

Finetech Research and Innovation Corporation

CE
2460



Finetech Septa (PTFE & Silicone)

Physical and Chemical Analysis of
Septa and Caps



Taipei: +866-2-29964887
Taichung (Head Office): +886-4-23873526
Vietnam: +84-8-35030769
China: +86-511-86206672

patrick@finetech-filter.com (General Sales Manager)
sales03@finetech-filter.com (North America)
sales02@finetech-filter.com (South America, Europe)
sales01@finetech-filter.com (Africa, Asia, Australia)
mona@finetech-filter.com (China, Hong Kong, Singapore)



Website



LINE

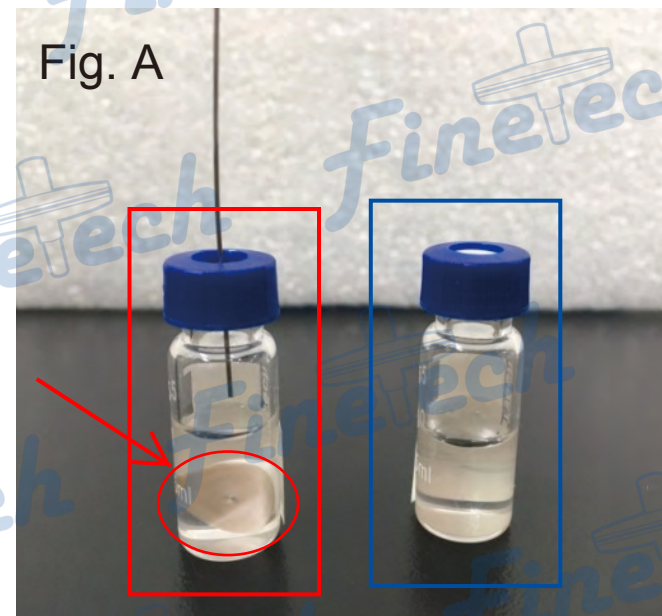
Finetech Septa (PTFE & Silicone)

Finetech's Advantage Over the Competition

*Finetech's septa have undergone extensive physical and chemical tests; demonstrating both durability and low bleed performance.

*High quality Teflon material imported from Europe and cut to precise specifications. After multiple uses, septa **will not easily dislodge** into vials as can be seen in cheaper brands (Fig. A) and puncture marks **stays sealed** (Fig. B).

*Finetech offers septa with quality comparable to European, American, and other international brands.



Brand A Finetech®

Fig. B



Brand A



Finetech®

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Chemical Testing Summary

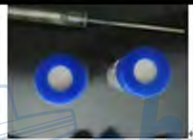
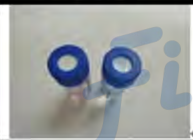
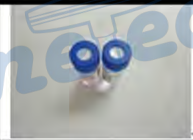
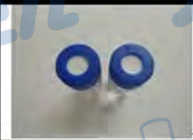


P50

Physical Testing

Physical Testing - F1-1 : Septa Puncture Appearance Test

- **Purpose:** Simulate the injection process during HPLC or GC analysis and observe the ability to reseal itself after multiple punctures.
- **Procedure:** Take 2 pcs of Finetech's sample vials and caps. Pierce the septa 5 times with a micro syringe. Observe the puncture mark and measure with vernier caliper. Puncture mark should not exceed $0.15\text{mm} \pm 0.1\text{mm}$.
- **Result:** The PTFE/silicon septa pierced 5 times with a needle pass the test with puncture marks of less than 0.15mm .

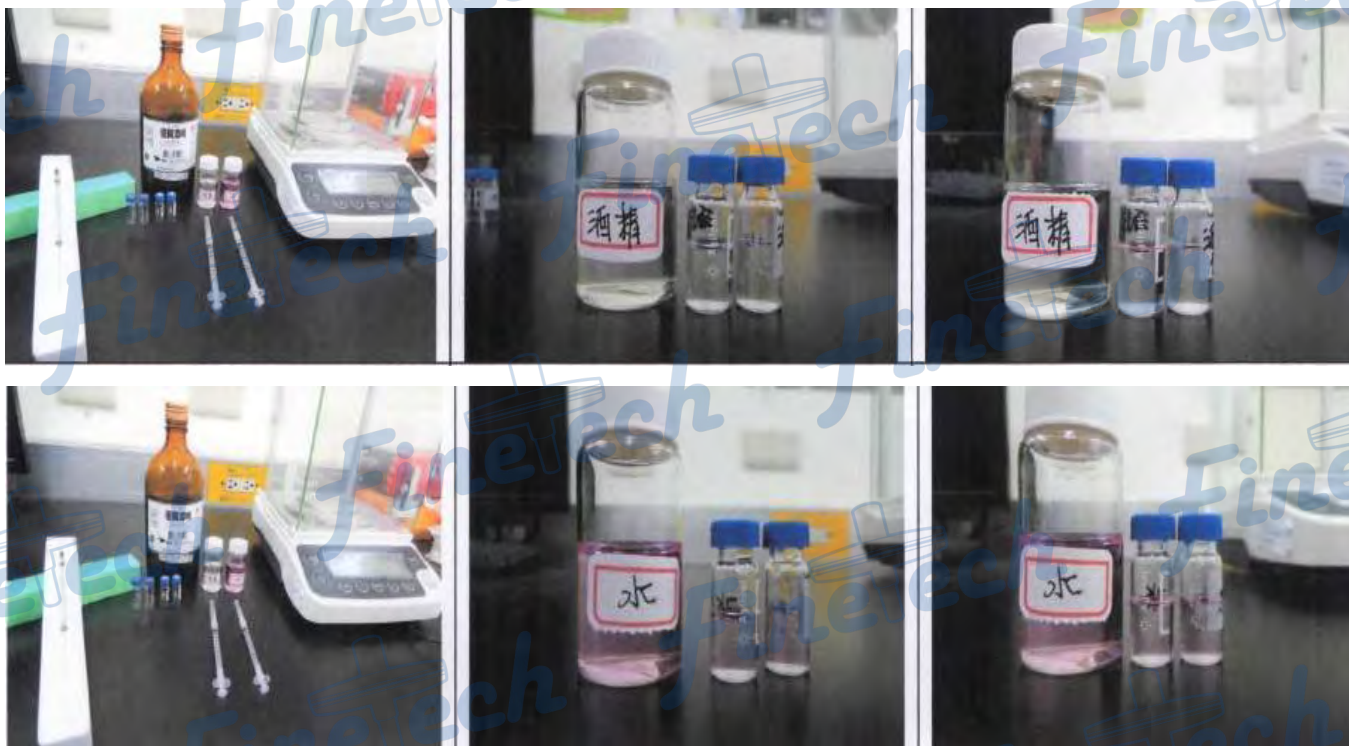


Start	1 st Puncture	2 nd Puncture	3 rd Puncture	4 th Puncture	5 th Puncture
					
	Pass	Pass	Pass	Pass	Pass

Physical Testing - F1-2 : Self-Sealing Septa Test

Procedure :

1. Fill Brand A and Finetech's sample vials with two different solvents, alcohol and water. Puncture septa 5 times, weighed with lab balance, and record amount lost due to volatilization.
2. Use syringe to inject 1 mL of alcohol and water into the vials (left: Brand A, right: Finetech).
3. The following photos shows the results of puncturing the septa 5 times at 30 minute intervals.



4. Data Results:

Alcohol – Vial Weight

	No Puncture	1st Puncture	2nd Puncture	3rd Puncture	4th Puncture	5th Puncture	Amount Lost (g/%)
Brand A	3.154 g	3.154 g	3.154 g	3.154 g	3.153 g	3.152 g	0.002g/0.06%
Finetech	3.507 g	3.506 g	3.506 g	3.506 g	3.505 g	3.504 g	0.003g/0.08%

Water – Vial Weight

	No Puncture	1st Puncture	2nd Puncture	3rd Puncture	4th Puncture	5th Puncture	Amount Lost (g/%)
Brand A	3.337 g	3.337 g	3.336 g	3.336 g	3.335 g	3.335 g	0.002g/0.06%
Finetech	3.745 g	3.745 g	3.745 g	3.754 g	3.744 g	3.744 g	0.001g/0.03%

Conclusion: After 5 punctures, 0.08% of the alcohol and 0.03% of the water is lost in Finetech's vials, which is much lower than the checkpoint criteria of $1.5\% \pm 0.5\%$. The results show that Finetech's septa passes the self-sealing test after multiple punctures.

Physical Testing - F2-1 : Temperature Test

Purpose: Observe the effects of subjecting septa to high, low, and room temperature.

Procedure:

1. Take 10 pcs of Finetech's septa.
2. Place septa in oven at 100°C, freezer at -4°C, and room temperature at 25°C ± 2°C for 6 hours each.

High Temperature: 100°C Oven



Results:

Septa	1	2	3	4	5	6	7	8	9	10
Peeling or Separation	X	X	X	X	X	X	X	X	X	X
Degradation	X	X	X	X	X	X	X	X	X	X
Changed Puncturability	X	X	X	X	X	X	X	X	X	X
Sealing Ability	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓

Room Temperature: 25°C ± 2°C



Low Temperature: -4°C Freezer



Conclusion:

Finetech's PTFE (Teflon) and silicon septa were tested at high, low, and room temperatures for 6 hours. The results show no significant effects to the septa in this temperature range.

Physical Testing - F2-2 : Peel Strength Test

Purpose : To test the adhesion strength between the PTFE and silicon layer. Results should exceed 19.6 N (about 2 kgf).

Procedure : After bonding PTFE and silicon, let stand for 1 minute. Take 10 pcs and test the peel strength with tensile testing machine set at 50mm/min load rate.



Results:

	1	2	3	4	5	6	7	8	9	10
Peel Strength (N)	28	23.2	21	26.3	38.5	24.2	27.9	32	27.1	28.4

Conclusion: The peel strength test conducted on the PTFE/silicon septa material shows to exceed the criteria of 19.6 newtons.

Physical Testing Summary

Test	Standard	Result
Puncture	Puncture marks smaller than 0.15mm±0.05mm.	Pass
Self-sealing	Amount lost does not exceed 1.5%±0.5% of total weight.	Pass
Peel Strength	Load rate set at 50mm/min, force exceeds 19.6 N.	Pass
Temperature	No degradation from 6 hours of exposure at temperatures of -4°C, 25°C ± 2°C, or 100°C.	Pass

Conclusion: Based on the results of the tests, the septa material conforms with or exceeds the standards developed for this material.

Chemical Testing

Chemical Testing

Bleed Test of Septa with GC Fixed Syringes

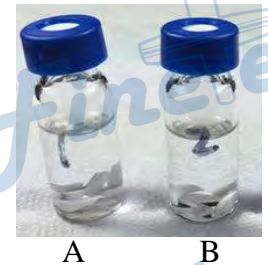
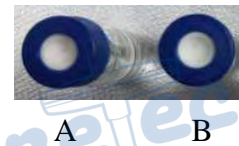
Experiment Goal

Bleed test of septa by using GC Fixed Syringes.

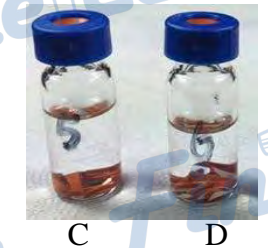
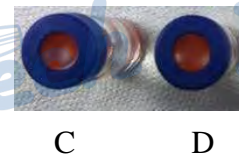
Experiment Steps

1. Different brands of septa were mounted in the vial caps.
2. 1 mL of MeOH / DCM (1: 1) solvent were added to the vials.
3. Spike vials A and C with 100 ppm of Thiram.
4. Septa were cut into smaller pieces and placed into vials (50mg).
5. After 24 hours, the solvents were analyzed by GC-FID (n = 1.6.9.18).

Finetech



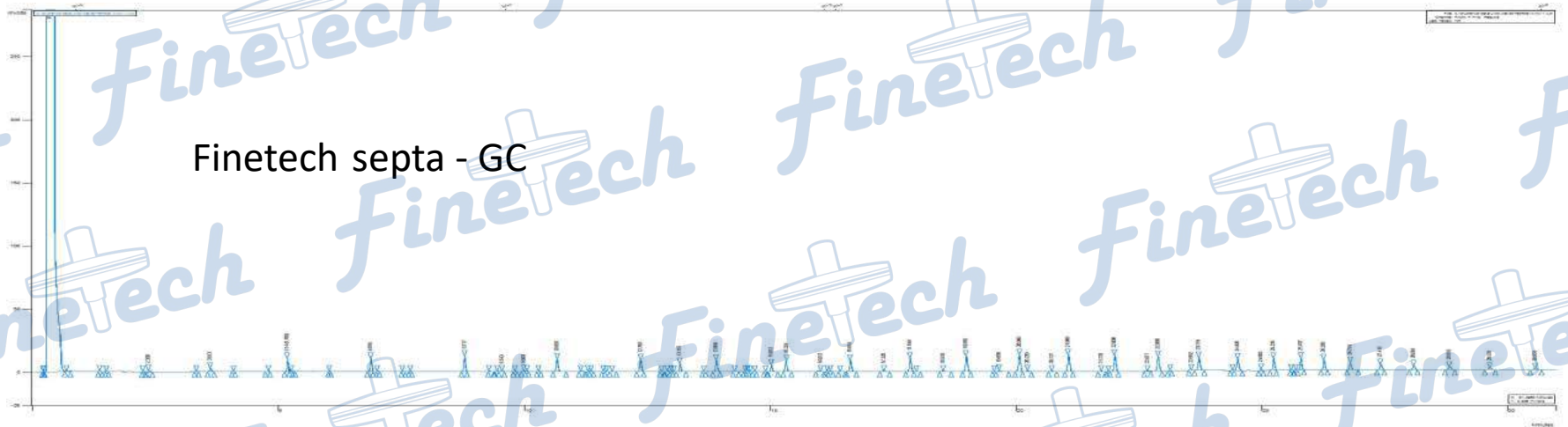
Brand A



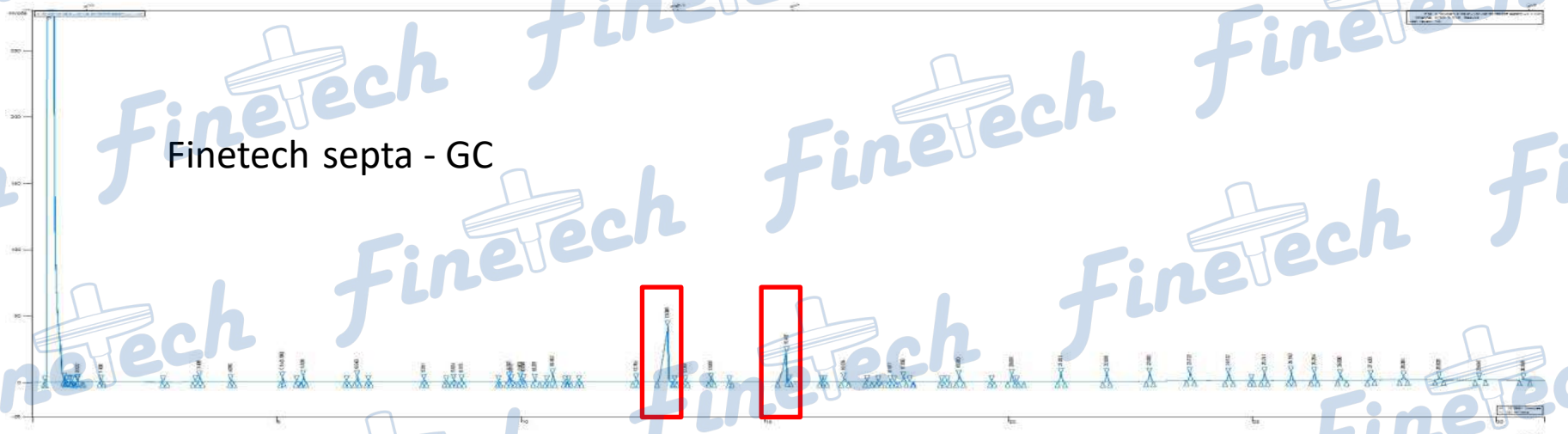
Experimental Conditions

Column	BR-5ms FS 15 m x 0.25 mm ID x 0.25 μ m
Detection	FID
H ₂ flow	30 ml/min
Air flow	400 ml/min
Makeup flow	29 ml/min (He)
Injection size	1.0 μ L
Injection temperature	250 °C
Column oven	50 °C hold for 3 minutes, 50-100 °C @ 25 °C/min, 100-300 °C @ 10 °C/min, , 300-350 °C @ 25 °C/min (hold for minutes)
Pressure	25 psi for 27 minutes, 50 psi from 27-31 minutes

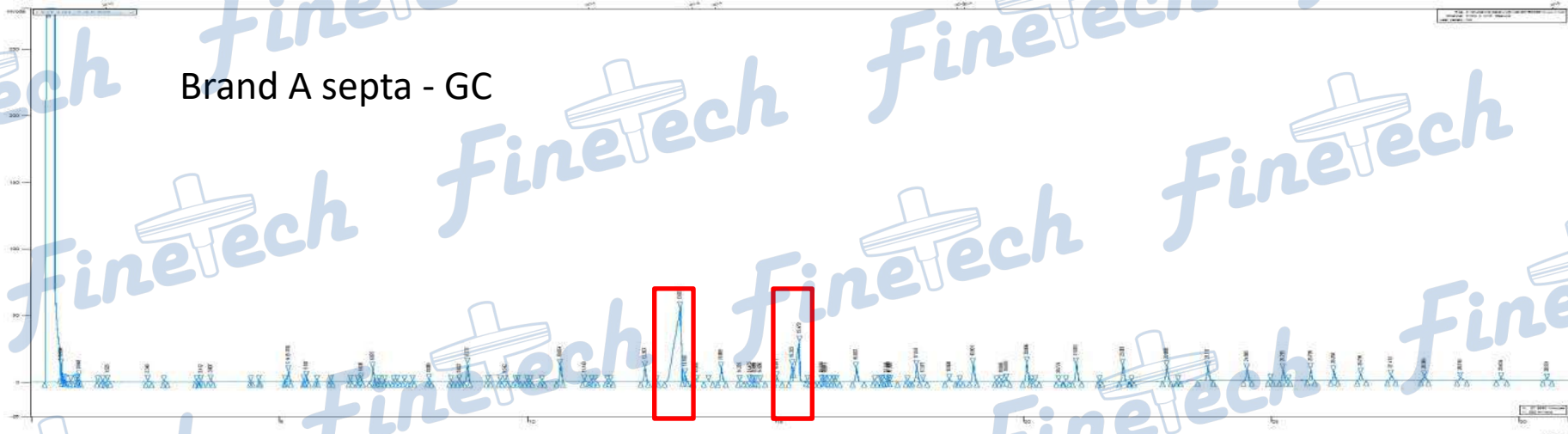
Without 100 ppm Thiram



Added 100 ppm Thiram



Finetech septa - GC



Brand A septa - GC

Note : Red area =Thiram area

Conclusion

1. In the non-spiked samples, the signal area of Finetech's dissolved septa material was more than 10% larger than Brand A septa.

	Finetech	Brand A
Without Thiram	604126	255546

2. The peak area and retention time of the samples spiked with Thiram are shown in the following table. The retention time was not affected by the different brands. The results show that the peak area of Thiram from Finetech's septa is higher than Brand A.

Finetech				
	Retention Time	Peak Area	Retention Time	Peak Area
1	13.073	497635	15.478	143068
2	13.08	499009	15.473	131444
3	13.079	513208	15.473	129843
Average	13.07733	503284	15.47467	134785
RSD	0.003786	0.017131	0.002887	0.053551

Brand A				
	Retention Time	Peak Area	Retention Time	Peak Area
1	13.008	266645	15.437	98695
2	12.998	247762	15.423	85314
3	12.994	237227	15.423	88068
Average	13	250544	15.42767	90692
RSD	0.007211	0.059491	0.008083	0.077912

Chemical Testing

Headspace Test of Gasket Dissolution (GC-FID)

Experimental goal

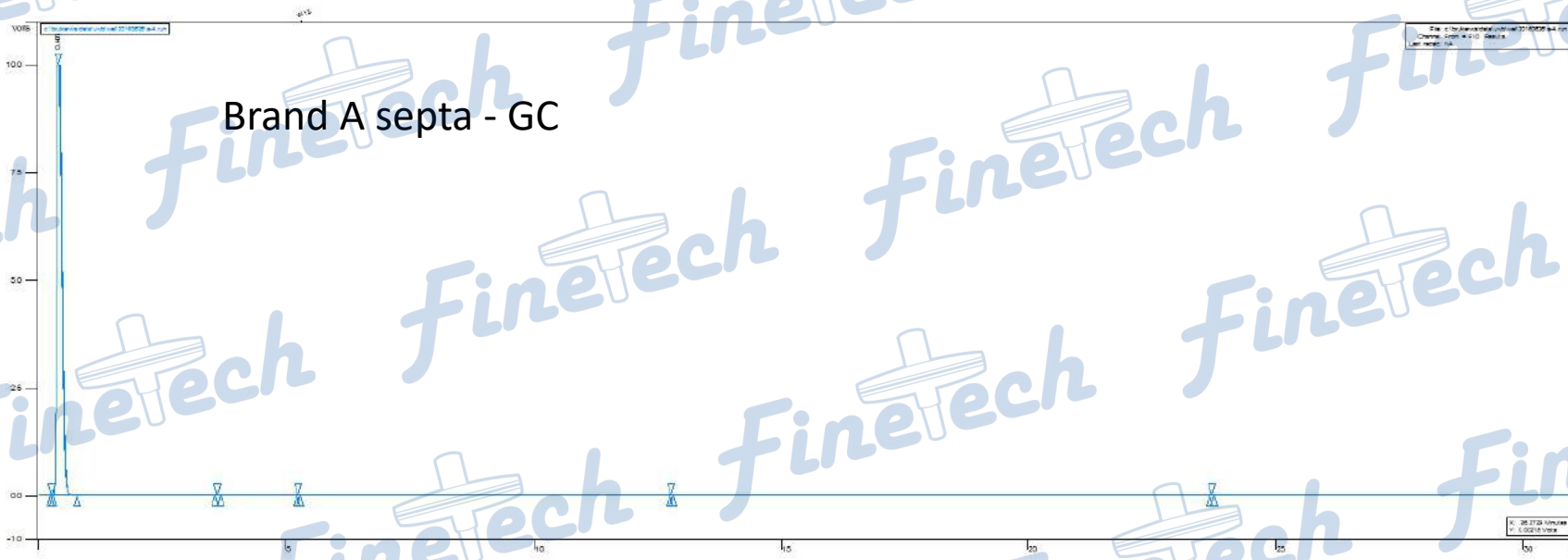
To test how the gaskets will decompose at high temperature.

Experimental steps

1. Install different brand septa onto vial caps.
2. Add 1 ml of DCM/MeOH(1:1) to the vials.
3. Add 50 mg of cut septa pieces to the vials and let stand for 24 hours.
4. The vials were heated by hot water bath to promote the formation of gases.
5. Use gastight needle to draw 1 μ L of gas from the headspace and inject into GC-FID
6. Use GC-FID to test for gasket components.



Finetech septa - GC



Brand A septa - GC

Conclusion

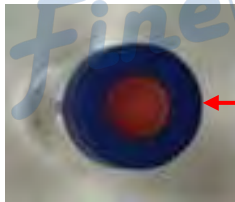
Headspace Test (GC—FID) :

The signal measured from Finetech's gasket is < 10% of Brand A.

Times	Finetech		Brand A	
	Retention Time	Area	Retention Time	Area
1	0.25	6762620	0.413	58790248
2	0.28	7635842	0.401	58417628
3	0.232	5967651	0.407	61962344
Average	0.254	6788704	0.407	59723407

Chemical Testing

Septa Composition Molecular Weight Test (GC-MS)



← 9 mm PTFE / Silicone septum

Experimental goal:

Test material to ensure there are only low levels of low molecular weight compounds.

Experimental steps:

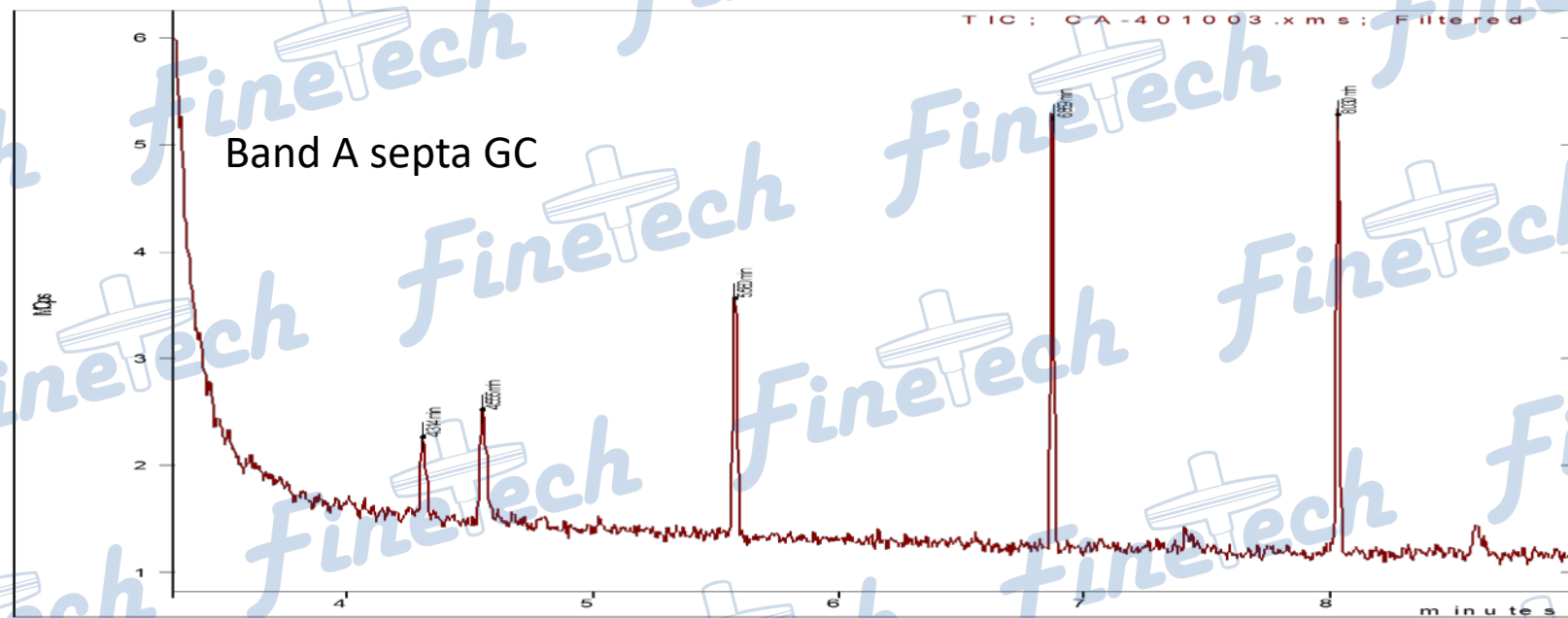
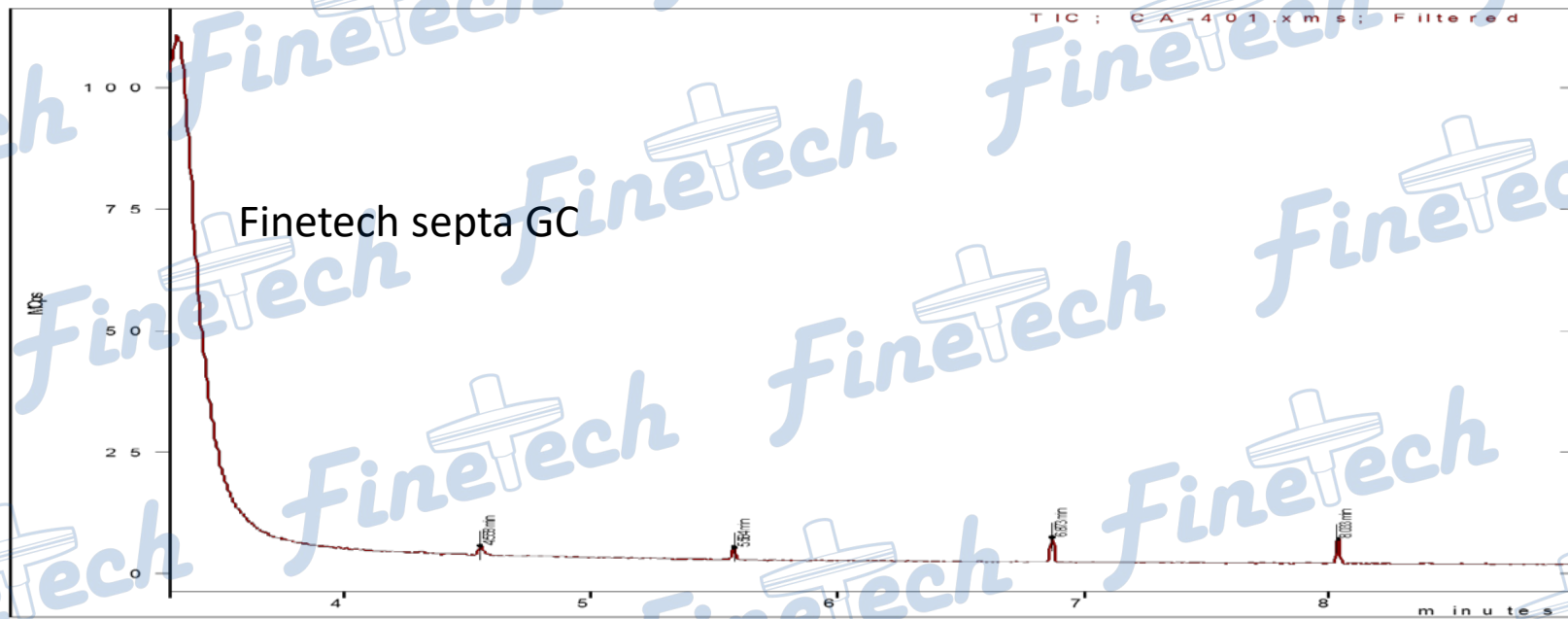
1. Attach PTFE/Silicone septa to caps.
2. Add 2 ml EtOH to the sample vial.
3. Analyze with GC-MS.
4. Set detector to $m/z < 250$.

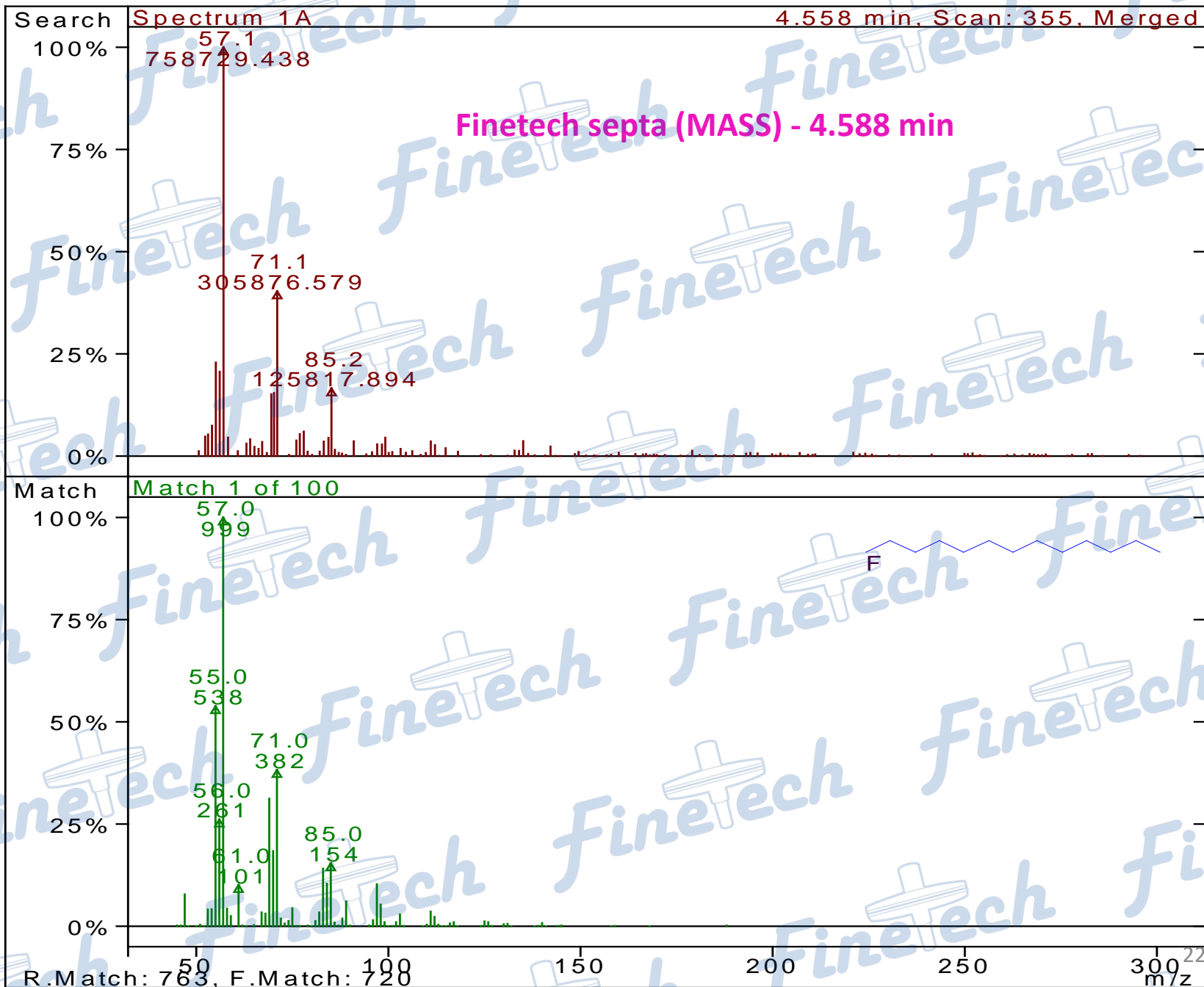


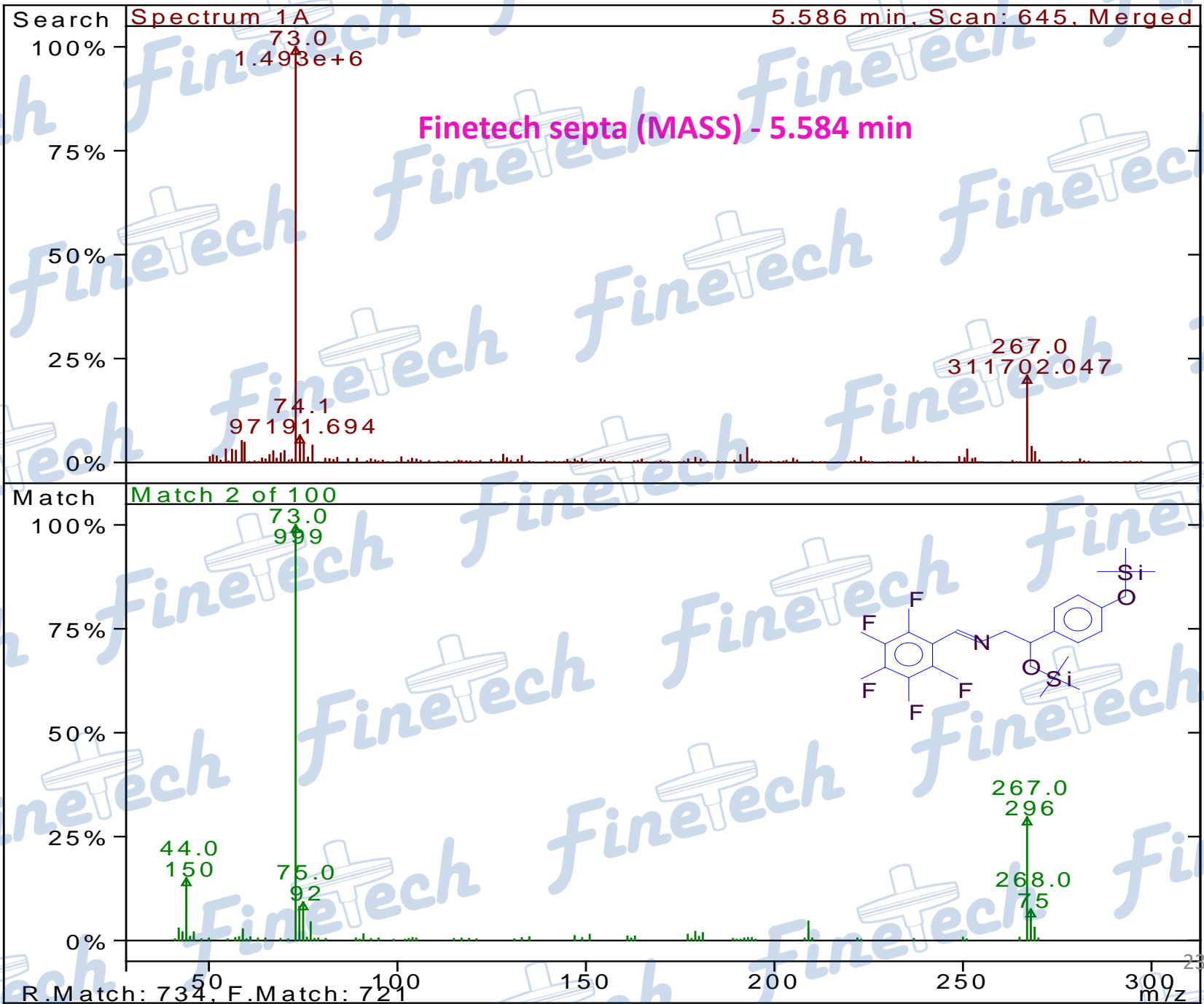
GC-MS Settings

Experimental Condition	
Column	BR-5ms FS 15 m x 0.25 mm ID x 0.25 um
Detection	MS
H ₂ flow	0 ml/min
Air flow	0 ml/min
Makeup flow	29 ml/min (He)
Injection size	1.0 uL
Injection temperature	250 °C
Column oven	50 °C hold for 0.5 minutes, 50-220 °C @ 20 °C/min
First mass	50.00 g / mole
Last mass	300.00 g / mole
Mass start time	3.3 min
Mass end time	9.0 min

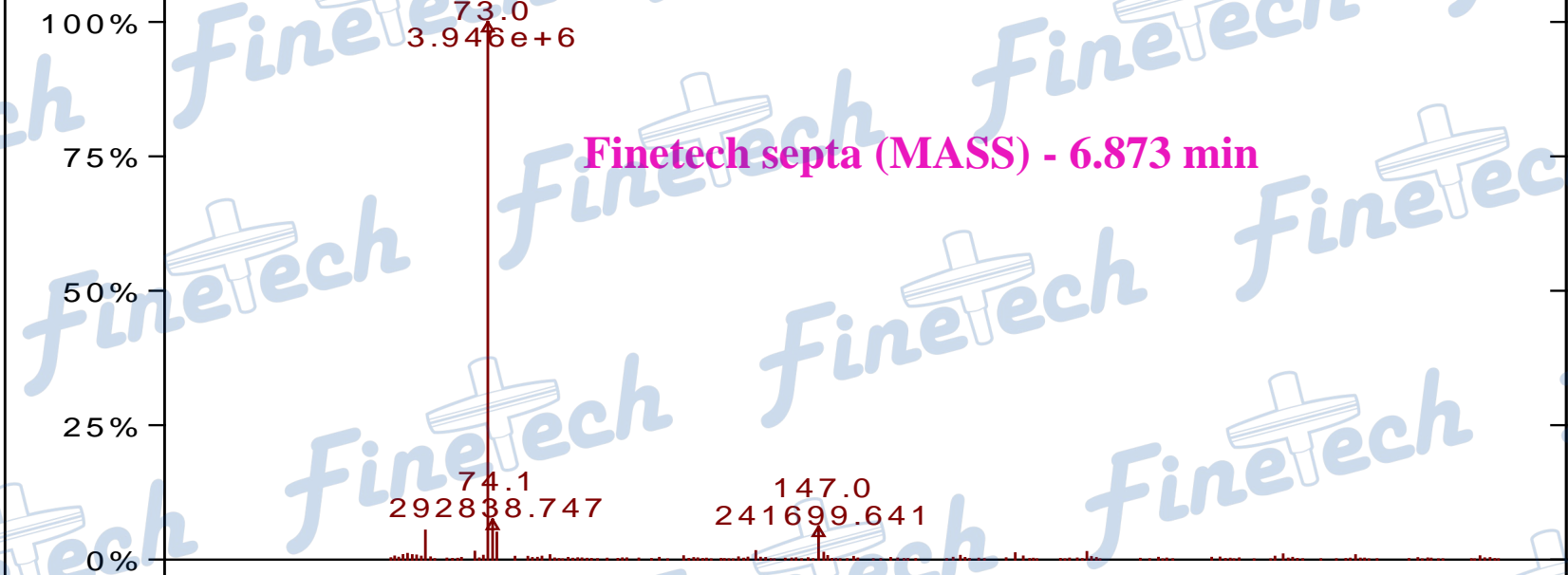
GC—MS





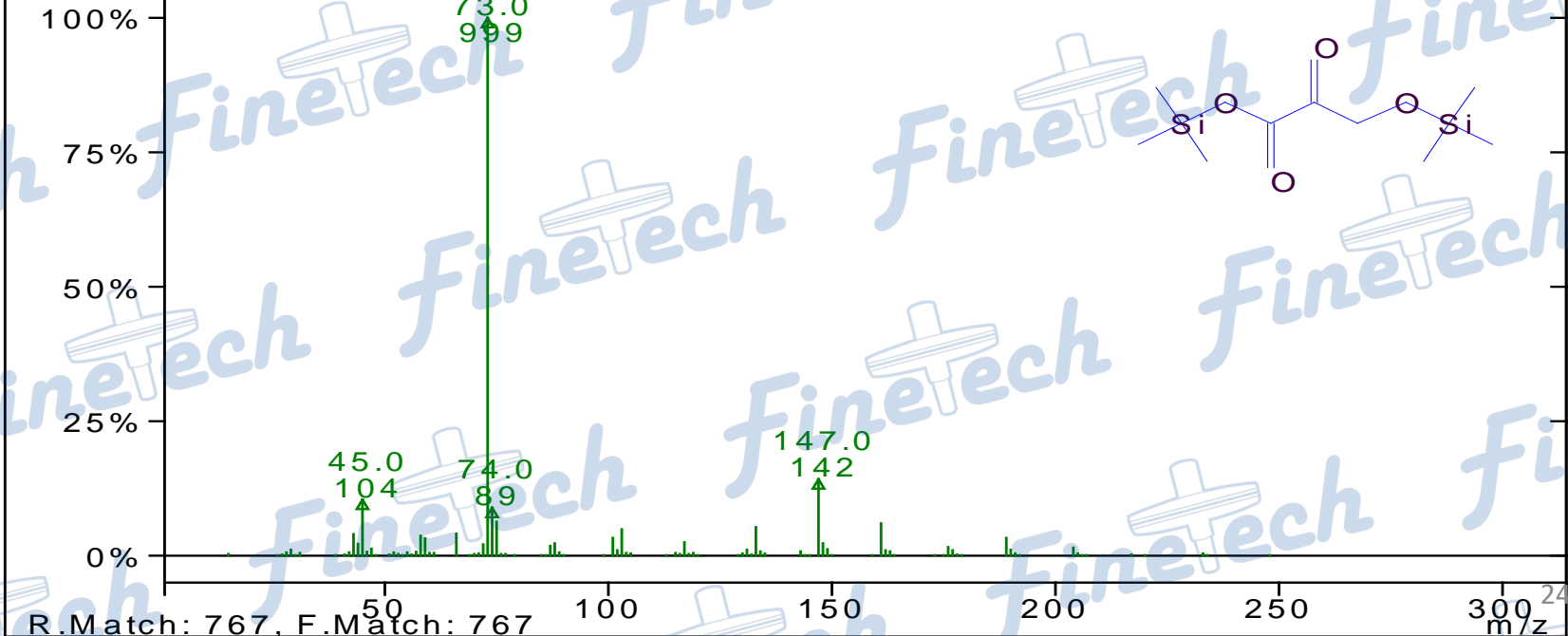


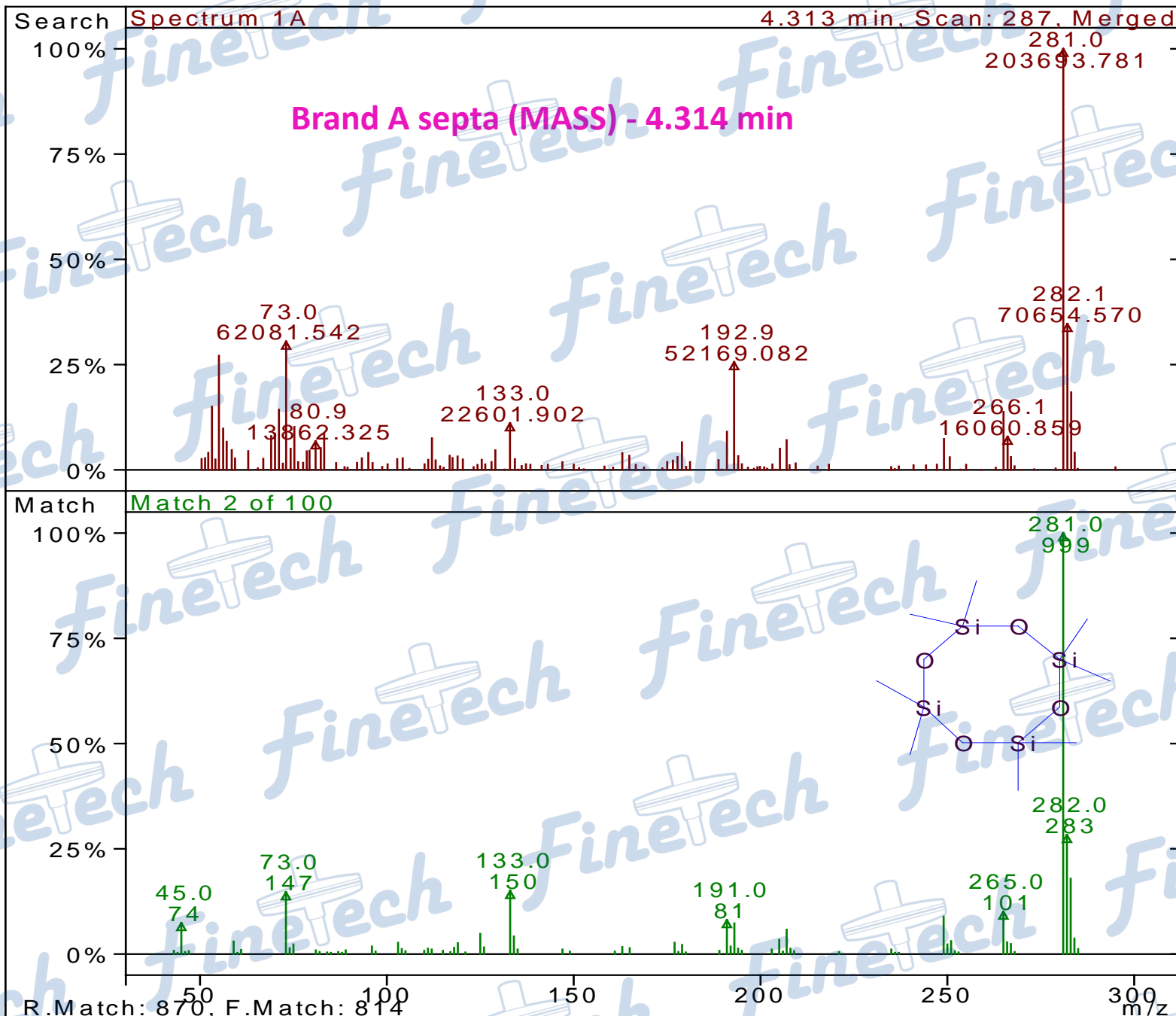
Search Spectrum 1A 6.872 min, Scan: 1009, Merged

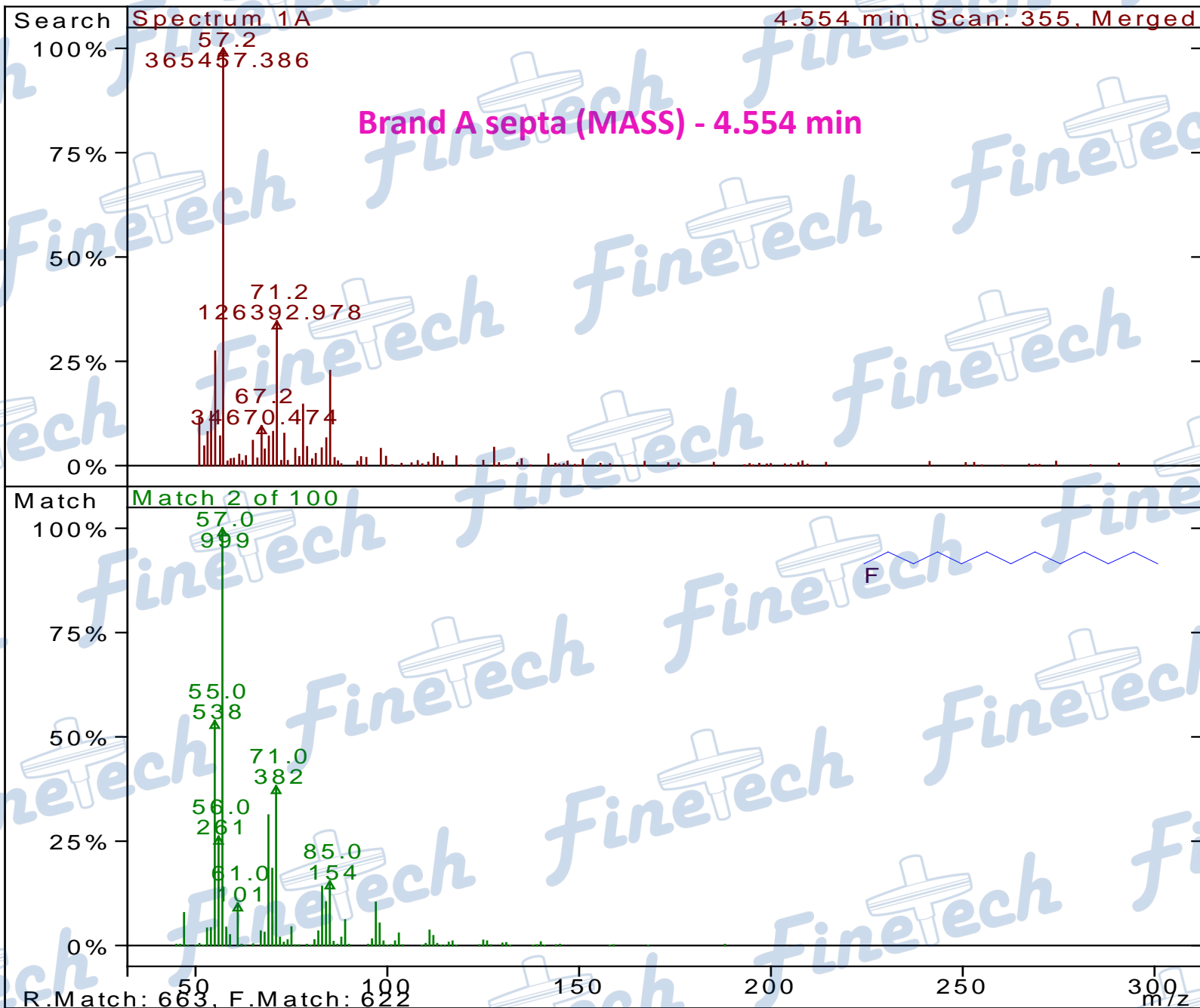


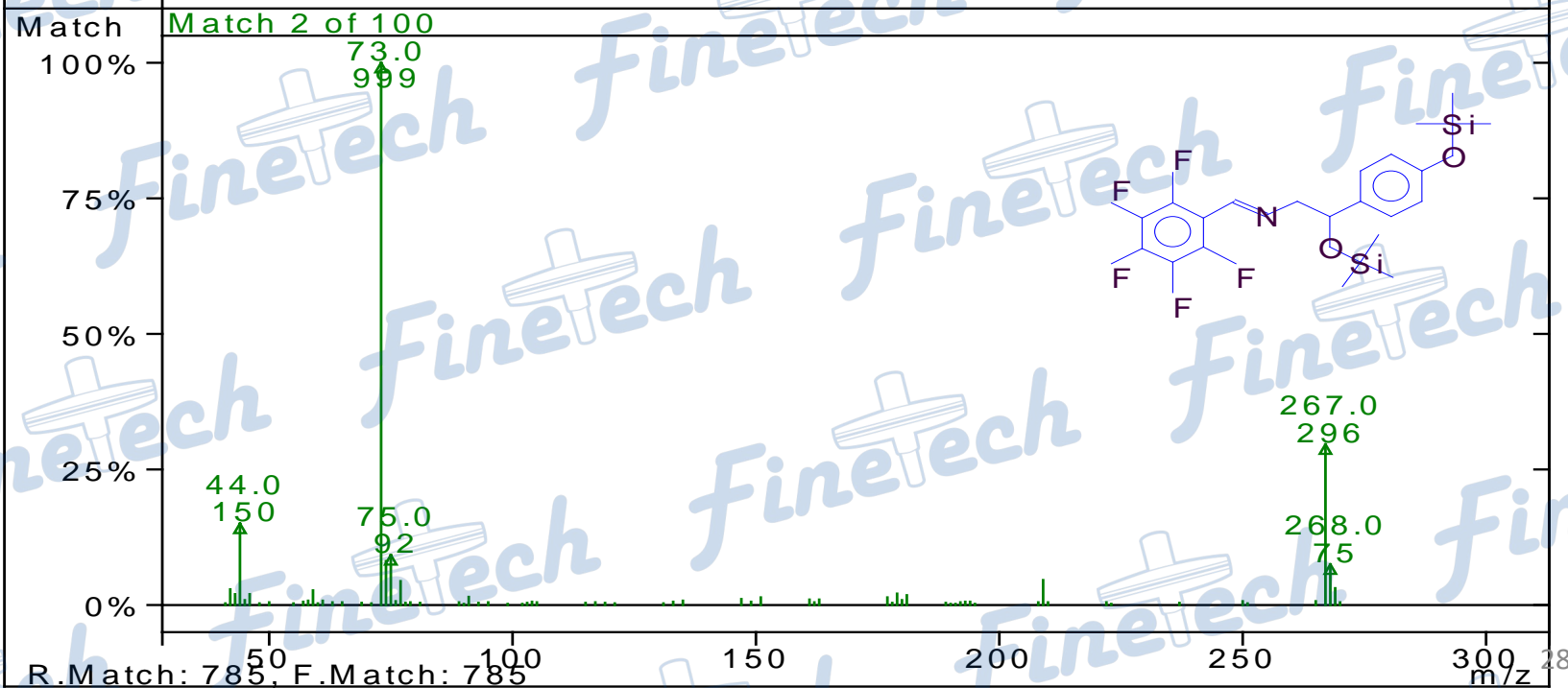
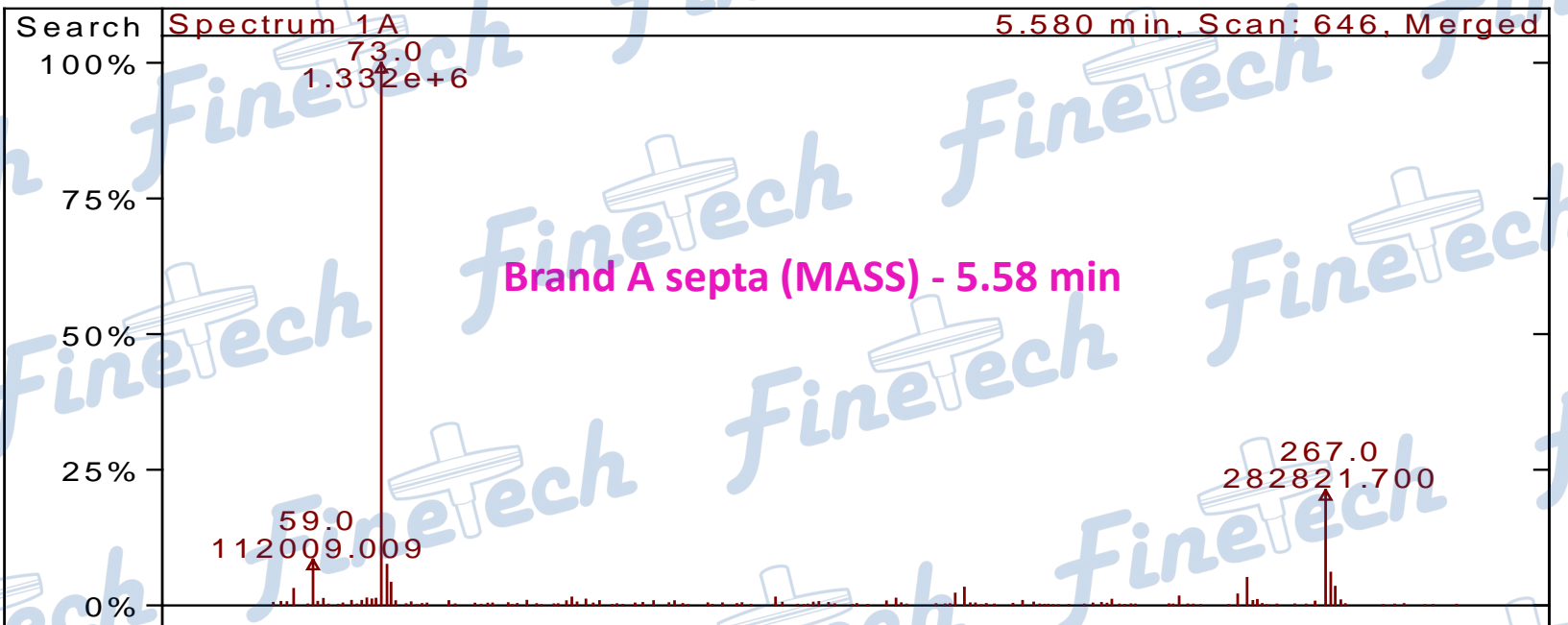
Finetech septa (MASS) - 6.873 min

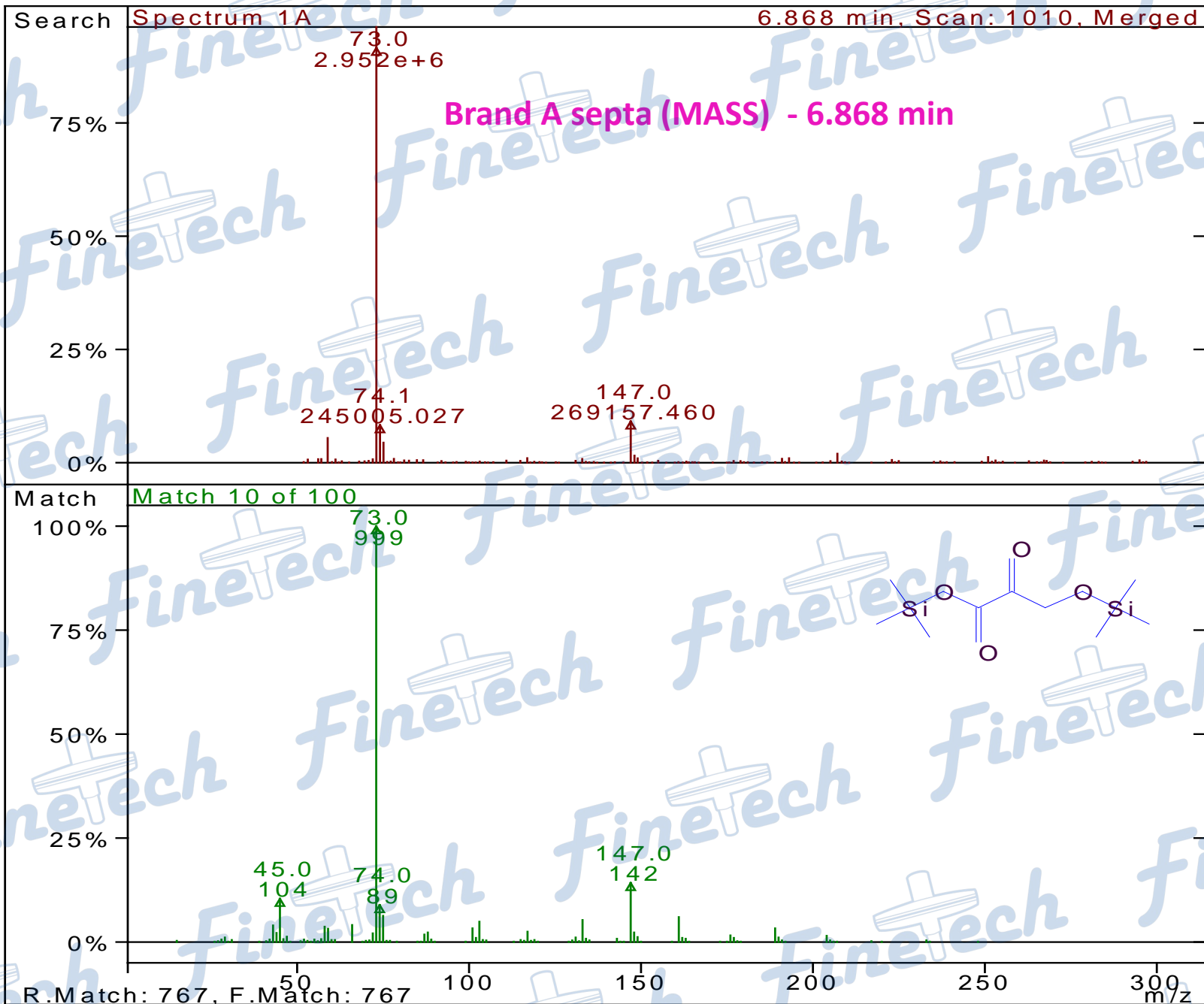
Match Match 11 of 100











Conclusion

Septa composition molecular weight test (GC-MS):

Finetech's septa materials' molecular composition with $m/z < 250$ is 10-15% less than Brand A and is suitable for analysis at $\mu\text{g/mL}$ concentration levels.



Chemical Testing

pH Test of Septa by UV-Visible

Experimental goal:

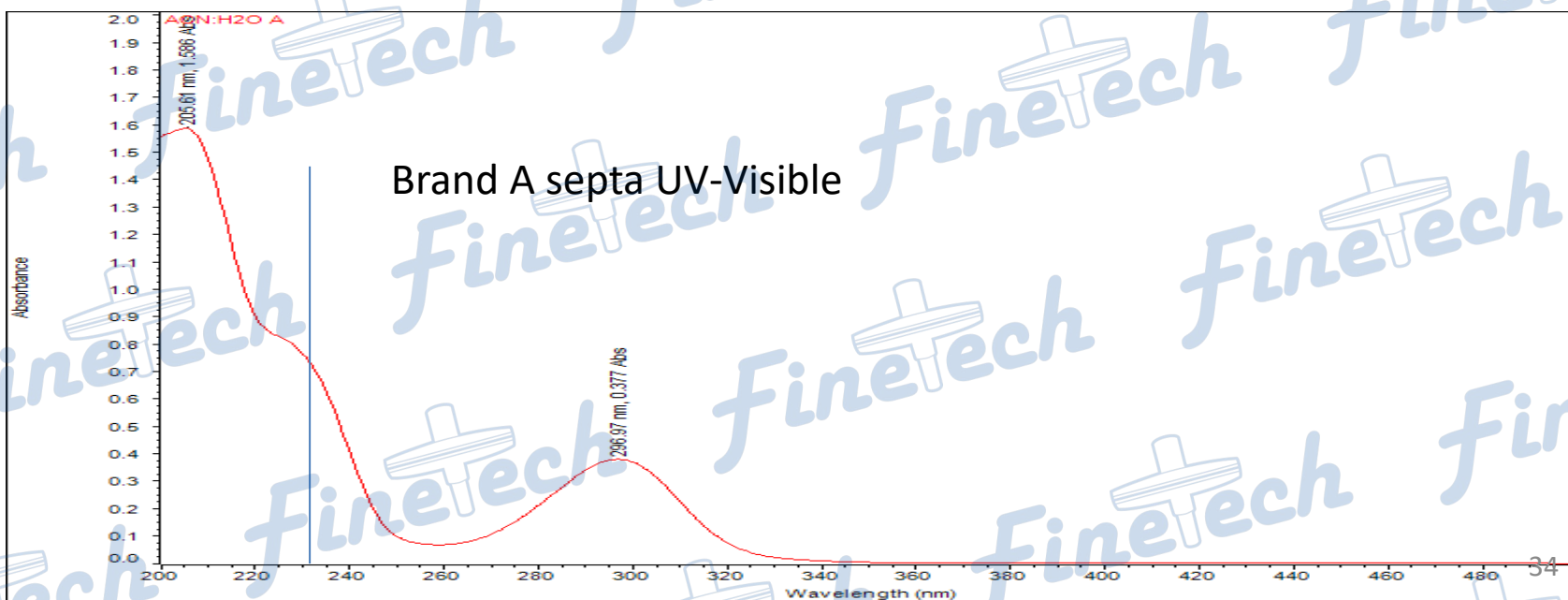
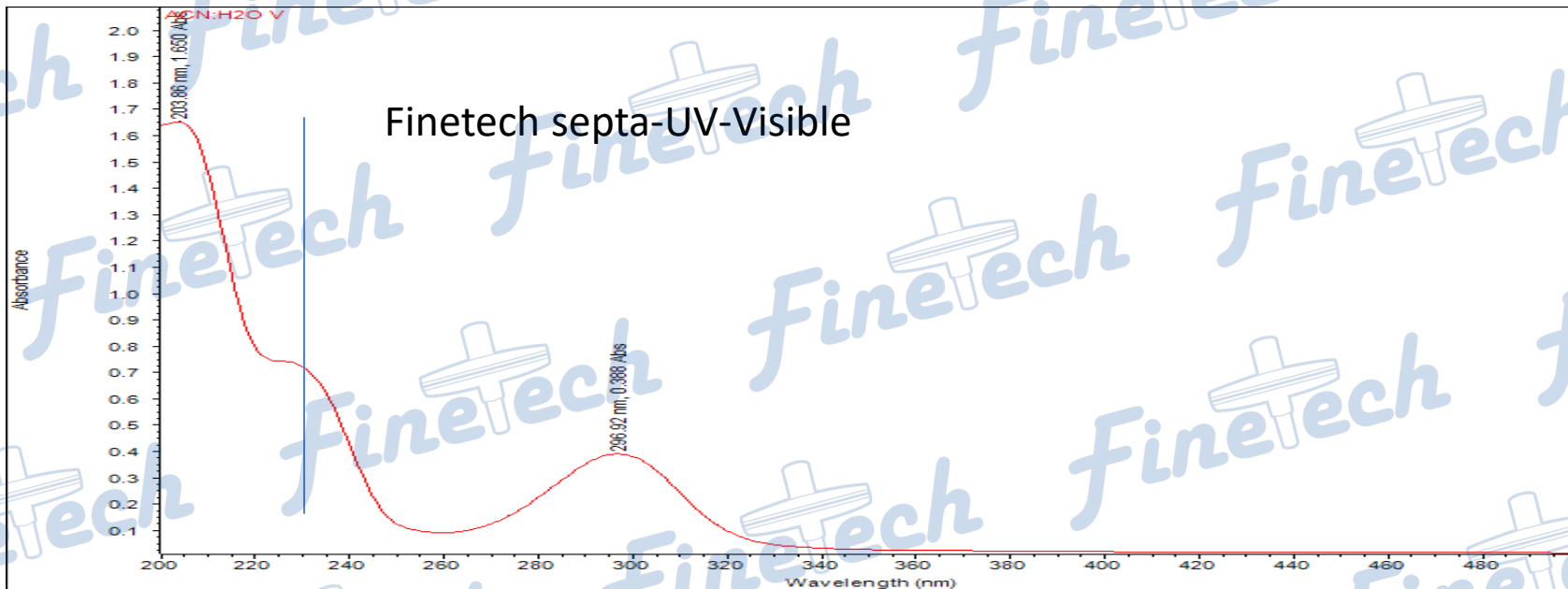
Bleed test of septa at different pH values by UV-Visible detection.

Experimental steps:

The different brands of septa were mounted on the vial caps. Salicylic acid 1×10^{-4} M was used as an internal standard. After 24 hours, septa were extracted (50 mg/mL) in phosphate buffer at different pH values and then analyzed by UV-Visible.

Experimental Condition	
Instrument	EVOLUTION 220, UV-Visible Spectrophotometer
Wavelength	190 nm-500 nm
Integration time	0.05 sec
Solvent	Phosphate Buffer





pH 1			
nm(Brand A)	Abs	nm(Finetech)	Abs
296.84	0.353	296.78	0.324
210.91	1.159	210.57	1.083
pH 4			
nm(Brand A)	Abs	nm(Finetech)	Abs
296.81	0.307	296.80	0.333
202.65	1.618	203.27	1.629
pH 7			
nm(Brand A)	Abs	nm(Finetech)	Abs
296.65	0.337	296.75	0.326
210.06	1.180	209.90	1.148
pH 9			
nm(Brand A)	Abs	nm(Finetech)	Abs
296.76	0.307	296.71	0.329
209.83	1.121	209.96	1.148
pH 11			
nm(Brand A)	Abs	nm(Finetech)	Abs
296.84	0.353	296.78	0.324
210.91	1.159	210.57	1.083

Experimental Results

1. After 24 hours, the signal intensity of salicylic acids was not interfered by the extracts from both septa.
2. The RSD% of signal intensity for both septa were less than 1.2%. Use of lower bleed materials reduces the potential for sample errors caused by septa bleed and improves overall reliability in phosphate buffer solution (pH 1.4-11.0) solvent conditions.

Chemical Testing

Bleed Test of Septa Using HPLC Fixed Syringes

Experimental goal:

Bleed test of septa by using HPLC fixed syringes.

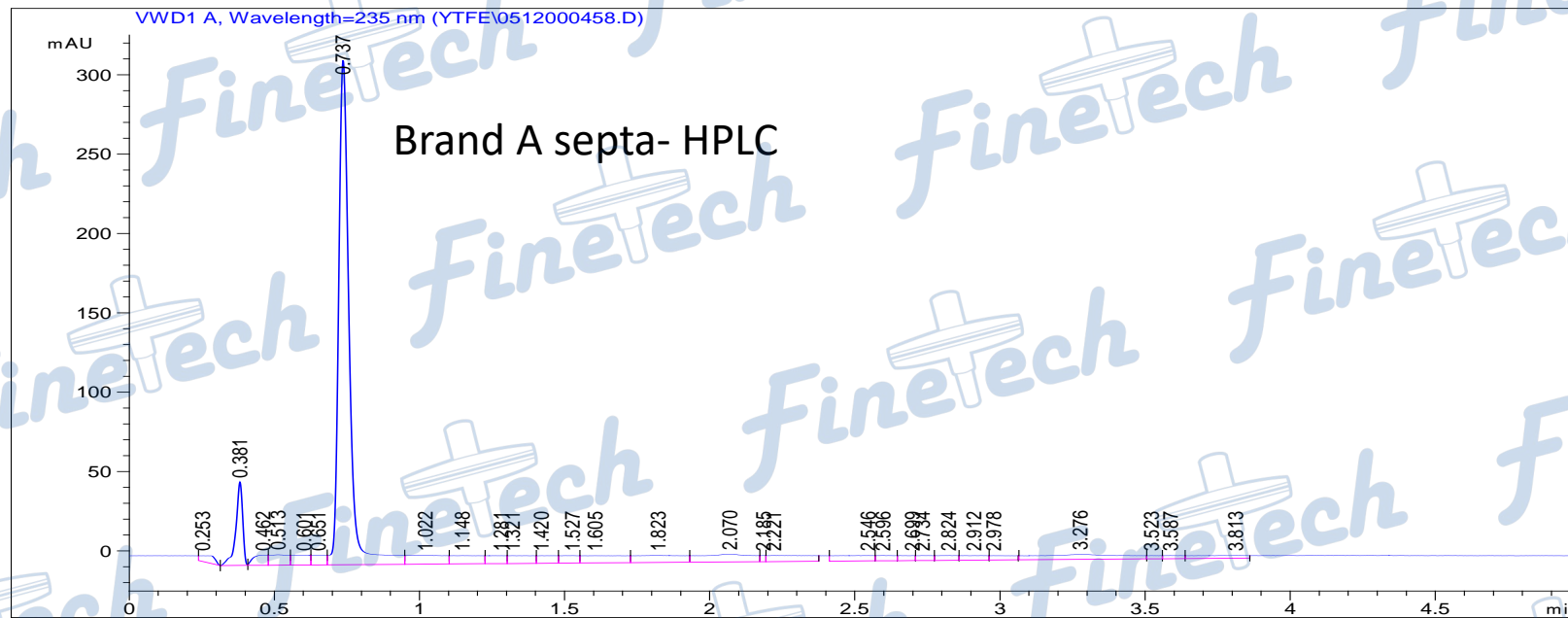
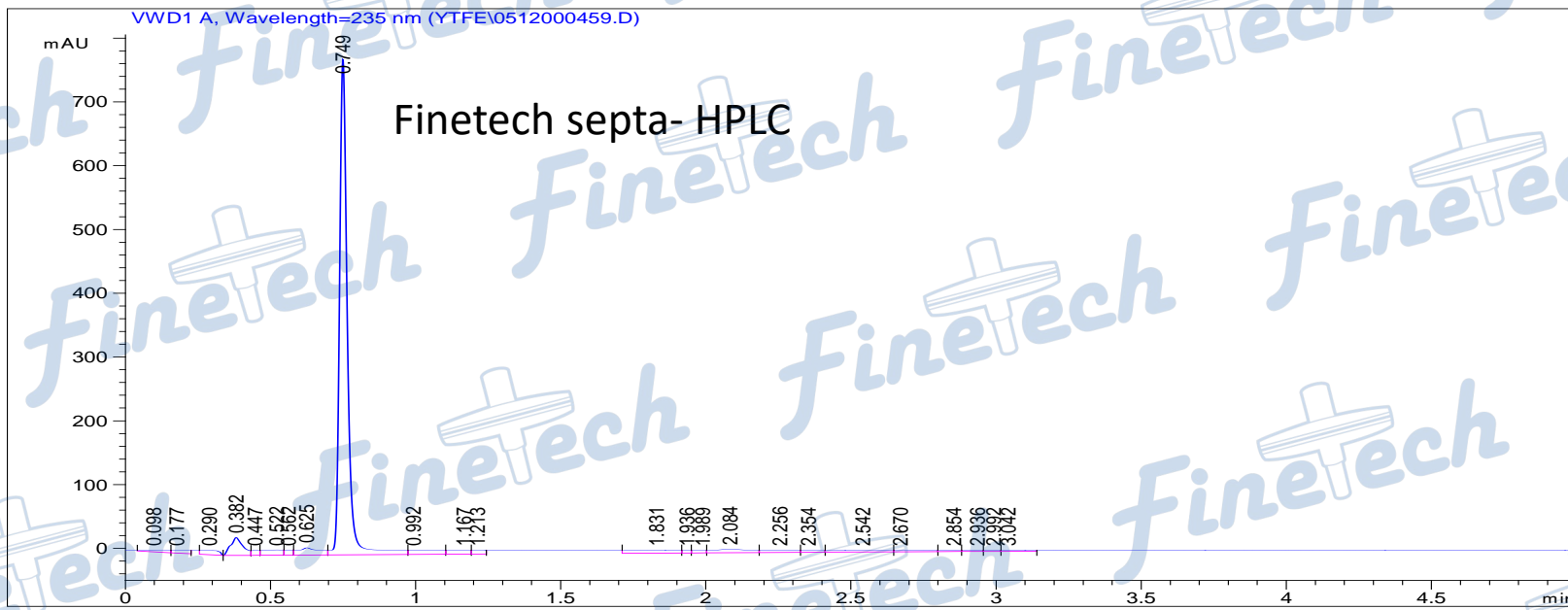
Experimental steps:

1. The different brands of septa were mounted on the vial caps.
2. Use salicylic acid as standard and add 1 mL of ACN:H₂O=1:1 as solvent to the vials.
3. Analyze the solvents by HPLC-UV (n = 18).

EXPERIMENTAL

Instrument	Brand A 1200 LC
Wavelength	235 nm
Mobile phase	ACN:H ₂ O=40:60
Column	Kinetex 5 μ C18 100A
Flow	1.5 mL/min
Injection size	20.0 μ L
Solvent	ACN:H ₂ O=1:1





Experimental results

1. The HPLC results show less interference and more signal area of the standard in Finetech's septa than Brand A. Therefore, Finetech's septa can be seen as being less likely to dissolve interfering compounds into the sample.

	RetTime (min)	Width (min)	Area (mAU)	Height (mAU)	Area %
Finetech	0.749	0.0296	1481.08228	773.61224	69.021
Brand A	0.737	0.0386	814.18939	317.87524	49.748

2. Recovery rate of standard was 96-104%.

Chemical Testing

Septa Degree of Gas Sealing Test by GC-FID

Experimental goal:

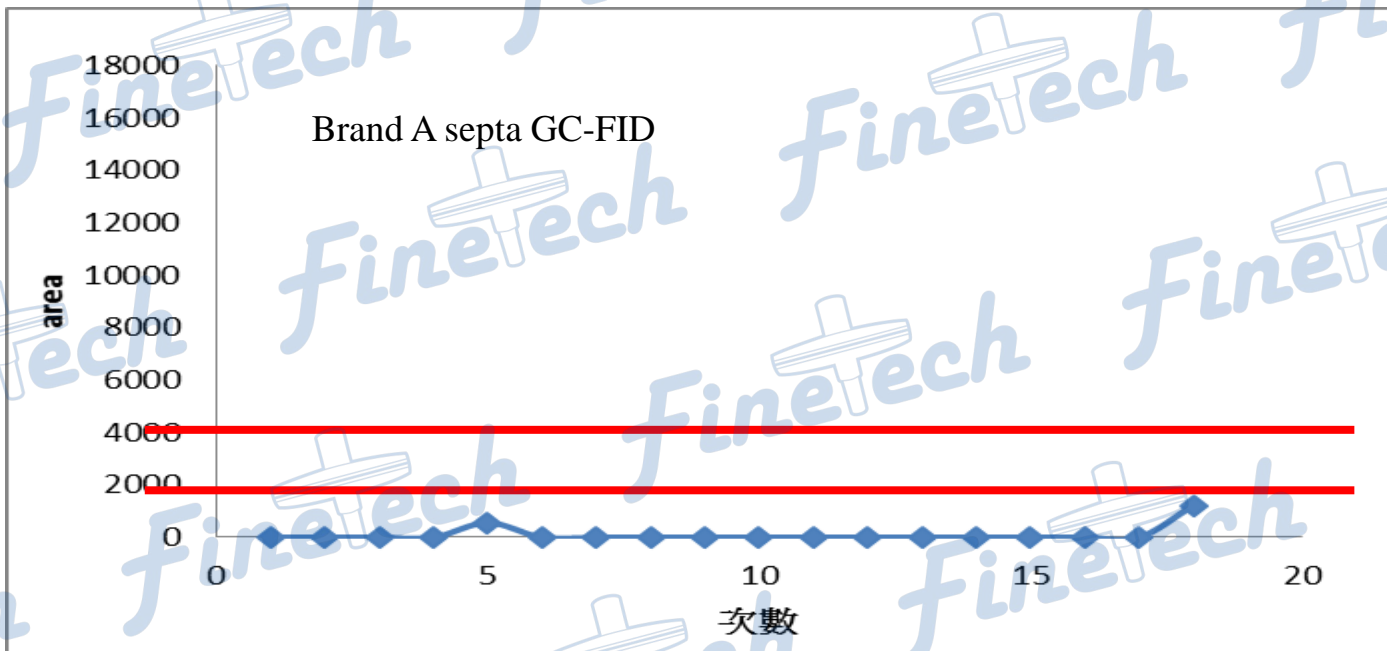
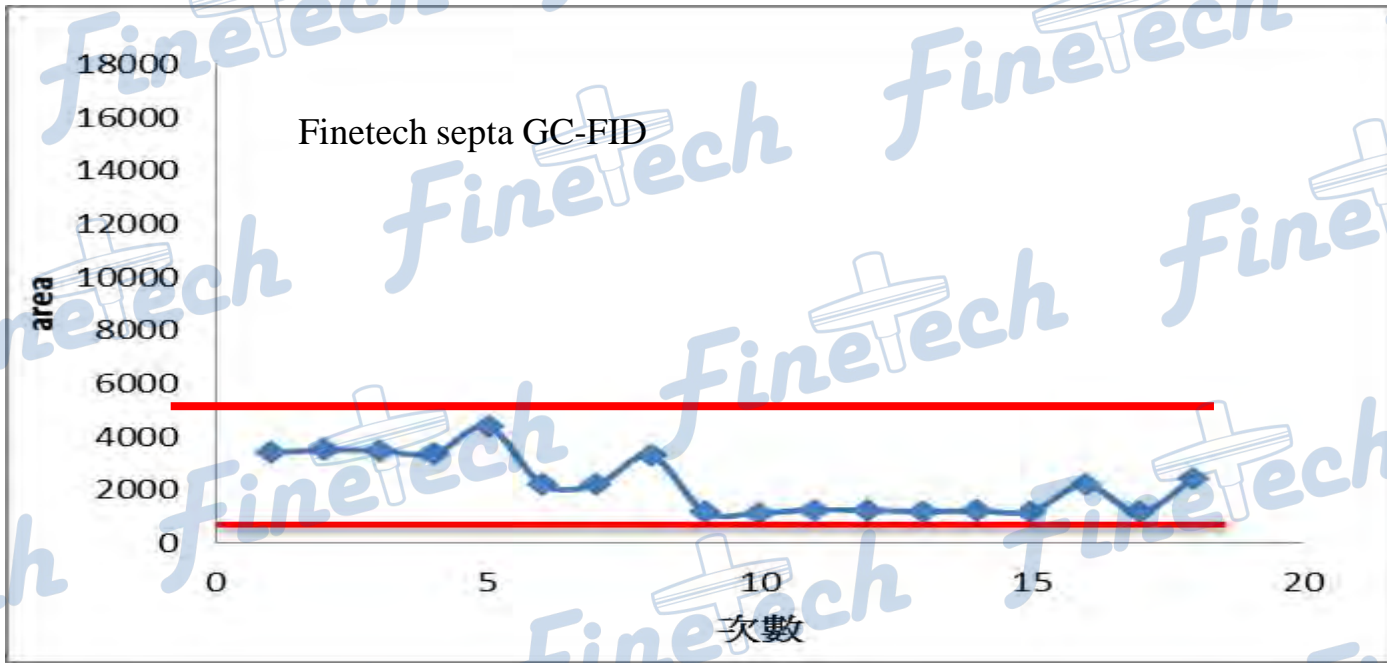
To test the septa on the degree of gas sealing after repeated use.

Experimental steps :

1. The different brands of septa were mounted on the vial caps.
2. 1.5 mL of MeOH / DCM (1: 1) solvent were added to the vials.
3. After 24 hours, analysis of the solvents by GC-FID (n = 18) was used to determine the degree of gas sealing.

Experimental Condition

Column	BR-5ms FS 15 m x 0.25 mm ID x 0.25 um
Detection	FID
H ₂ flow	30 ml/min
Air flow	400 ml/min
Makeup flow	29 ml/min (He)
Injection size	1.0 uL
Injection temperature	250°C
Column oven	50°C hold for 3 minutes, 50-100°C@ 25°C/min, 100-300°C@ 10°C/min, , 300-350°C@ 25°C/min (hold for minutes)
Pressure	25 psi for 27 minutes, 50 psi from 27-31 minutes



Experimental results

The peak area values throughout the test (n = 18) showed normal variations and did not illustrate a significant loss, which would indicate septum failure (RSD 99.99 - 100%). Overall, testing showed that the degree of gas sealing of Finetech's septum continued to perform well to 18 injections. This data suggests that Finetech's septa may continue to perform well after many more injections.



Chemical Testing

Temperature Test of Septa by GC-FID

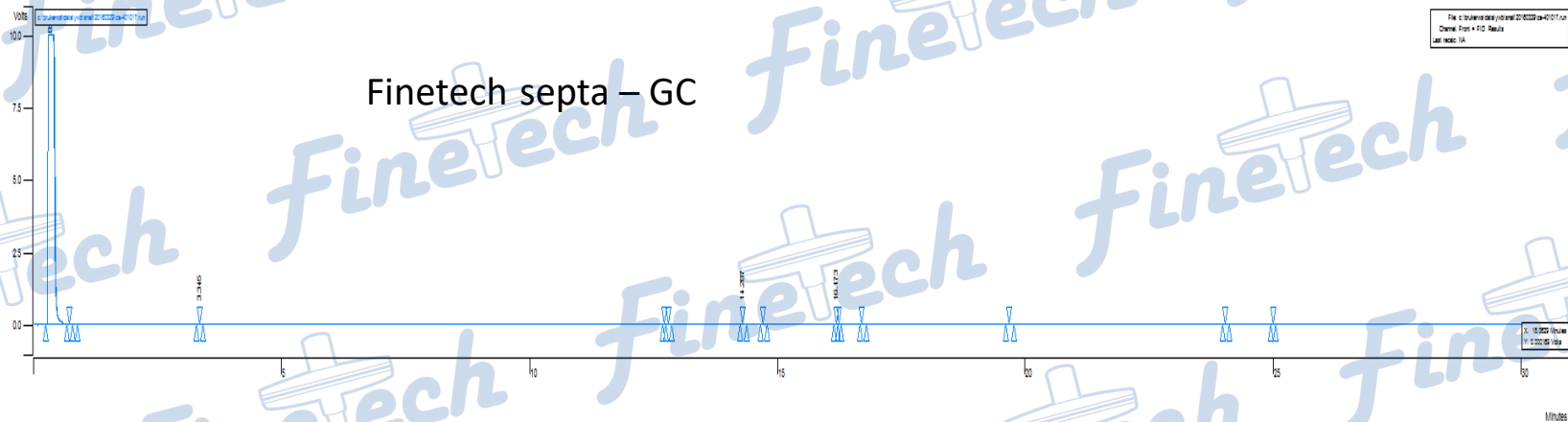
Experimental goal:

Use GC-FID to determinate the substances that are extracted from the septa material at different temperatures.

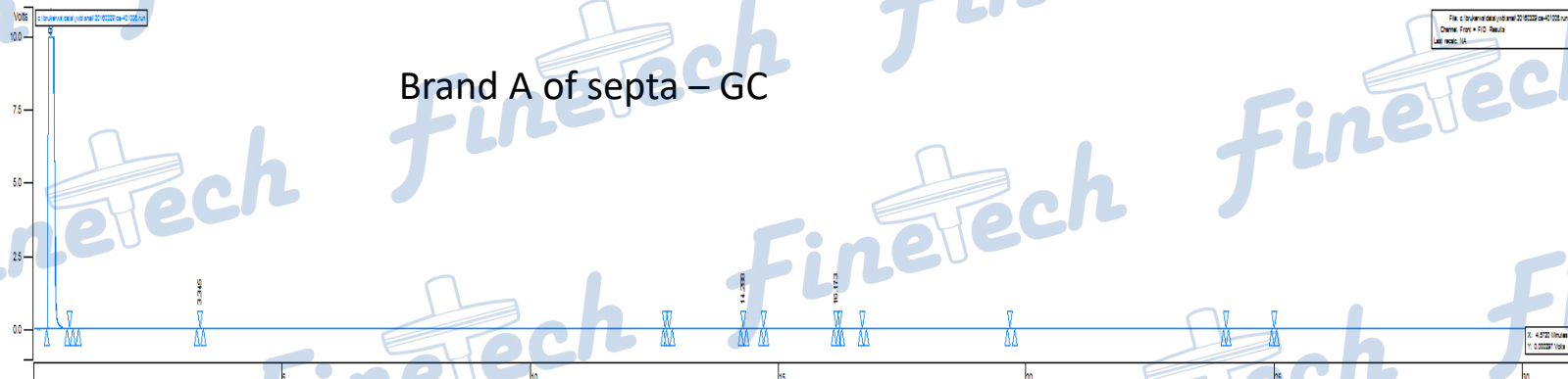
Experimental steps:

The septa were extracted in MeOH/DCM (50:50) at -4, 25, and 60°C for 72 hours, then analyzed by GC-FID.

-4°C

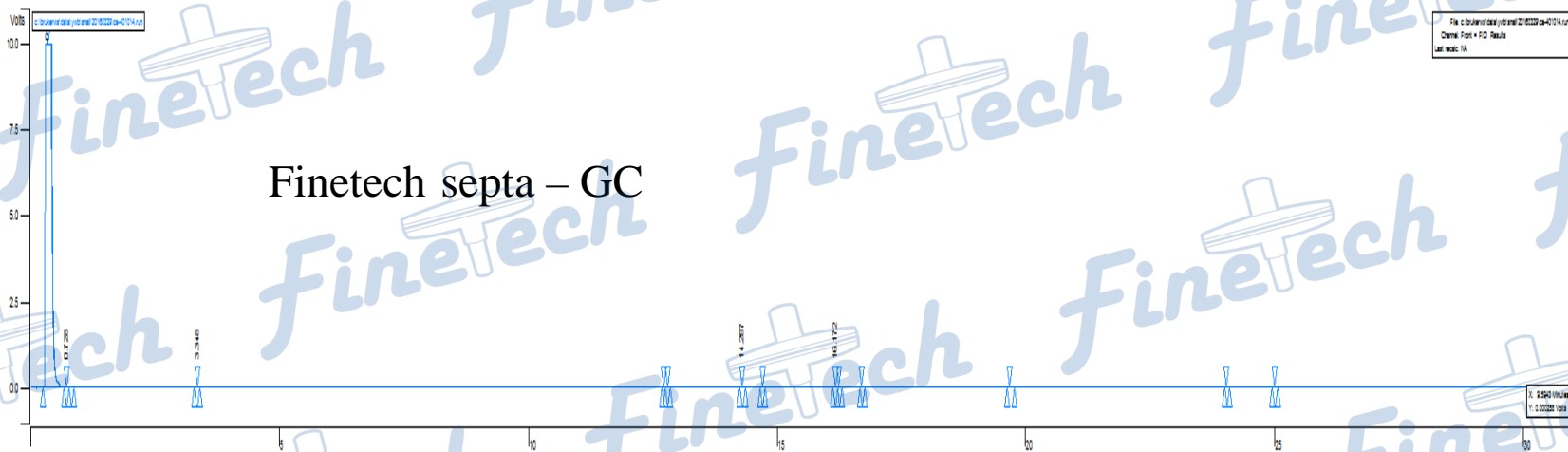


Finetech septa - GC

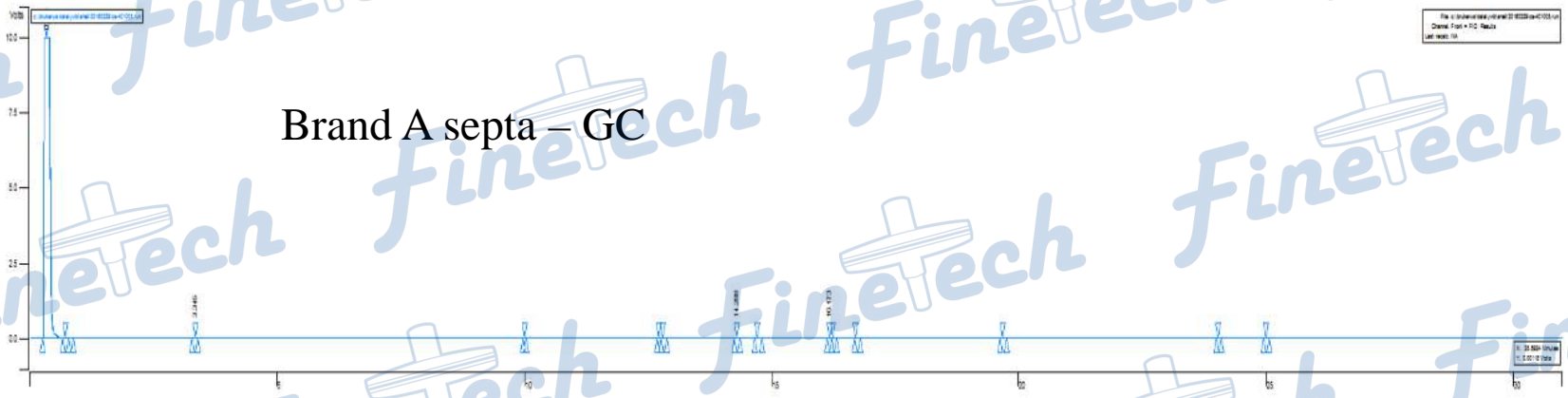


Brand A of septa - GC

25°C



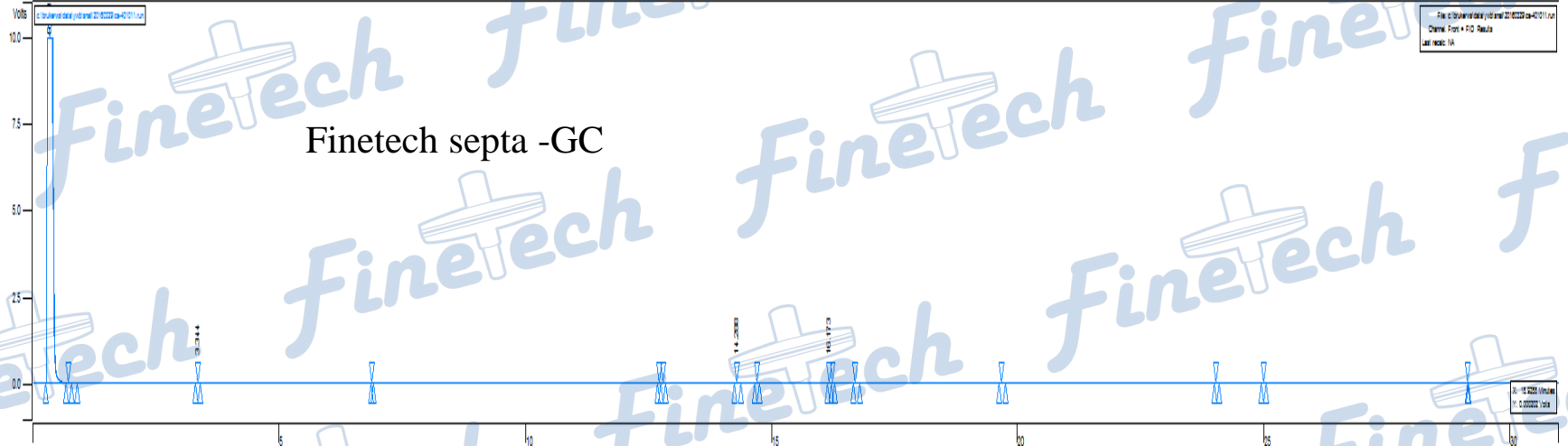
Finetech septa - GC



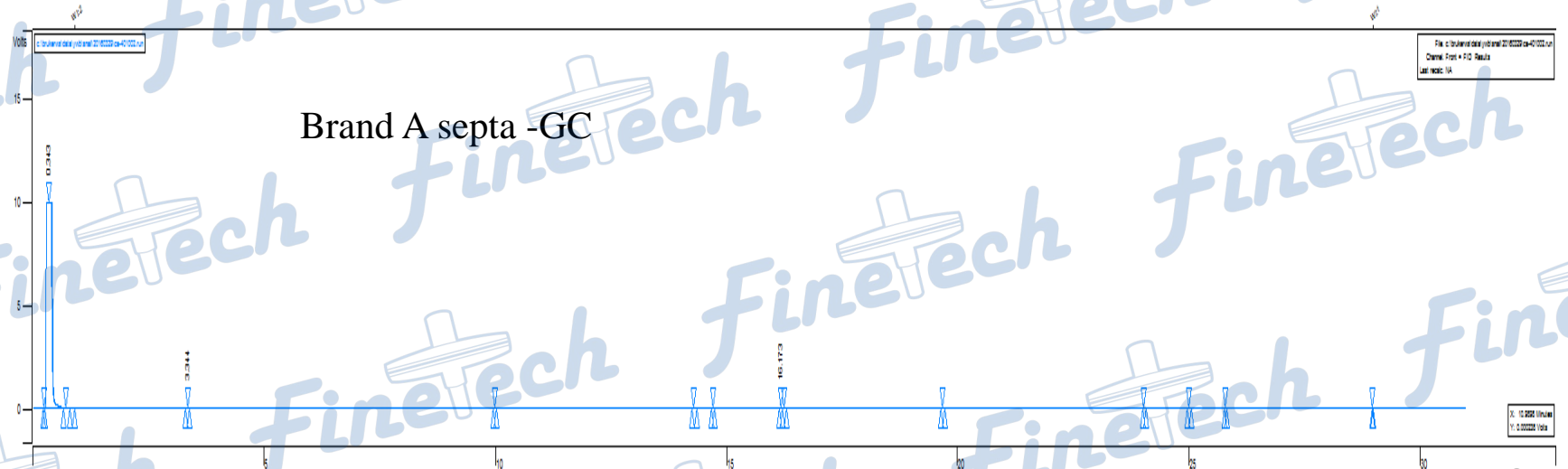
Brand A septa - GC

Minutes

60°C



Finetech septa -GC



Brand A septa -GC

Experimental result

1. From the results, there are no substances dissolved from the septa of Finetech or Brand A at different temperatures. The table shows that the difference of peak intensities for both septa are under acceptable range.

Temperature	60°C		25°C		-4°C	
Brand	Finetech	Brand A	Finetech	Brand A	Finetech	Brand A
1	4778	3371	5188	5268	3720	3898
2	4451	3458	5768	5596	3760	3634
3	4669	3597	5412	5334	3836	3757
RSD(%)	3.5940	3.2800	5.3609	3.2131	1.5621	3.5105
Average	4632	3475	5456	5399	3772	3763

2. Finetech's septa has similar total signal area to other brands, with a difference of about <5-10%.

Chemical Testing Summary

Test	Standard	Result
GC-FID: Septa Leaching and Headspace Test	Signal strength of Finetech's septa has a smaller total area (5-10%) than other brands.	Pass
GC-MS: Septa Composition Molecular Weight	m/z < 250, suitable for analysis at µg/mL concentration levels.	Pass
UV: Septa Leaching and pH test	UV wavelength 254 nm, signal strength of < 0.4 a. u..	Pass
LC-UV: Septa Bleed Test	Signal strength of Finetech's septa has a smaller total area (5-10%) than other brands. Recovery rate of standard was 96-104%.	Pass
Degree of Gas Sealing Test	Solvent signal strength and reproducibility (relative standard deviation < 3%).	Pass
Septa Temperature Test	Finetech's septa has similar total signal area to other brands, with a difference of about <5-10%.	Pass