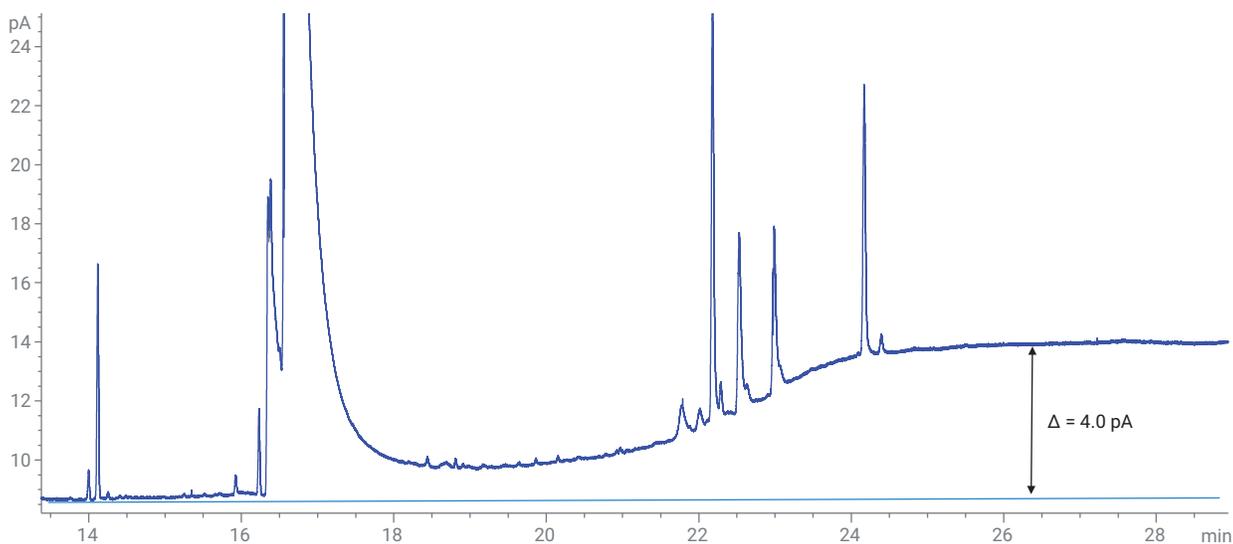


Figure 3. A sample of Flavor A e-liquid analyzed on a DB-HeavyWAX column—zoomed in on the 14–30 minutes range. Bleed only 4 pA at 280 °C.

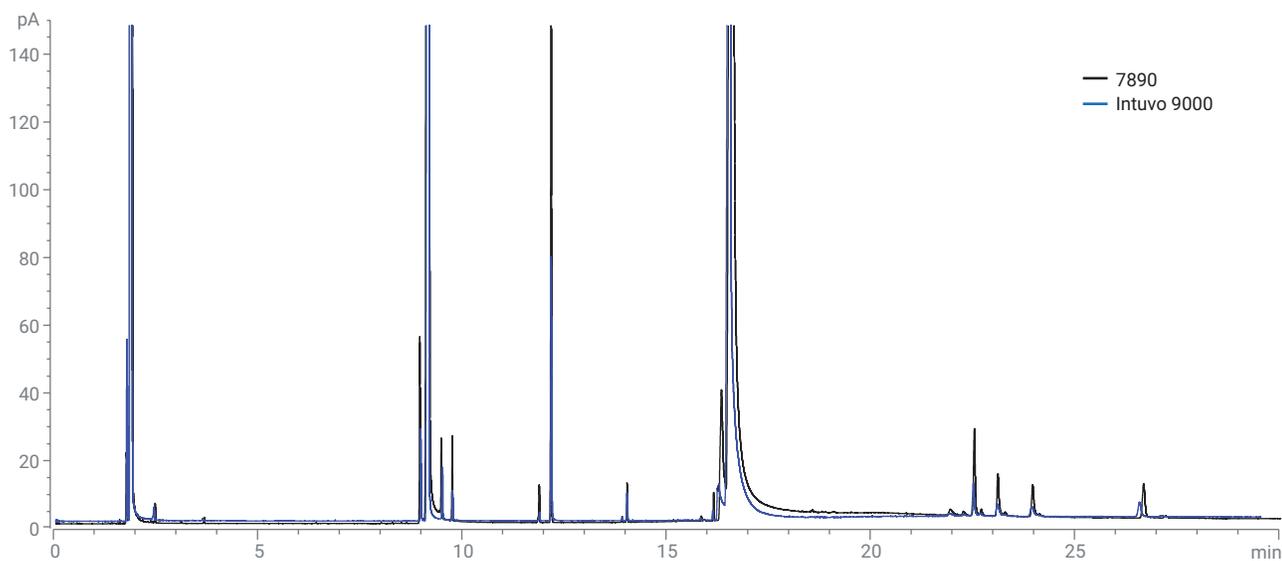


Figure 4. Sample of Flavor A liquid analyzed on a 7890 GC and Intuvo 9000 GC using a DB-HeavyWAX GC column.

In addition, using the fast ramping capabilities of the Intuvo 9000 GC, the analysis time of e-liquids was significantly reduced. Figure 5 illustrates that, by maximizing the ramping rate of 250 °C/min of the Intuvo 9000 GC, the run time can be reduced from 20 to 10 minutes.

The DB-HeavyWAX has a maximum program temperature of 290 °C, which can be sustained for short periods of time without damaging the column. Figure 6 demonstrates that, by combining the fast ramping rate of the Intuvo 9000, and the maximum programmed temperature of the DB-HeavyWAX of 290 °C, the analysis

time can be decreased to less than six minutes. DB-HeavyWAX can be taken to a final temperature of 290 °C without a significant increase in column bleed. The column bleed increased from 4 pA (Figure 3) at a final temperature of 280 °C to only 10.5 pA at a final temperature of 290 °C (Figure 6), clearly demonstrating the improved thermal stability of DB-HeavyWAX.

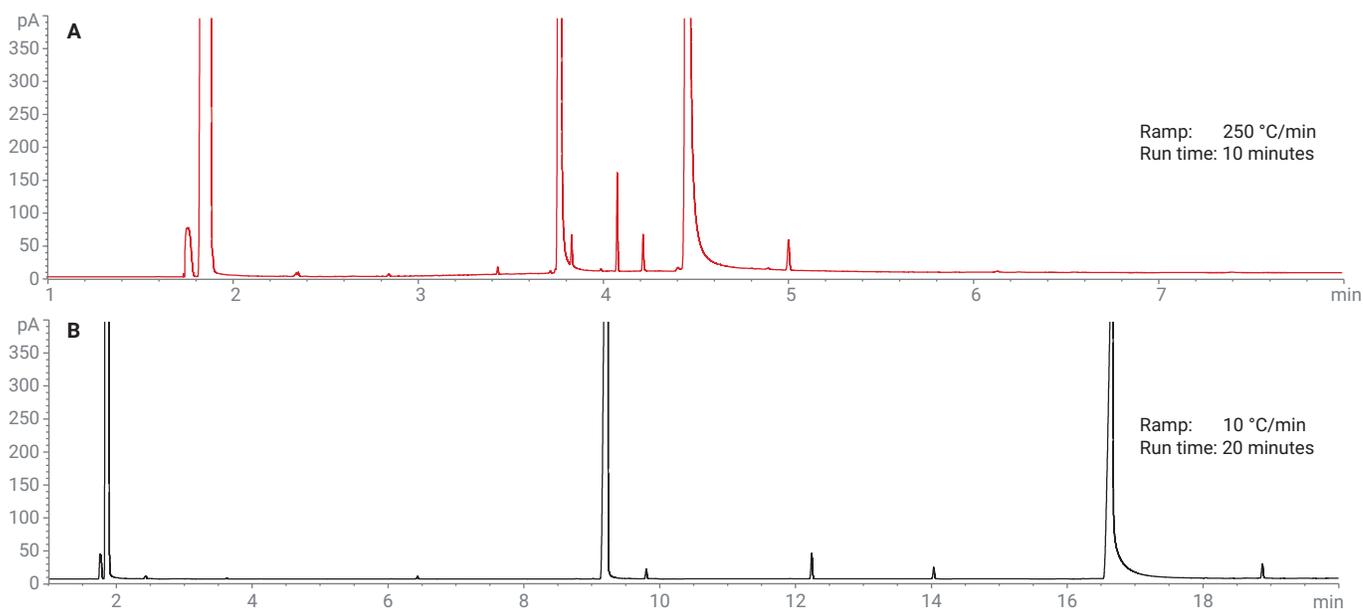


Figure 5. A sample of Flavor B e-cigarette liquid analyzed on a DB-HeavyWAX column using an Intuvo 9000 GC with a final temperature of 280 °C and ramping rates of 10 °C/min and 250 °C/min.

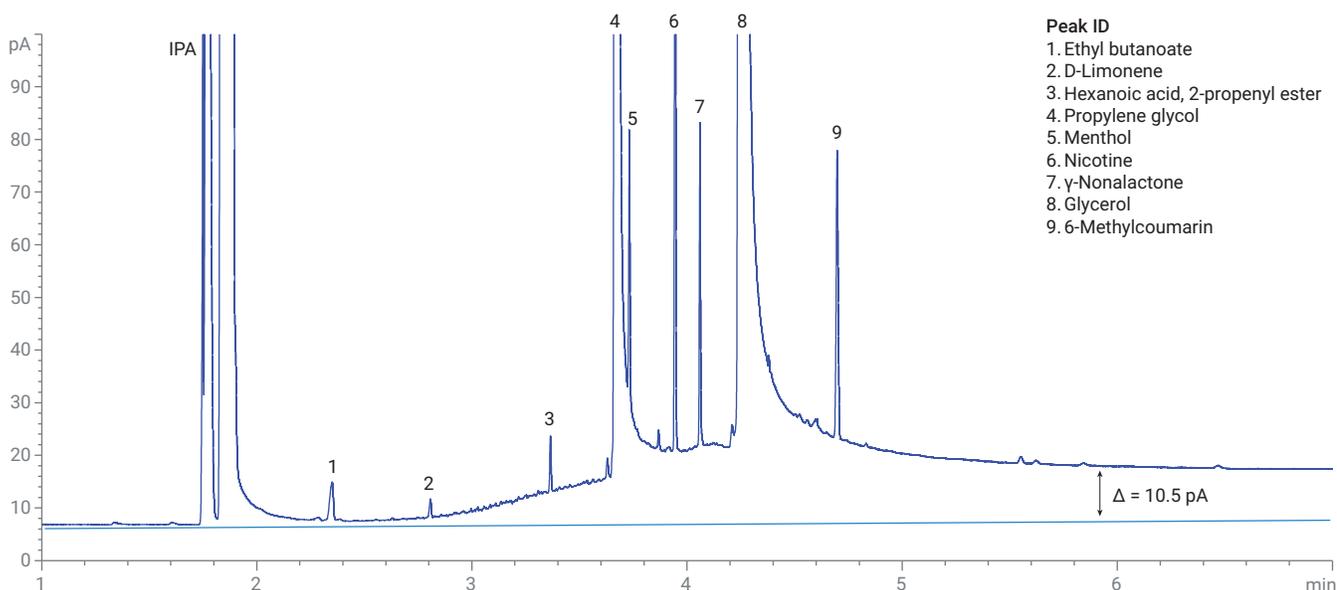


Figure 6. A sample of Flavor B e-cigarette liquid analyzed on a DB-HeavyWAX column using an Intuvo 9000 GC with a final temperature of 290 °C.

Conclusion

DB-HeavyWAX can be used for the analysis of e-cigarette liquid both on a 7890 GC and an Intuvo 9000 GC, with equivalent results. The improved thermal stability of the DB-HeavyWAX with an increased temperature limit of 280 °C isothermal and 290 °C programmed, allows for greater sensitivity for later eluting compounds that can be present in e-cigarette liquids. Additionally, using the fast ramping rate of 250 °C/min of the Intuvo 9000 and the maximum programmed temperature of the DB-HeavyWAX of 290 °C, the overall run time can be decreased to less than six minutes.

References

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2. F. David, B. D'Haenens, C. Devos. Determination of nicotine, propylene glycol, and glycerol in e-liquids according to ISO/CD 20714 using an Agilent Intuvo 9000. *Agilent Technologies Application Note*, publication number 5991-8990EN, **2018**.
3. V. Abercrombie, L. Provoost. Increased Thermal Stability and Maximum Temperature of the Agilent J&W DB-HeavyWAX Column. *Agilent Technologies Application Note*, publication number 5991-9035EN, **2018**.

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Printed in the USA, July 12, 2018
5994-0062EN