

# High-Throughput BTEX Analysis in Nail Products by SPME and GC/TQ

Minimal steps, maximum precision

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## Abstract

This application note describes a rapid headspace-solid phase microextraction (HS-SPME) method for the determination of BTEX (benzene, toluene, ethylbenzene, and xylenes) in nail product matrices using an Agilent 8890 GC coupled to the Agilent 7000 Series triple quadrupole mass spectrometer (GC/TQ) operated in dynamic multiple reaction monitoring (dMRM) mode. A 100- $\mu$ m PDMS Agilent Smart SPME fiber was used for solvent-free extraction and automated fiber tracking, enabling streamlined setup and robust performance for volatile, nonpolar analytes. The method uses a 1-minute SPME extraction at 40 °C with an 8-minute GC run time and an ~11-minute total cycle time. Calibration achieved  $R^2$  up to 0.999 with average response factor RSDs of 4.44 to 10.37% over 10 to 5,000 total ng (*m*- and *p*-xylenes 20 to 10,000 total ng). Method accuracy and precision were verified with laboratory control samples (LCS), yielding mean recoveries of 100 to 108% with  $\leq$  8% RSD. This method was developed by the California Department of Toxic Substances Control (DTSC) to evaluate products for compliance with the Nail Products Containing Toluene regulations.<sup>1</sup>

## Introduction

BTEX compounds—benzene, toluene, ethylbenzene, and xylenes—are volatile aromatic hydrocarbons widely used as solvents in industrial and consumer products. Among these, toluene is the most prevalent in nail polish formulations, where it serves as a solvent to dissolve resins and pigments, ensuring smooth application, uniform color dispersion, and rapid drying. Historically, these properties made toluene a preferred ingredient in nail coatings, including traditional polishes, gel formulations, and nail art products. However, its inclusion presents challenges for regulatory compliance and product reformulation. In response to these challenges, the California Department of Toxic Substances Control (DTSC) has listed nail products containing toluene as Priority Products under the Safer Consumer Products Regulations.<sup>1</sup> Manufacturers selling such products in California must submit a Priority Product Notification and conduct an Alternative Analysis if toluene concentrations exceed the 100-ppm threshold.

To support compliance and ensure accurate monitoring of BTEX levels in nail products, robust analytical methods are essential. Solid phase microextraction (SPME) coupled with gas chromatography/triple quadrupole mass spectrometry (GC/TQ) has emerged as a preferred approach for volatile organic compound analysis due to its solvent-free sample preparation, high sensitivity, and compatibility with complex cosmetic matrices. The 8890 GC with 7000 Series GC/TQ offers enhanced detection capabilities through dynamic multiple reaction

monitoring (dMRM), enabling precise quantification of BTEX compounds at trace levels, below regulatory thresholds. This platform provides the analytical rigor required for method validation, including limits of detection, recovery, and precision, ensuring compliance with California's stringent performance criteria and supporting industry efforts toward improving consumer product quality.

## Experimental

### Sample preparation

Calibrants and samples were prepared by adding 50  $\mu$ L of the BTEX calibrant or sample and 50  $\mu$ L of 10 mg/L labeled toluene-d8 internal standard into a 20 mL headspace vial with a screw cap (part numbers 5188-6537 and 5188-2759). The samples were vortexed gently before placement on the CTC PAL3 Series 2 RTC autosampler. Calibration ranged from 10 to 5,000 total ng (20 to 10,000 total ng for *m*- and *p*-xylenes).

### SPME

In headspace-SPME, BTEX compounds partition between the sample matrix, the vapor phase, and a polymer coating on a fused silica fiber. After extraction, analytes are thermally desorbed into the GC inlet for separation and MS/MS detection. This method uses a 100- $\mu$ m polydimethylsiloxane (PDMS) Smart SPME fiber (Figure 1, part number 5610-5872), optimized for low molecular weight volatile compounds such as BTEX (MW 60 to 275). Smart SPME technology provides automated fiber recognition and intelligent tracking via an embedded chip, ensuring correct method parameters are applied and usage history is monitored for greater reliability. Headspace-SPME parameters are listed in Table 1.



**Figure 1.** 100- $\mu$ m PDMS Agilent Smart SPME fiber (part number 5610-5872), with automated fiber recognition.

**Table 1.** SPME headspace parameters.

SPME Parameter	Value
Script Name	SPME-STD-V7.3
SPME Fiber Phase	PDMS 100 $\mu$ m (p/n 5610-5872)
Heat Agitator	On
Incubation Time	0.5 min
Incubation Temperature	40 °C
GC Cycle Time	11 min
Fiber Conditioning Station Temperature	270 °C
Sample Vial Penetration Depth	50 mm
Sample Vial Penetration Speed	20 mm/s
Sample Extraction Time	1 min
Agitation During Extraction	On
Inlet Penetration Depth	40 mm
Inlet Penetration Speed	100 mm/s
Sample Desorption Time	2 min
Desorption Signal Mode	Before fiber expose
Pre Desorption Conditioning Time	60 s
Post Desorption Conditioning Time	300 s
Agitator Speed	250 rpm
Agitator On Time	5 s
Agitator Off Time	2 s

## GC/TQ analysis

Analyses were performed on an 8890 GC coupled to a 7000 Series GC/TQ equipped with an EI extractor ion source (Figure 2). The instrument operating parameters are listed in Table 2. The system was operated in splitless mode with an inlet temperature of 200 °C, a purge flow of 3 mL/min, and a purge flow to split vent of 50 mL/min at 2 minutes. Carrier gas was helium at a constant flow of 0.693 mL/min with a total flow of 53.7 mL/min. The chromatographic separation was accomplished on an Agilent J&W DB-624 Ultra Inert column (20 m × 0.18 mm, 1 µm, part number 121-1324UI). The oven program started at 40 °C (hold 2 minutes), ramped at 35 °C/min to 160 °C, and held for 2.58 minutes, giving a total run time of 8.0 minutes. The transfer line was maintained at 230 °C, with the ion source at 230 °C and quadrupoles at 150 °C.



**Figure 2.** The CTC PAL3 Series 2 RTC autosampler combined with an Agilent 8890 GC and Agilent 7000 Series GC/TQ.

**Table 2.** Agilent 8890 GC and Agilent 7000 Series GC/TQ conditions for BTEX analysis.

GC/TQ Parameter	Value
Inlet	Split/splitless
Mode	Splitless
Heater	200 °C
Pressure	14.517 psi
Total Flow	53.7 mL/min
Septum Purge Flow	3 mL/min
Septum Purge Flow Mode	Standard
Purge Flow to Split Vent	50 mL/min at 2.5 min
Inlet Liner	Agilent straight Ultra Inert 0.75 mm id liner (part number: 5190-4048)
Column	Agilent J&W DB-624 Ultra Inert column, 20 m × 0.18 µm, 1 µm (p/n 121-1324UI)
Control Mode	Constant flow
Flow	0.6931 mL/min
Pressure	3.5146 psi
Purge Flow	5 mL/min
Oven Program	40 °C (hold for 2 min) 35 °C/min to 160 °C (hold for 2.58 min) Total run time: 8.00 min
Transfer Line Temperature	230 °C
Electron Energy	70 eV
Quench Gas He	2.25 mL/min
Collision Gas N <sub>2</sub>	1.5 mL/min
Ion Source Temperature	230 °C
Quadrupole Temperature	150 °C
Scan Type	dMRM
Gain Factor	8

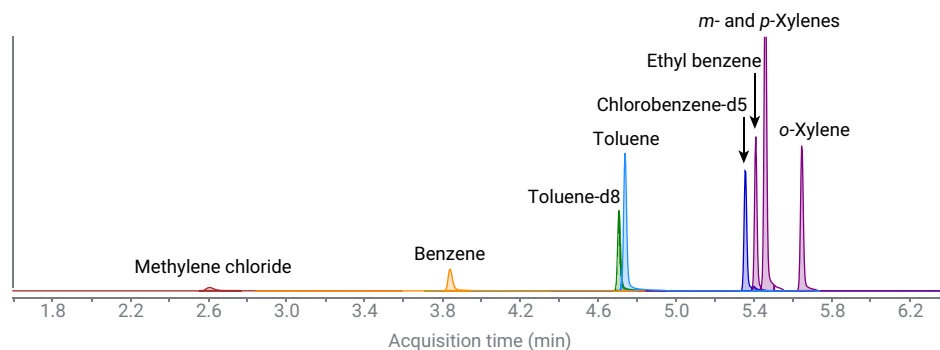
The mass spectrometer operated in dynamic multiple reaction monitoring (dMRM) mode for enhanced selectivity and sensitivity. Quench and collision gas flows were 2.25 mL/min He and 1.5 mL/min N<sub>2</sub>, respectively, with electron ionization at 70 eV. Compound transitions and dMRM parameters can be found in Table 3.

## Results and discussion

Headspace-SPME combined with GC/TQ enabled efficient extraction and selective quantification of BTEX in nail product matrices. Using a 100 µm PDMS Smart SPME fiber, the 1-minute extraction at 40 °C delivered consistent responses while avoiding solvent dilution and minimizing matrix effects typical of viscous formulations. Smart SPME simplified autosampler setup and ensured fiber traceability via an embedded chip for automated recognition on PAL3 platforms.

Chromatographic separation on the J&W DB-624 UI column with an 8.0-minute oven program enabled baseline or near-baseline resolution of BTEX compounds (Figure 3). The ~11-minute GC cycle time supports throughput of approximately five samples per hour per system without compromising peak shape or reproducibility. Acquisition in

dMRM on the 7000 Series provided high selectivity and low noise in complex cosmetic matrices. Transitions were optimized for abundant quantifier and qualifier ions; for example, toluene 92 → 91 and 92 → 65, consistent with California DTSC guidance for compliance testing.



**Figure 3.** Chromatogram of 500 ng calibration standard using dMRM extracted transitions with target, internal standard, and surrogate compounds labeled.

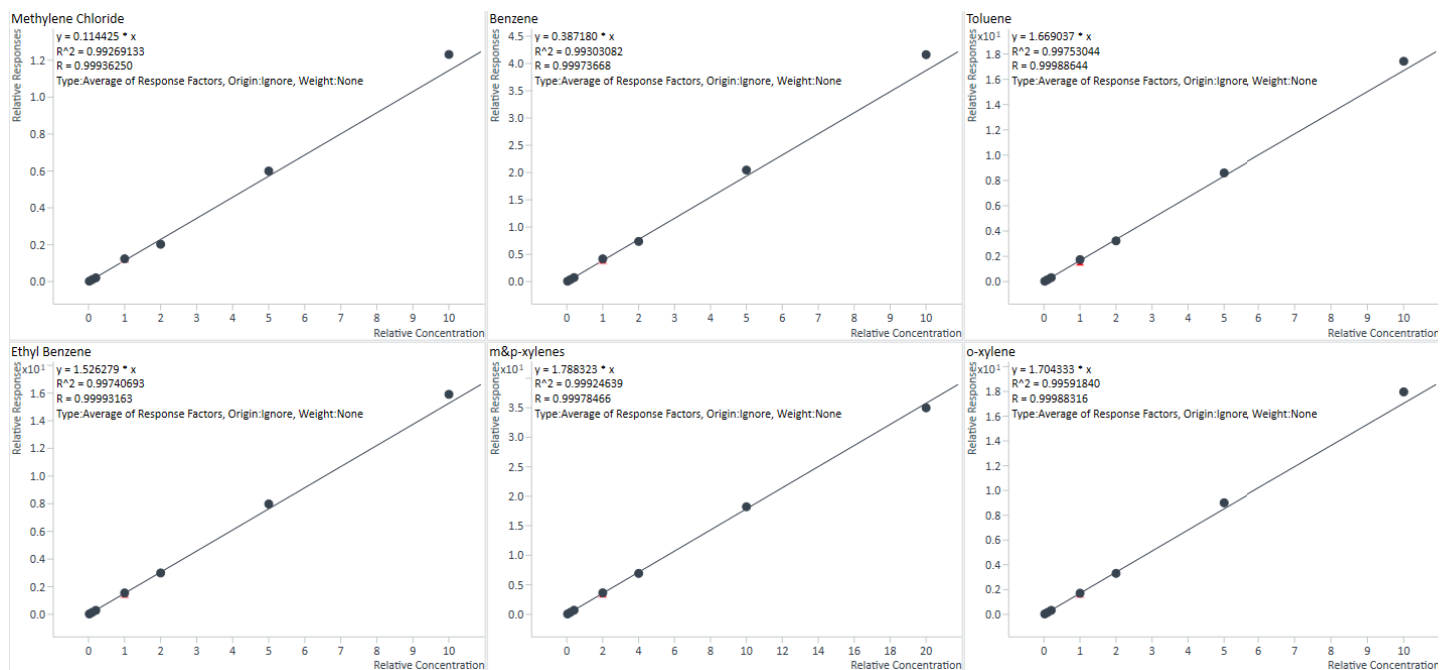
**Table 3.** Compound table with parameters used in dMRM mode.

Compound Name	ISTD	Precursor Ion	MS1 Resolution	Product Ion	MS2 Resolution	RT	Left RT	Right RT	CE	Dwell (ms)
Methylene Chloride	No	84	Wide	49	Wide	2.6	0.3	0.2	15	99.3
Methylene Chloride	No	84	Wide	48	Wide	2.6	0.3	0.2	15	99.3
Benzene	No	78.1	Wide	77	Wide	3.83	0.3	0.2	15	99.3
Benzene	No	78.1	Wide	52	Wide	3.83	0.3	0.2	15	99.3
Toluene-d8	Yes	98.1	Wide	70	Wide	4.7	0.3	0.2	15	74.2
Toluene-d8	Yes	98.1	Wide	42	Wide	4.7	0.3	0.2	15	74.2
Toluene	No	92.1	Wide	91	Wide	4.73	0.3	0.2	15	74.2
Toluene	No	92.1	Wide	65	Wide	4.73	0.3	0.2	15	74.2
Chlorobenzene-d5	No	117	Wide	82	Wide	5.34	0.3	0.2	15	51.3
Chlorobenzene-d5	No	117	Wide	54	Wide	5.34	0.3	0.2	15	51.3
Ethyl Benzene	No	106.1	Wide	91	Wide	5.4	0.3	0.2	15	34.6
Ethyl Benzene	No	106.1	Wide	65	Wide	5.4	0.3	0.2	15	34.6
m- and p-Xylenes	No	106.1	Wide	105.1	Wide	5.45	0.3	0.2	15	34.7
m- and p-Xylenes	No	106.1	Wide	91	Wide	5.45	0.3	0.2	15	34.7
o-Xylene	No	106.1	Wide	105.1	Wide	5.63	0.3	0.2	15	51.4
o-Xylene	No	106.1	Wide	91	Wide	5.63	0.3	0.2	15	51.4

Calibration curves (Table 4 and Figure 4) demonstrated excellent linearity across the working range, with average response factor (RF) RSDs  $\leq 8.2\%$  for all compounds, meeting method acceptance criteria. Validated ranges were 10 to 5,000 total ng (20 to 10,000 total ng for *m*- and *p*-xylenes), with 89 to 108% accuracy across all calibration points, ensuring reliable quantification at regulatory thresholds. Excellent accuracy and precision were verified with replicate LCS spikes (Table 5), with mean recoveries from 100 to 108% and RSDs  $\leq 8\%$ .

**Table 4.** Calibration information including retention time, average response factor relative standard deviation (avg. RF RSD), relative standard error ( $R^2$  value), and calibration range in total ng.

Name	Retention Time (min)	Avg. RF RSD (%)	$R^2$	Calibration Limit Low (ng)	Calibration Limit High (ng)
Methylene Chloride	2.60	8.2	0.993	10	5,000
Benzene	3.83	6.6	0.993	10	5,000
Toluene	4.73	4.5	0.998	10	5,000
Chlorobenzene-d5	5.34	2.6	Surrogate at 500		
Ethyl Benzene	5.40	3.7	0.997	10	5,000
<i>m</i> - and <i>p</i> -Xylenes	5.45	2.3	0.999	20	10,000
<i>o</i> -Xylene	5.63	4.3	0.996	10	5,000

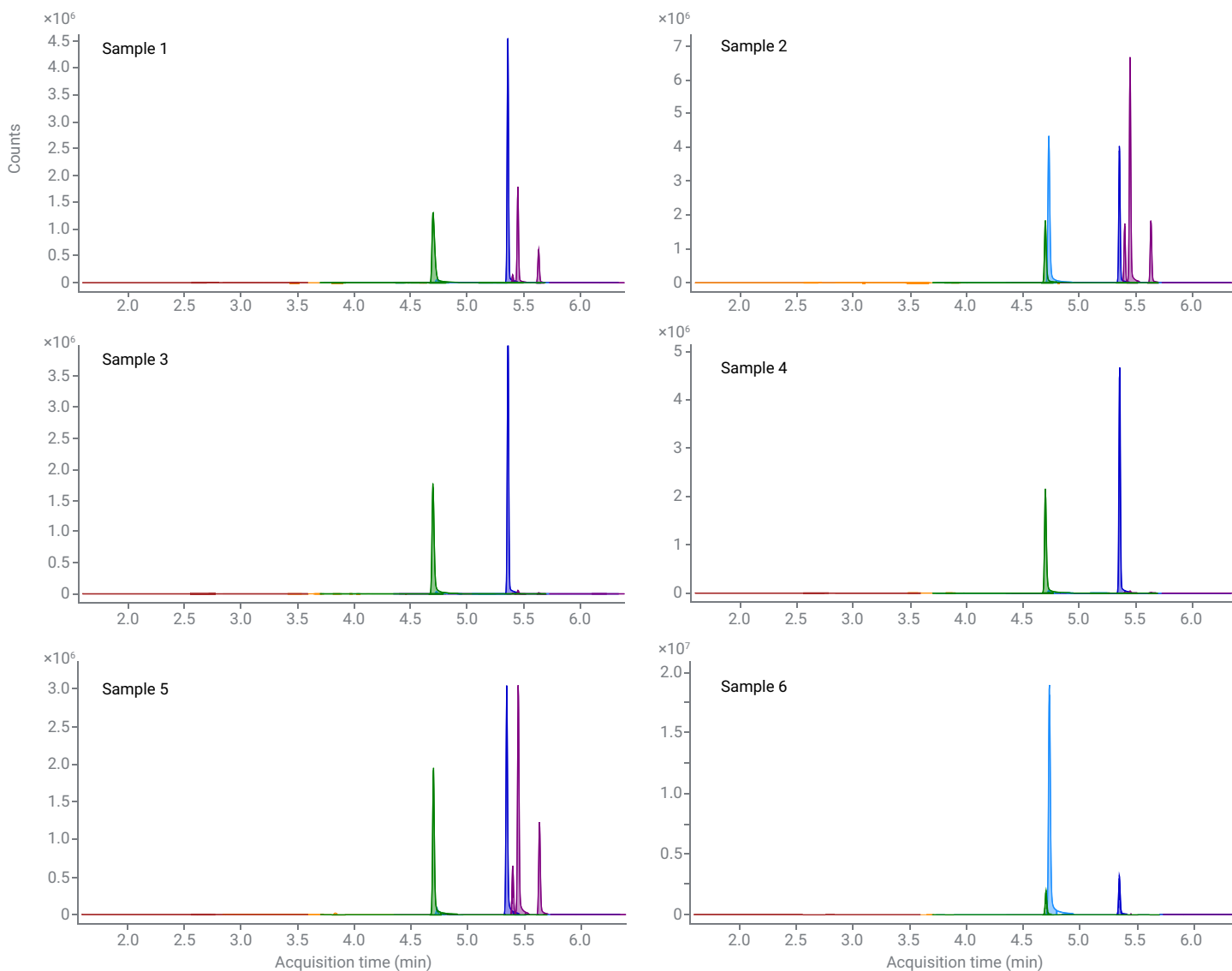


**Figure 4.** Calibrations for BTEX, with a calibration range from 10 to 5,000 total ng (20 to 10,000 total ng for *m*- and *p*-xylenes), all using average response factors (avg. RF).

Figure 5 and Table 6 show that BTEX concentrations varied widely across the nail product samples, with toluene levels ranging from low ng amounts in Sample 1 to more than 3,000 ng in Sample 6. Across all quantified analytes, precision was strong, with relative percent difference (%RPD) values falling between approximately 0.1% and 17%, demonstrating reliable reproducibility even at low concentration levels.

**Table 5.** Accuracy and precision of laboratory control spikes (LCS, N = 4) in total weight (ng), with % recovery and % RSD.

Compound	Spike (ng)	LCS 1 (ng)	LCS 2 (ng)	LCS 3 (ng)	LCS 4 (ng)	Mean (ng)	Recovery (%)	RSD (%)
Methylene Chloride	500	553	526	523	530	533	107	2.6
Benzene	500	519	528	502	505	513	103	2.4
Toluene	500	508	512	504	483	502	100	2.6
Ethyl Benzene	500	551	519	514	453	510	102	8.0
<i>m</i> - and <i>p</i> -Xylenes	1,000	1,090	1,030	1,020	898	1,010	101	8.0
<i>o</i> -Xylenes	500	556	523	519	462	515	103	7.5
Surrogate Recovery								
Chlorobenzene-d5	500	566	527	535	529	539	108	3.4



**Figure 5.** Chromatograms of nail product samples 1 to 6, with duplicates overlaid, using extracted quantification *m/z* ions.

**Table 6.** Sample results in total ng  $\pm$  relative percent difference (%RPD).

	Total ng $\pm$ %RPD						
	Methylene Chloride	Benzene	Toluene	Chlorobenzene-d5	Ethyl Benzene	<i>m</i> - and <i>p</i> -Xylenes	<i>o</i> -Xylene
Sample 1	–	–	13.9 $\pm$ 8.3	614 $\pm$ 0.65	17.6 $\pm$ 7.2	187 $\pm$ 8.5	75.5 $\pm$ 7.3
Sample 2	–	–	715 $\pm$ 3.0	578 $\pm$ 0.48	218 $\pm$ 4.0	773 $\pm$ 4.1	239 $\pm$ 4.4
Sample 3	–	–	–	554 $\pm$ 0.13	–	–	–
Sample 4	–	–	–	558 $\pm$ 0.37	–	–	–
Sample 5	–	19.8 $\pm$ 5.4	15.6 $\pm$ 5.5	494 $\pm$ 2.7	90.6 $\pm$ 0.21	386 $\pm$ 0.96	167 $\pm$ 1.2
Sample 6	–	–	3,083 $\pm$ 17	519 $\pm$ 0.80	–	–	–

## Conclusion

A rapid, high-throughput headspace-SPME GC/TQ method was developed for quantifying BTEX in nail product matrices, using a 100- $\mu$ m PDMS Agilent Smart SPME fiber on an Agilent 8890 GC with Agilent 7000 Series GC/TQ. The method provides solvent-free extraction, 8-minute chromatographic runs, and an  $\sim$ 11-minute total cycle time, enabling high-throughput analysis with robust selectivity in dMRM mode. Quality control results demonstrated mean recoveries of 100 to 107% with RSDs  $\leq$  8%, meeting method acceptance criteria for compliance-focused testing

for toluene. Integration of Smart SPME automation and the intelligent features of the 8890 GC and 7000 Series GC/TQ allows a practical, defensible solution for routine laboratories monitoring BTEX in nail products. Calibration performance showed high linearity and low variance across the working ranges, with all compounds meeting initial calibration criteria via  $R^2 \geq 0.995$  or average RF RSD  $\leq 20\%$ . Combined with the short cycle time and automated fiber tracking, this workflow is ideally suited for routine compliance testing of BTEX in nail products using the 8890 GC and 7000 Series GC/TQ.

## Reference

1. California Department of Toxic Substances Control. Safer Consumer Products Regulations – Listing Nail Products Containing Toluene as a Priority Product; Final Regulatory Text. Office of Administrative Law Reference Number: 2021-0921-06; Department of Toxic Substances Control Reference Number: R-2019-04; Sacramento, CA, 2023. Available at: [https://dtsc.ca.gov/wp-content/uploads/sites/31/2023/08/Final-Regulation-Text-with-OAL-Comments\\_FINAL\\_Final-Accessible.pdf](https://dtsc.ca.gov/wp-content/uploads/sites/31/2023/08/Final-Regulation-Text-with-OAL-Comments_FINAL_Final-Accessible.pdf).

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