Technical Overview

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Examination of 624-type GC Column Phases and its Performance for Amine Analysis

Abstract

Agilent J&W VF-624ms GC columns (low-bleed 6% cyanopropyl/phenyl, 94% polydimethylsiloxane) were compared to other 624-type GC columns from other manufactures with respect to their chromatographic behaviors for active amine compounds. Superior performance for active basic compounds was observed on the VF-624ms columns depicted as improved in peak shape characteristics that allows for more accurate and reliable analytical results.

Introduction

The analysis of active and basic compounds, such as amines, by gas chromatography can be challenging if not using the proper inertness throughout the flow path. When working with active compounds, it is important to select all parts within the flow path that will provide the most inertness, to ensure sharp, symmetrical peaks and maintain sensitivity.[1] Even with similar types of phases, there can be variation in inertness for difficult compounds such as amines, where peak deformations, such as peak tailing and complete adsorption, can occur. In this technical overview, active basic compounds were examined on the Agilent J&W VF-624ms GC column, and other 624-type columns from three different vendors, to compare the inertness toward basic compounds at low concentrations. Both qualitative (peak shape) and quantitative (USP tailing factor, T_{f5}) data were used to assess the activity of the GC columns.

Experimental

Materials and methods

An Agilent 8890 GC/FID equipped with a multimode inlet, and an Agilent 7693A automatic liquid sampler with Agilent OpenLab ChemStation software was used for GC/FID experiments. The method was set up to perform dual injections. GC and FID conditions are listed in Table 1, and flow path supplies are listed in Table 2.

Table 1. GC and FID conditions.

GC Conditions	
Column	Agilent J&W VF-624ms, 30 m × 0.32 mm × 1.8 µm (p/n CP9104) Brand X-624ms column, 30 m × 0.32 mm × 1.8 µm Brand Y-624ms column, 30 m × 0.32 mm × 1.8 µm Brand Z-624 plus column, 30 m × 0.32 mm × 1.8 µm
Carrier	Helium, constant flow, 2.2 mL/min
Oven	40 °C (3.0 min), ramp 10 °C/min to 120 °C (4.0 min)
Inlet	S/SL inlet, split mode, 270 °C, split ratio 50:1, injection volume 0.2 µL
Inlet Liner	Inlet liner, split, single taper, glass wool, deactivated, low pressure drop (part number 5183-4702) Inlet liner, Ultra Inert, split, low pressure drop, glass wool (part number 5190-2295)
GC/FID	Agilent 8890 GC equipped with FID
Sampler	Agilent 7693A automatic liquid sampler
FID Conditions	
Temperature	300 °C
Hydrogen	30 mL/min
Air	400 mL/min
Column Flow + Make-Up Gas	25 mL/min

Table 2. Flow path supplies.

Parameter	Value
Septum	Inlet septa, bleed and temperature optimized (BTO), nonstick, 11 mm (part number 5183-4757, 50/pk)
Vials	Vial, screw top, amber, write-on spot, certified, 2 mL (part number 5182-0716, 100/pk)
Vial Inserts	Vial insert, 250 µL, deactivated glass with polymer feet, (part number 5181-8872, 100/pk)
Vial Caps	Cap, screw, blue, PTFE/red silicone septa, 9 mm (part number 5185-5820, 500/pk)
Inlet/FID	Ferrule, 0.5 mm id, 15% graphite/85% Vespel (part number 5062-3514, 10/pk) Column nut, collared, self-tightening, inlet/detector (part number G3440-81011)

Standards preparation

Individual standards were purchased from Sigma Aldrich and prepared to concentrations ranging from 100 to 10,000 µg/mL in methanol.

Results and discussion

Comparison of 624-type GC column phases

Standards of triethylamine (TEA) and diethylamine (DEA), prepared in methanol, were analyzed and compared on the Agilent J&W VF-624ms GC column, as well as three 624-type columns from alternative brands. Figure 1 shows a sample of 800 pg of DEA and TEA analyzed on a VF-624ms. The tailing factor for triethylamine was calculated to be 1.09, indicating that the VF-624ms phase has good inertness for this compound. Diethylamine was found to be a more active compound for the VF-624ms phase but still maintained a tailing factor of 2.25, indicating that this phase is still adequate to analyze this compound.

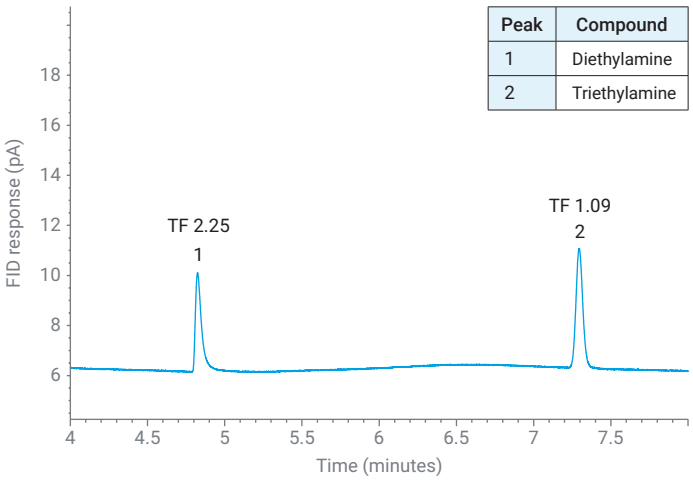

Figure 1. Standard of 800 pg on a column of DEA and TEA, analyzed on an Agilent J&W VF-624ms GC column. TF indicates trailing factor.

Figure 2 shows how the VF-624ms compared to three other 624-type columns when analyzing the same standard of 800 pg on column of DEA and TEA. While the brand X-624ms column had a similar tailing factor to the VF-624ms for TEA, it had an increased tailing factor for DEA at 3.68. This indicates that the brand X-624ms had slightly more activity for amines than the VF-624ms. The brand Y-624ms column had a greater tailing factor for TEA, and also a large tailing factor for DEA at 5.68. With the brand Z-624 plus GC column, the DEA was not detected because it had been fully adsorbed by the column. The increased tailing factor of TEA at 6.64 confirms the overall increased activity of the brand Z-624 plus column for the analysis of amines.

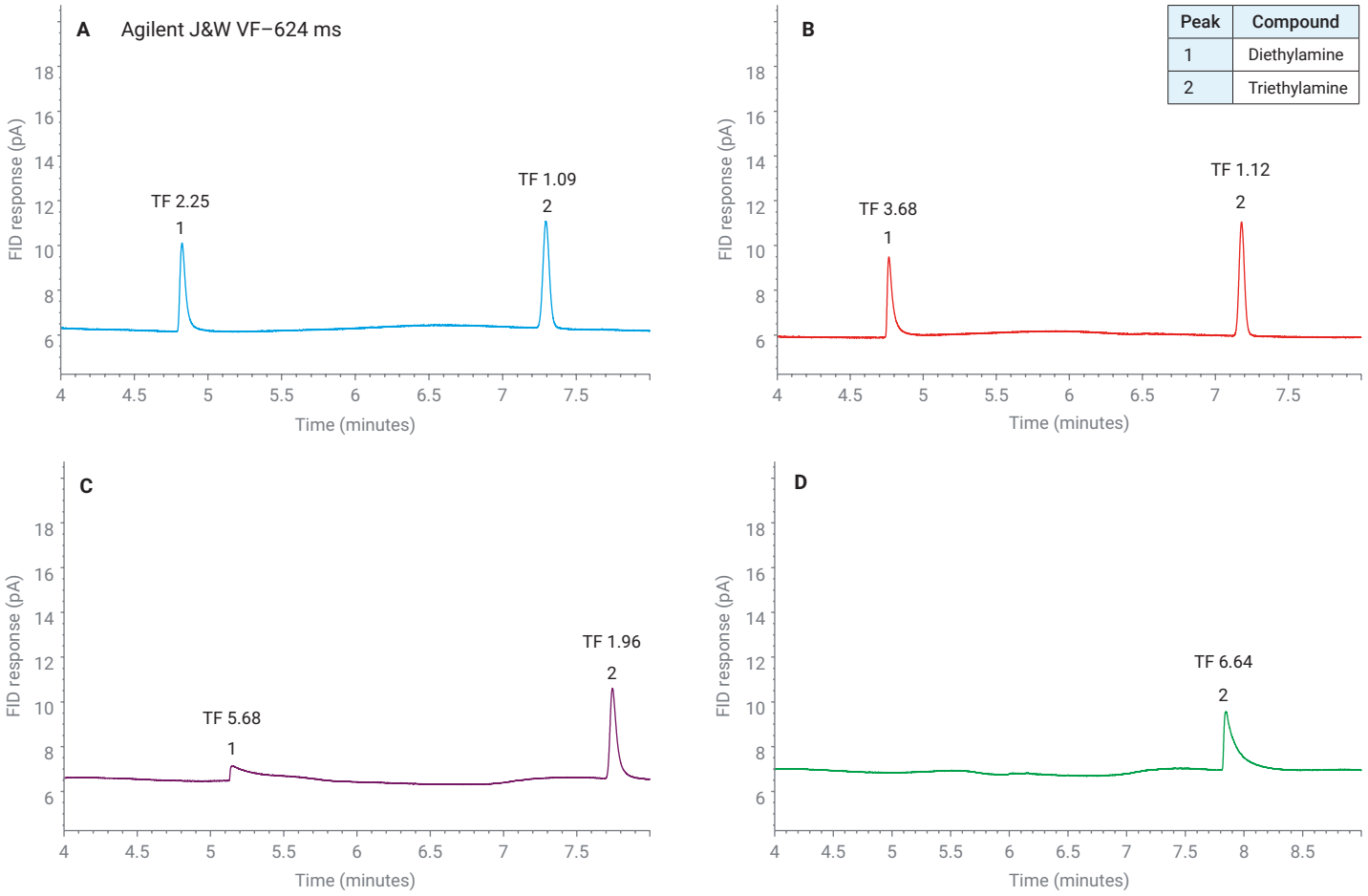

Figure 2. A standard of 800 pg on column of DEA and TEA analyzed on: (A) Agilent J&W VF-624ms GC column, (B) brand X-624ms GC column, (C) brand Y-624ms GC column, and (D) brand Z-624 plus GC column. TF indicates trailing factor.

Calibration and linearity

Calibration standards of TEA and DEA were prepared ranging from 100 to 4,000 pg on column, and analyzed on the VF-624ms GC column. Figures 3 and 4 demonstrate the good linearity for both TEA and DEA on the VF-624ms GC column, respectively.

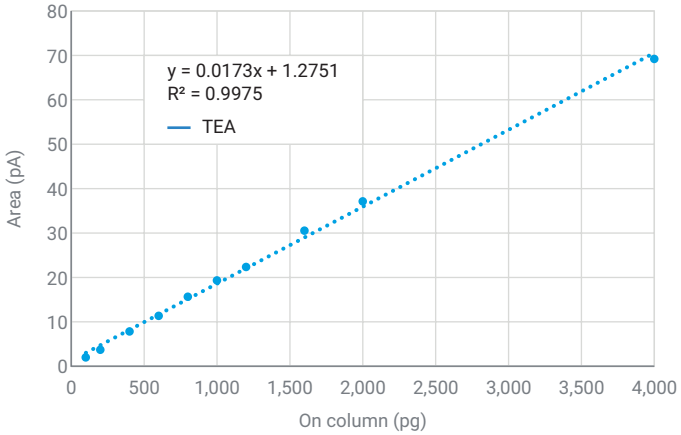

Figure 3. Calibration of TEA in MeOH, analyzed on the Agilent J&W VF-624ms GC column.

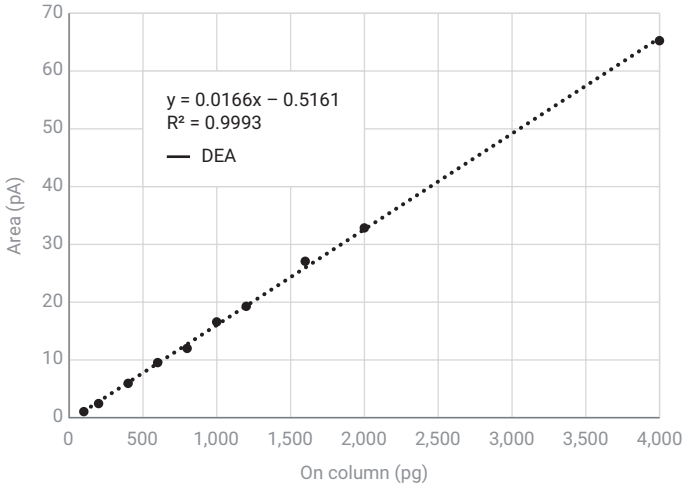

Figure 4. Calibration of DEA in MeOH, analyzed on the Agilent J&W VF-624ms GC column.

Conclusion

The Agilent J&W VF-624ms GC column demonstrated superior performance in the analysis of active basic compounds, in comparison to three similar competitor columns. The increased inertness resulted in better overall peak shape, as calculated by the tailing factor for TEA and DEA, and an increase in sensitivity. This increase in sensitivity allows for a lower detection limit and a more linear and accurate calibration range.

References

1. Berry, J.; Lynam, K.; Cai, C.; Zou, Y. Competitive Column Inertness Analysis with Active Basic Compounds. *Agilent Technologies application note*, publication number 5991-4626EN, **2014**

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