Application Note

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Routine Trace-Level Analysis of *m*-Xylene in High-Purity *p*-Xylene

Using the Agilent 8890B GC system

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Abstract

This application note demonstrates the analysis of trace-level *m*-xylene in high-purity *p*-xylene using the Agilent 8890B GC system. Two different columns were used to demonstrate a high-resolution separation (19.3 minutes) that prioritizes system stability and robustness, along with a high-speed approach (13.8 minutes) using a narrower column, trading some robustness for a 40% faster run time. Linearity was established in the range of 10–50 ppm *m*-xylene, with R^2 values of 0.99950 and 0.99975 for the high-resolution and high-speed methods, respectively. Method precision was evaluated across 200 consecutive injections of 50 ppm *m*-xylene, showing peak area relative standard deviations (RSDs) of 1.68% and 2.88%, and average signal-to-noise ratios (S/Ns) of 98.5 and 27.7 for the high-resolution and high-speed methods, respectively. The configuration and use of the on-board early maintenance feedback (EMF) counters and peak evaluation capabilities of the 8890B GC were also demonstrated.

Introduction

The Agilent 8890B GC system builds on the exceptional performance of the Agilent 8890A GC, with the addition of many features designed to enhance the user experience for both new and expert-level operators. This application note explores several use cases of these features applied to a challenging GC purity method—quantifying trace-level *m*-xylene (MX) in *p*-xylene (PM). This separation requires careful balancing of optimized chromatographic parameters—speed, resolution, and sensitivity. Leveraging Agilent GC Assist features, including early maintenance feedback (EMF) counters and automated peak evaluation capabilities integrated into the 8890B GC, helps maximize method robustness and instrument uptime.

Though conceptually simple, separating and measuring small quantities of *m*-xylene in *p*-xylene is a challenge that requires precise instrument control and the highest-quality GC parts and consumables. As both compounds are positional isomers and have nearly identical boiling points, separation has historically been achieved using a long and highly polar column, such as a polyethylene glycol "wax" phase. ASTM D7504 recommends a wax column with dimensions 60 m × 0.320 mm and 0.25 µm phase thickness. A 0.50 µm phase thickness can be used for increased resolution.[1] The separation is validated with a *p*-xylene standard containing approximately 1,000 ppm *m*-xylene. Measuring *m*-xylene at significantly lower concentrations requires an increase in resolution between MX and PX, which can be accomplished by increasing the phase thickness of the column at the cost of increased peak broadening. This broadening negatively impacts the limit of detection (LOD) and the limit of quantification (LOQ) for MX and must be offset by reducing the inner diameter of the column to resharpen the peak. This application note demonstrates the use of 0.200 mm and 0.100 mm id wax columns to yield optimized separations, presenting the associated benefits and limitations of both separations.

Experimental

An 8890B GC was configured with an Agilent 7650A automatic liquid sampler (ALS), split/splitless (SSL) inlet, and flame ionization detector (FID). Two columns were used to explore two different separation speeds—a faster separation with less sample capacity using an Agilent J&W DB-WAX FF column, 20 m × 100 µm, 0.2 µm (p/n 127-7023FF) and a higher-resolution separation with more sample capacity using an Agilent J&W HP-INNOWax column, 50 m × 200 µm, 0.4 µm (p/n 19091N-205I). Both methods used hydrogen carrier gas, and Agilent Gas Clean filters were used to remove moisture, hydrocarbons, and oxygen from the carrier gas and FID hydrogen. The FID air was filtered for moisture and hydrocarbons. The GC configuration and method details can be found in Tables 1 and 2, respectively.

High-purity *p*-xylene was produced from reagent-grade *p*-xylene (p/n 134449, Sigma-Aldrich) using the recrystallization procedure outlined in ASTM D7504. Low-concentration standards of *m*-xylene were produced gravimetrically using the purified *p*-xylene and 99% *m*-xylene (p/n 296325, Sigma-Aldrich). Data acquisition and analysis was performed using Agilent OpenLab CDS v2.8.

The inlet consumables were chosen as an optimized balance between inertness, longevity, and cost. The Agilent Advanced Green septum (p/n 5183-4759) has very low bleed and a long lifetime under these method conditions at inlet temperatures below 300 °C. The tapered needle on the 5 µL Agilent Blue Line autosampler syringe (p/n G4513-80206) further increases septum life by reducing coring. The Agilent inert split inlet liner (p/n 5183-4647) contains glass wool that traps pieces of septa and wipes the tip of the syringe needle, thereby increasing the precision of the injection volume.

Table 1. Agilent 8890B GC configuration.

Configuration		
	High-Resolution Method	**High-Speed Method**
Sampler	7650A automatic liquid sampler (ALS)	7650A automatic liquid sampler (ALS)
Inlet	High-pressure split/splitless	High-pressure split/splitless
Column	HP-INNOWax 50 m × 0.200 mm, 0.4 µm (p/n 19091N-205I)	DB-WAX FF 20 m × 0.100 mm, 0.2 µm (p/n 127-7023FF)
Detector	FID	FID
Carrier Gas	Hydrogen	Hydrogen
Consumables		
Inlet Septa	Nonstick Advanced Green (p/n 5183-4759)	
Inlet Liner	Inert, low pressure drop, split liner with glass wool (p/n 5183-4647)	
ALS Syringe	Blue Line, 5 µL, fixed needle, 23–26 s/42/cone (p/n G4513-80206)	
Carrier Gas Filter	Agilent Gas Clean purifier kit for carrier gas, 1/8 in (p/n CP17976)	
FID Hydrogen Gas Filter	Agilent Gas Clean purifier kit for carrier gas, 1/8 in (p/n CP17976)	
FID Air Gas Filter	Agilent Gas Clean moisture purifier (p/n CP17971) and Agilent Gas Clean hydrocarbon purifier (p/n CP17972)	

Table 2. Method conditions.

	High-Resolution Method	High-Speed Method
Run Time	19.3 minutes	13.83 minutes
ALS and Inlets		
Carrier Gas	Hydrogen, 1.5 mL/min constant flow	Hydrogen, 0.5 mL/min constant flow
Septum Purge	6 mL/min	6 mL/min
Injection Volume	0.3 µL	0.3 µL
Mode	Split, 250:1	Split, 750:1
Temperature	250 °C	240 °C
Oven Program		
Initial Temperature	40 °C	40 °C
Initial Hold	0 minutes	0 minutes
Ramp No. 1 Rate	3 °C/min	3 °C/min
Ramp No. 1 Setpoint	85 °C	65 °C
Ramp No. 1 Hold	–	–
Ramp No. 2 Rate	50 °C/min	50 °C/min
Ramp No. 2 Setpoint	250 °C	240 °C
Ramp No. 2 Hold	1 minute	2 minutes
Detector		
Data Rate	10 Hz	10 Hz
Temperature	260 °C	240 °C
Air	300 mL/min	300 mL/min
Hydrogen	30 mL/min	30 mL/min
Makeup (N_2)	25 mL/min	25 mL/min

Results and discussion

Establishing and ensuring the method's long-term stability requires protecting the column stationary phase by limiting the amount of oxygen in the system. While carrier gas contamination is typically the largest source of unwanted oxygen, the inlet septum can also contribute to oxygen ingress as successive syringe injections create a hole, necessitating regular changing. The Agilent portfolio of consumables and supplies has several solutions specifically designed to overcome these challenges. The Gas Clean purification system features a carrier gas filter that removes oxygen, moisture, and hydrocarbons and includes a color-changing visual indicator of filter life, which can be monitored remotely by the 8890B GC using an optional optical sensor. Advanced Green septa are engineered for low bleed and extended durability, further enhanced by using a Blue Line autosampler syringe with a tapered needle to reduce physical coring of the septum. Regular maintenance of these parts maximizes instrument uptime. The 8890B GC has several features designed to warn when maintenance is approaching, assist when maintenance is required, and track when maintenance is completed.

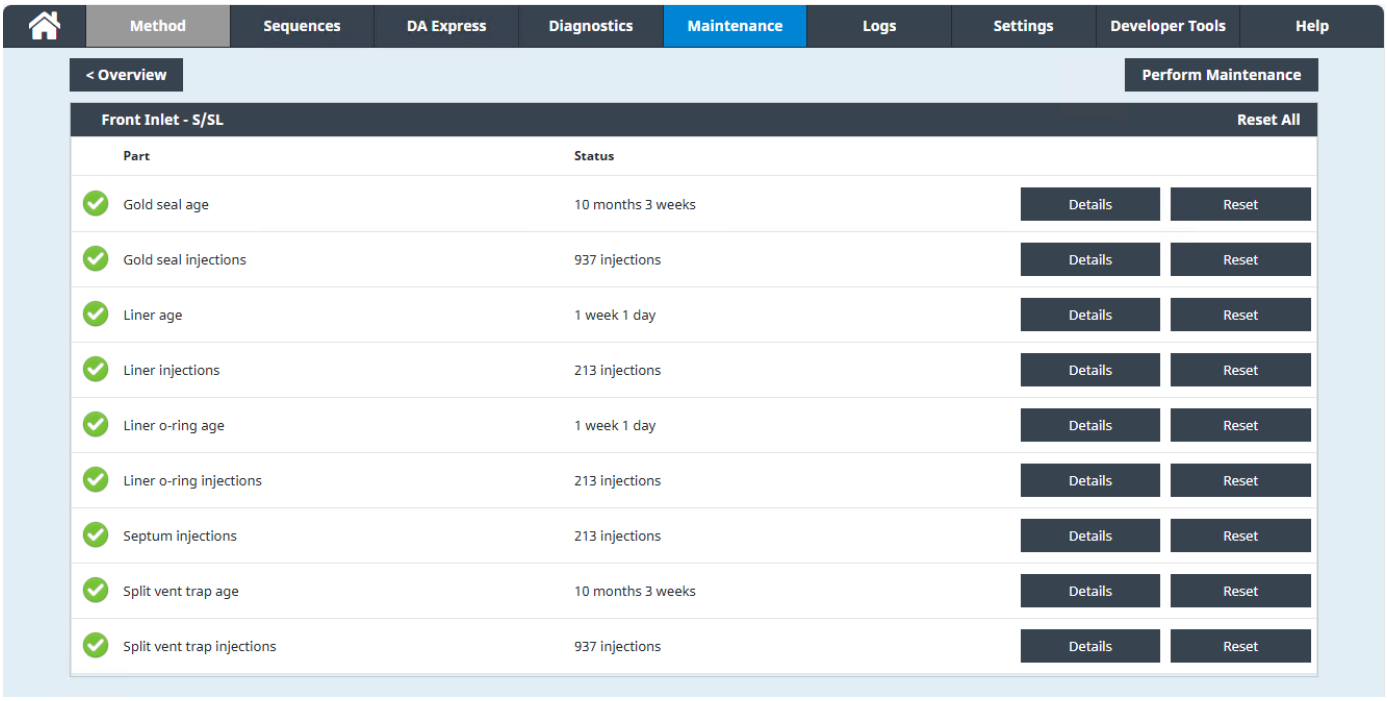

Figure 1. Early maintenance feedback (EMF) counters tracking elapsed time and number of injections for the front inlet consumables.

The 8890B GC features intelligent tracking of the ages of consumables using injection- and time-based counters. These EMF counters feature two optional warning thresholds—"Service Warning" and "Service Due"—that are user-configurable and function like the oil light on the dashboard of a vehicle, telling users of approaching maintenance. The EMF counters can be accessed directly through the GC touchscreen or through the GC Assist interface. Figure 1 shows an overview of the EMF counters for the front inlet, and Figure 2 presents the detailed history of septum maintenance, including a running tally of injections on the current septum. When performing maintenance on the 8890B GC using the guided maintenance process on the touch screen or GC Assist interface, the EMF counters are automatically reset at the end of the procedure and can also be reset manually. For this application, the EMF counters and appropriate warning thresholds for the septum, liner, syringe, column, inlet gold seal, and carrier gas filter will be explored.

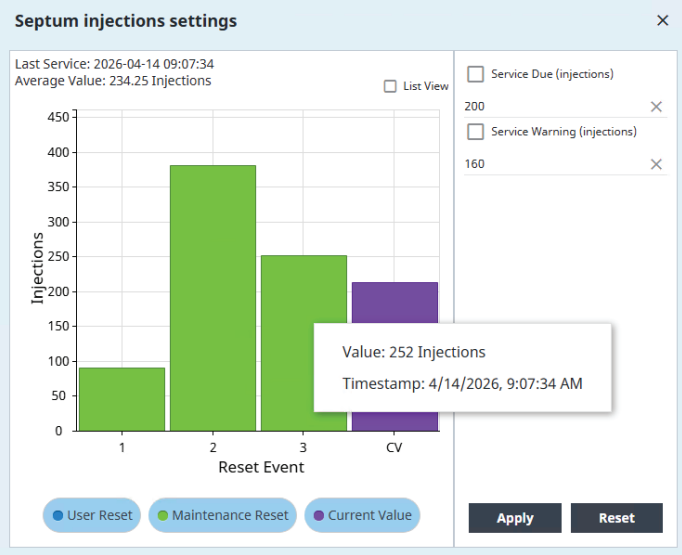

Figure 2. Detailed EMF counter history showing the number of injections for the current and previous front inlet septa.

Column performance

An important complication when separating MX and PX is that, because the PX peak is overloaded, both peaks experience retention time shifts as the injected sample amount varies. The 8890B GC leverages the unrivaled pneumatic precision and advanced control of Agilent seventh-generation electronic pneumatic control (EPC) modules. These EPCs ensure consistent sample splitting in the inlet during injection by correcting for localized drift in temperature and atmospheric pressure conditions around the GC. The result is exceptional retention time precision for MX, as shown in Figures 3 and 4, which were generated using Peak Explorer in OpenLab CDS. Peak Explorer facilitates the rapid identification of differences between chromatograms in large data sets. The tool simplifies chromatographic peaks in each run into circular markers on the horizontal time axis and vertically stacks the runs, with the first injection at the top and the final injection at the bottom. This visualization format makes it easy to quickly identify outlier runs and observe trends in chromatographic behavior.

Figures 3 and 4 show the long-term retention behavior of PX (left circles) and MX (right circles) for both columns across 200 injections of 50 ppm MX. The 100 µm id DB-WAX FF column separates PX and MX with retention times of approximately 7.63 minutes and 7.68 minutes, respectively; the total run time is 13.83 minutes, and the cycle time is approximately 17.5 minutes. The 200 µm id HP-INNOWax column yields higher retention than the 100 µm DB-WAX FF, with retention times of 12.90 minutes and 13.04 minutes for PX and MX, respectively. The HP-INNOWax column has a total run time of 19.3 minutes with a cycle time of approximately 23.25 minutes. A summary of key performance metrics is shown in Table 3.

Table 3. Key performance metrics from 200 consecutive injections of 50 ppmw MX standard for both methods.

	Retention Time (min)		Peak Area		Resolution (USP)		S/N	
	High Speed	High Resolution	High Speed	High Resolution	High Speed	High Resolution	High Speed	High Resolution
Average	7.6018	13.0410	0.2951	1.3736	0.4966	1.2758	27.65	98.47
Standard Deviation	0.0049	0.0010	0.0085	0.0231	0.0085	0.0147	2.36	9.86
%RSD	0.06	0.01	2.88	1.68	1.71	1.15	8.55	10.02

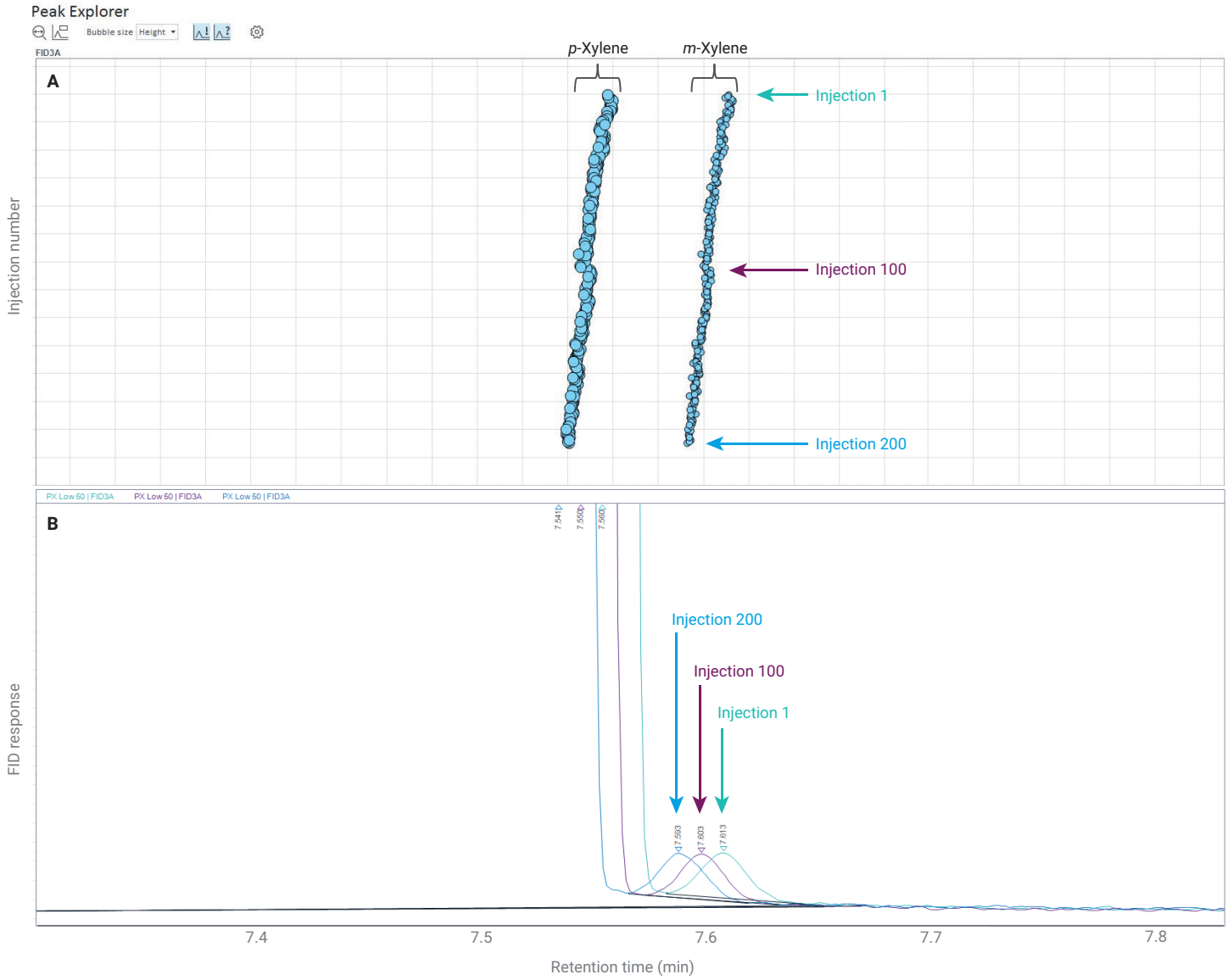

Figure 3. Retention precision for 50 ppm MX in PX across 200 injections using the Agilent J&W DB-WAX FF column, visualized using (A) Peak Explorer and (B) chromatographic overlay in Agilent OpenLab CDS.

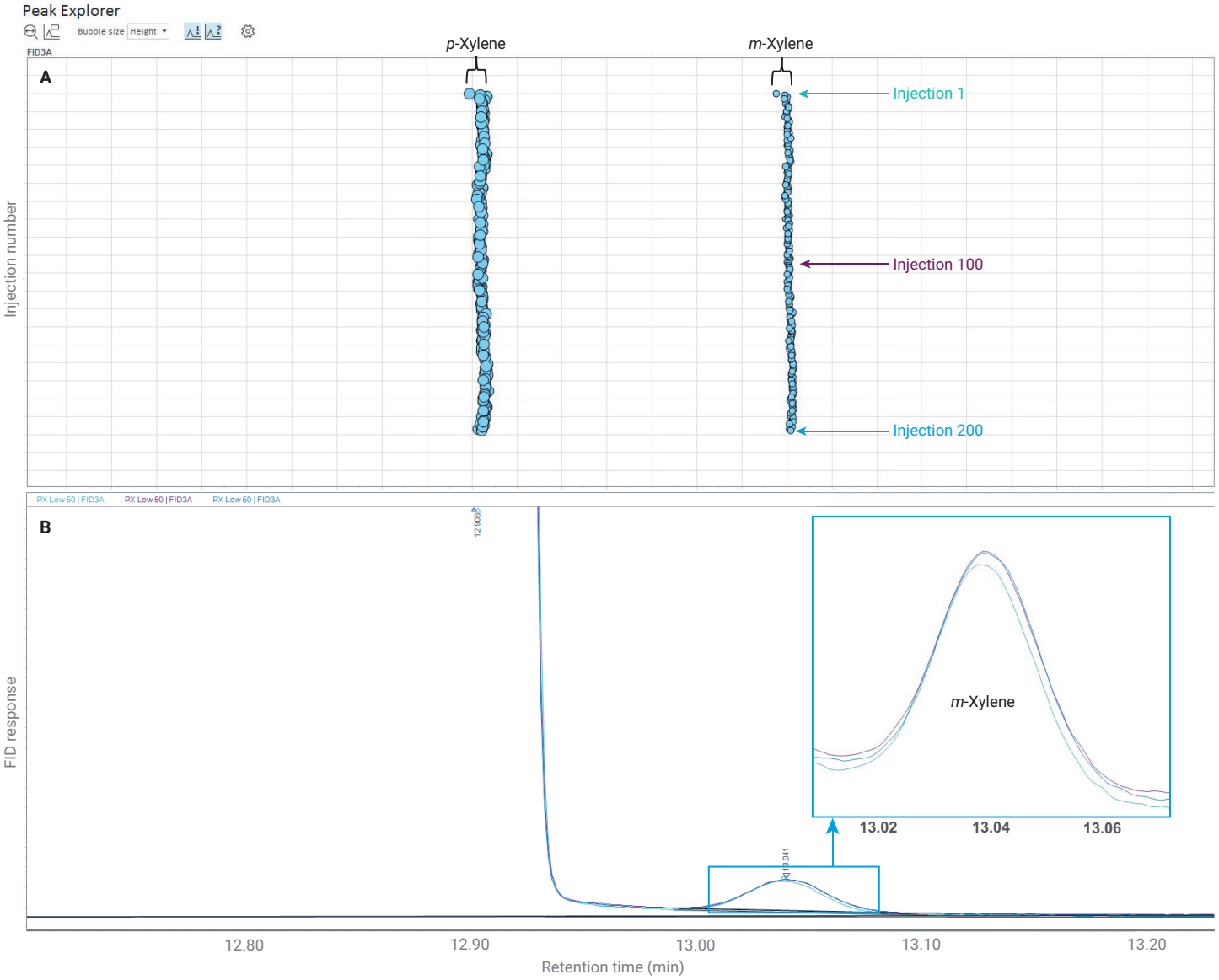

Figure 4. Retention precision for 50 ppm MX in PX across 200 injections using the Agilent J&W HP-INNOWax column, visualized using (A) Peak Explorer and (B) chromatographic overlay in Agilent OpenLab CDS.

As predicted, the narrower DB-WAX FF column yields a faster separation with less resolution than the wider HP-INNOWax column, with an approximate USP resolution of 0.49 calculated in OpenLab CDS. A major contributor to the lower resolution is the thinner stationary phase of the DB-WAX FF, which significantly reduces the sample capacity of the column and further broadens the PX matrix peak. This was partially offset by using a larger split ratio of 750:1 compared to 250:1 on the HP-INNOWax; however, this resulted in a reduction of S/N and, by extension, an increase in LOD and LOQ by approximately the same ratio.

The HP-INNOWax column yields excellent resolution for MX at approximately 1.28 using the USP resolution calculation in OpenLab CDS. Though still overloaded, the PX matrix peak is significantly sharper due to the increased sample capacity from the thicker phase. While this does lead to increased broadening of MX, more resolution permits increasing the injection volume to improve LOD and LOQ. Figure 5 shows the effect of increasing injection volume on the resolution and S/N for 10 ppm MX. It should be noted that resolution will decrease as the column ages, so this configuration effectively enables the user to choose their preferred balance between sensitivity and robustness by simply tuning the injection volume.

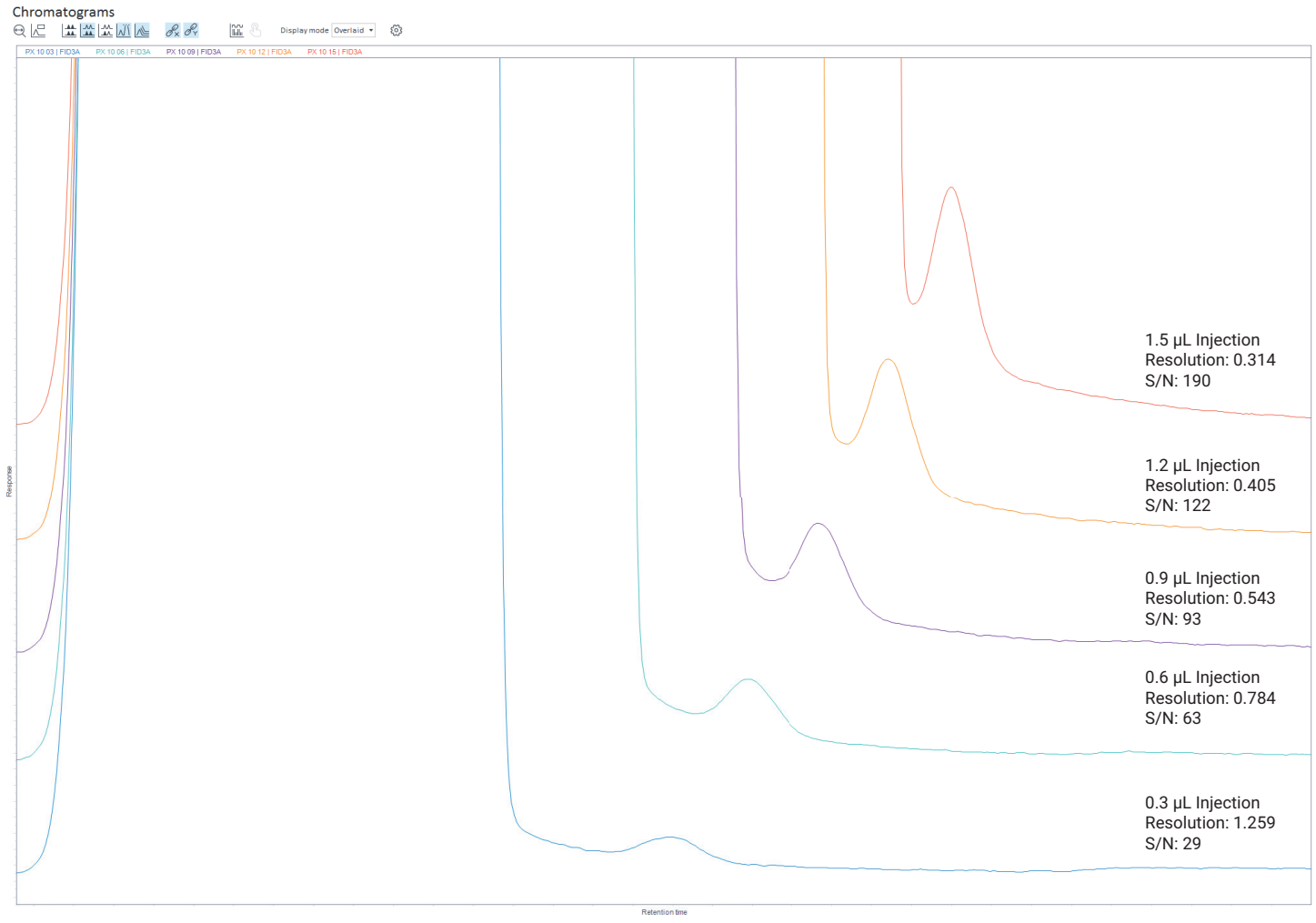

Figure 5. Stacked chromatograms showing the effect of injection volume on resolution and signal-to-noise for 10 ppmw MX in PX using the Agilent J&W HP-INNOWax column.

Trace-level linearity

Low-concentration standards of *m*-xylene were gravimetrically prepared in purified *p*-xylene at target concentrations of 10, 20, 30, 40, and 50 ppmw, with actual concentrations of 9.98, 20.01, 30.34, 42.23, and 49.75 ppmw. Each standard was analyzed in triplicate and plotted using a linear best-fit model with the origin ignored. The resulting models for both columns are shown in Figures 6 and 7, with R^2 values of 0.99975 and 0.99950, respectively. These results highlight the advantages of the advanced pneumatic and thermal controls of the 8890B GC, demonstrating consistent chromatographic performance and linear response for MX at concentrations near the LOD and LOQ.

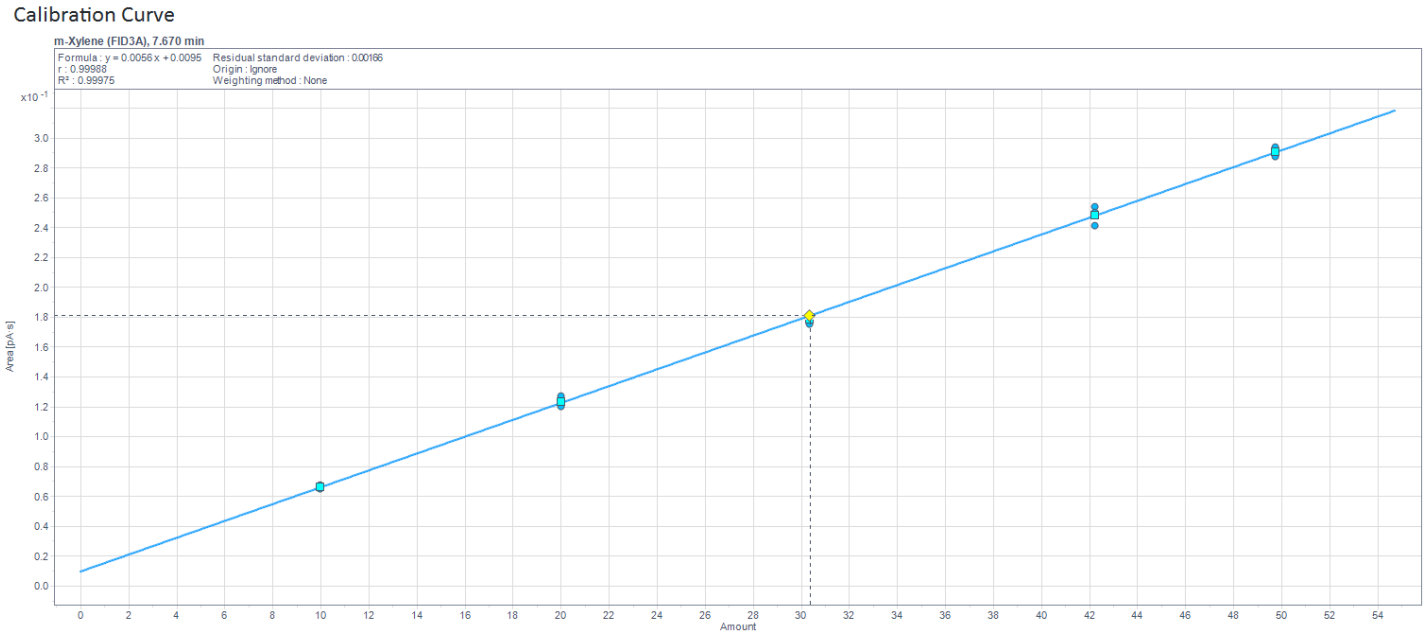

Figure 6. Calibration model for MX at concentrations of 10, 20, 30, 40, and 50 ppmw for the high-speed method using the Agilent J&W DB-WAX FF column.

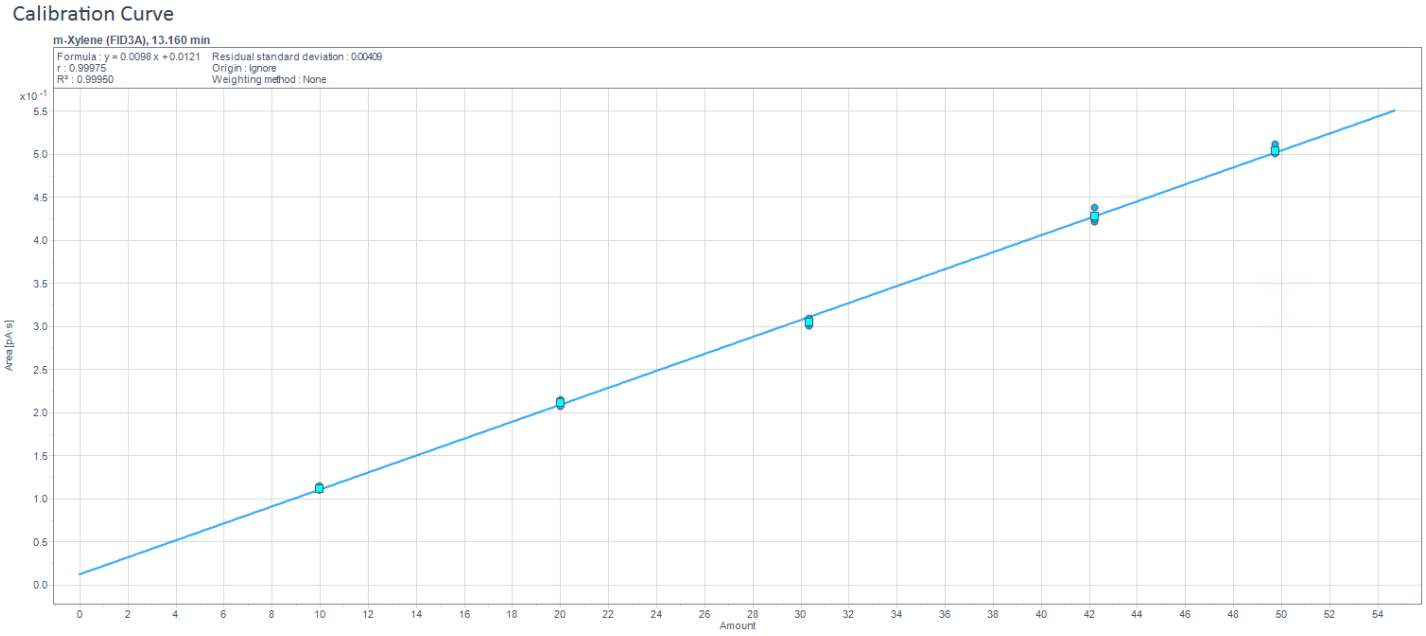

Figure 7. Calibration model for MX at concentrations of 10, 20, 30, 40, and 50 ppmw for the high-resolution method using the Agilent J&W HP-INNOWax column.

Noise reduction

There are many different approaches to calculating LOD and LOQ, and most are either directly or indirectly a function of measurement noise. Reducing noise is advantageous for trace analysis and was achieved by using filters on the FID gases and decreasing the FID data rate to 10 Hz.

Figure 8 shows a comparison of 10 ppm MX analyzed with the filters both in service and bypassed; ASTM noise[2] (calculated in OpenLab CDS) was approximately 45% lower with the filters in service. It should be noted that the impact of filters may vary from user to user based on gas quality and the cleanliness of the lines from the gas source to the GC.

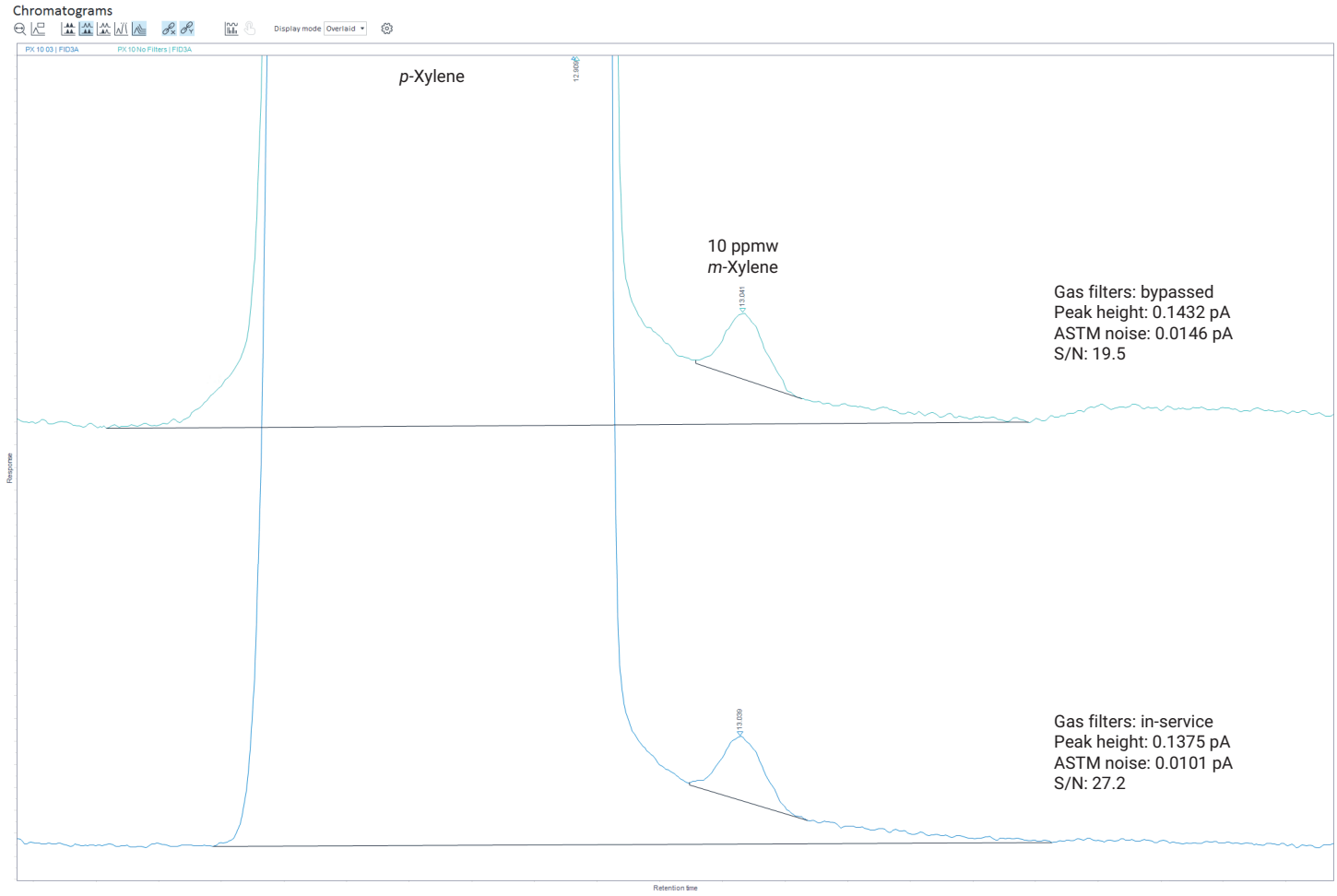

Figure 8. Chromatograms showing the effect of carrier and detector Agilent Gas Clean filters on peak height, noise, and signal-to-noise for 10 ppmw MX.

The 8890B GC FID can acquire data from 5 to 1,000 Hz to accommodate even the fastest chromatography, but higher data rates result in increased noise. Therefore, it is advantageous to use the slowest necessary data rate for trace analysis. Figure 9 shows 10 ppm MX acquired at FID data rates of 50, 20, and 10 Hz and the resulting impact on peak shape and ASTM noise. The three data traces in Figure 9 were captured simultaneously from a single injection, allowing users to easily evaluate the impact of data rate on their results by adding additional signal traces to the acquisition method in OpenLab CDS. A rate of 10 Hz was selected as the optimal rate for both the 200 µm HP-INNOWax and the 100 µm DB-WAX FF column configurations.

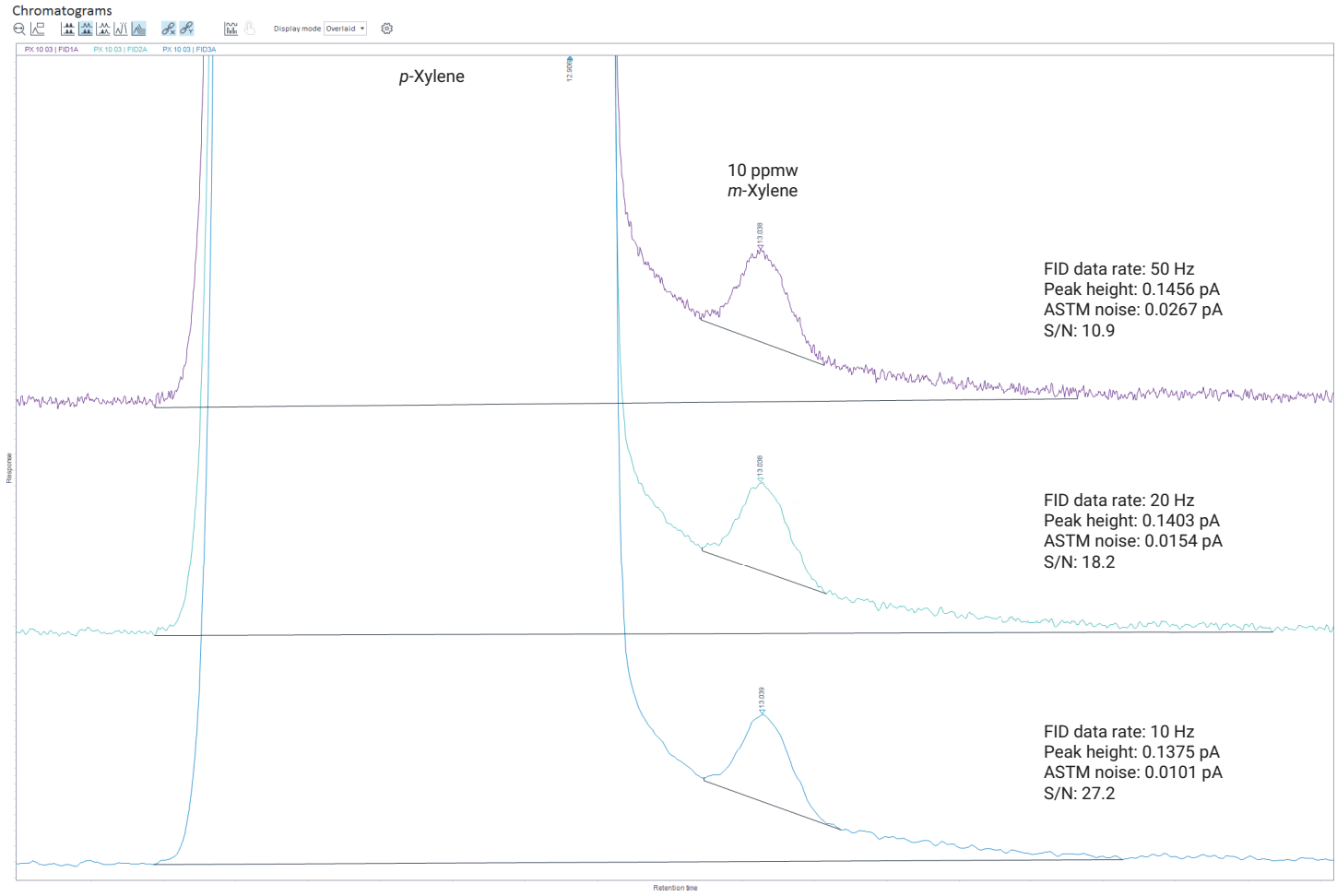

Figure 9. The effect of FID data rate on peak height, noise, and signal-to-noise for 10 ppmw MX.

GC Assist: EMF counter thresholds

The valuable EMF counters on the 8890B GC help customers maximize instrument uptime by tracking instrument consumable health and providing alerts when parts will soon require maintenance after exceeding a user-defined injection count or time threshold. Each EMF counter has thresholds for "Service Warning" and "Service Due," helping users proactively maintain control of their instrument rather than reactively making repairs when issues arise. The thresholds come preconfigured with default values; however, the optimal values, which are unique to each method and instrument configuration, are influenced by several factors. This section explains appropriate thresholds for the consumables described in this application note, providing users with a robust starting point. Users may need to adjust these values based on their specific use case, sample throughput, and quality of utilities. Some major factors that can impact these thresholds include:

- **Sample cleanliness:** Process control samples are more likely to contain contaminants that can reduce consumable life compared to finished product samples.
- **Gas cleanliness:** Dirtier gases and contaminated delivery systems (piping, tubing, valving, and others) will degrade both the filters and stationary phase more quickly.

Inlet septum

Service Warning at 250–300 injections: The Advanced Green septum will eventually fail due to coring by the syringe needle. Using a 23/26-gauge tapered needle can slow this process. The septum should be changed before failing to reduce oxygen exposure to the column.

Inlet liner

Service Warning at 500–1,000 injections: The primary liner contaminants include septa particles and contaminants from process samples that can yield ghost peaks in the chromatogram. Replace the liner every third or fourth septum change, or as needed.

Gas filter maintenance

Service Due at 365 days: The Gas Clean filters should be changed yearly or when the indicator changes color. Gas quality will strongly impact this frequency. The optional Gas Clean filter sensor monitors the filter life and will notify the user when the filter is saturated, automatically resetting the EMF counter when a new filter is installed.

Syringe injections

Service Warning at 2,000 injections, or as needed: Syringe failure can be difficult to predict and is highly dependent on sample cleanliness. PX samples are not expected to negatively impact syringe life, and the syringe should be changed as needed. Leveraging peak evaluation to monitor the PX peak height can give additional insight into current syringe life.

Injections since last column trim

Service Warning at 10,000 injections: Heavy contaminants in samples will eventually contaminate the column head and lead to loss of resolution between MX and PX. The column should be trimmed as needed. For systems injecting high-purity samples and using high-purity gases, this should be an infrequent activity.

Inlet gold seal

Use default thresholds, or as needed: PX samples typically do not contain anything that will damage the gold seal. A visual inspection of the gold seal can be done whenever the liner is changed, and the gold seal should be replaced if discoloration or debris can be seen.

GC Assist: peak evaluation

In addition to the EMF counters, the 8890B GC has a suite of self-monitoring and evaluation capabilities called GC Assist. Peak evaluation is a GC Assist feature that enables the GC to monitor chromatographic performance for one or more peaks against a user-defined reference chromatogram. When a sample produces data that is significantly different from the reference, the user is notified. Peak evaluation is conceptually similar to quality control charting, but is used to evaluate the performance of the GC independently from the performance of the method. This section will discuss how to leverage peak evaluation to support the long-term application of the 8890B GC for trace analysis of MX in PX.

Enabling peak evaluation requires generating a reference chromatogram for comparison, setting upper and lower relative percent thresholds for the desired comparison metric, and applying the settings to an acquisition method. For the separation of MX and PX, configuring peak evaluation to monitor the resolution between both peaks provides an early indication of approaching downtime for column maintenance. In addition, monitoring the height of the PX peak will alert users to an issue with the injection.

Table 4 includes recommended peak evaluation thresholds for monitoring PX peak height, MX resolution, and retention times for both compounds.

Table 4. Recommended initial peak evaluation thresholds for monitoring the chromatographic performance of MX and PX.

Compound Name	Metric	Lower Limit (%)	Upper Limit (%)	Comparison Peak
p-Xylene	Retention Time	5	5	–
	Peak Height	5	5	–
m-Xylene	Retention Time	5	5	–
	Resolution	10	10	*p*-Xylene

These metrics can be monitored for every injection, but note that process control samples can unexpectedly vary in composition and potentially cause a false-positive failure. Because the reference chromatograms used by peak evaluation are tied to individual acquisition methods, different sample types can be monitored for different characteristics using uniquely named acquisition methods specific to those sample types. For example, a manufacturing-site laboratory that analyzes both process control and product certification samples could monitor resolution between MX and PX on quality control samples while monitoring PX peak height on all samples. In this example, peak evaluation would provide long-term health monitoring of the separation using a quality control sample with consistent composition and immediate warning of an improper injection for all PX samples. Figure 10 shows the trend plot for PX peak height in the GC Assist interface, using a 5% threshold. When a peak evaluation threshold is exceeded, the 8890B GC can be programmed to abort the sequence or continue running. In this way, chromatographic performance monitoring can be highly customized based on user needs.

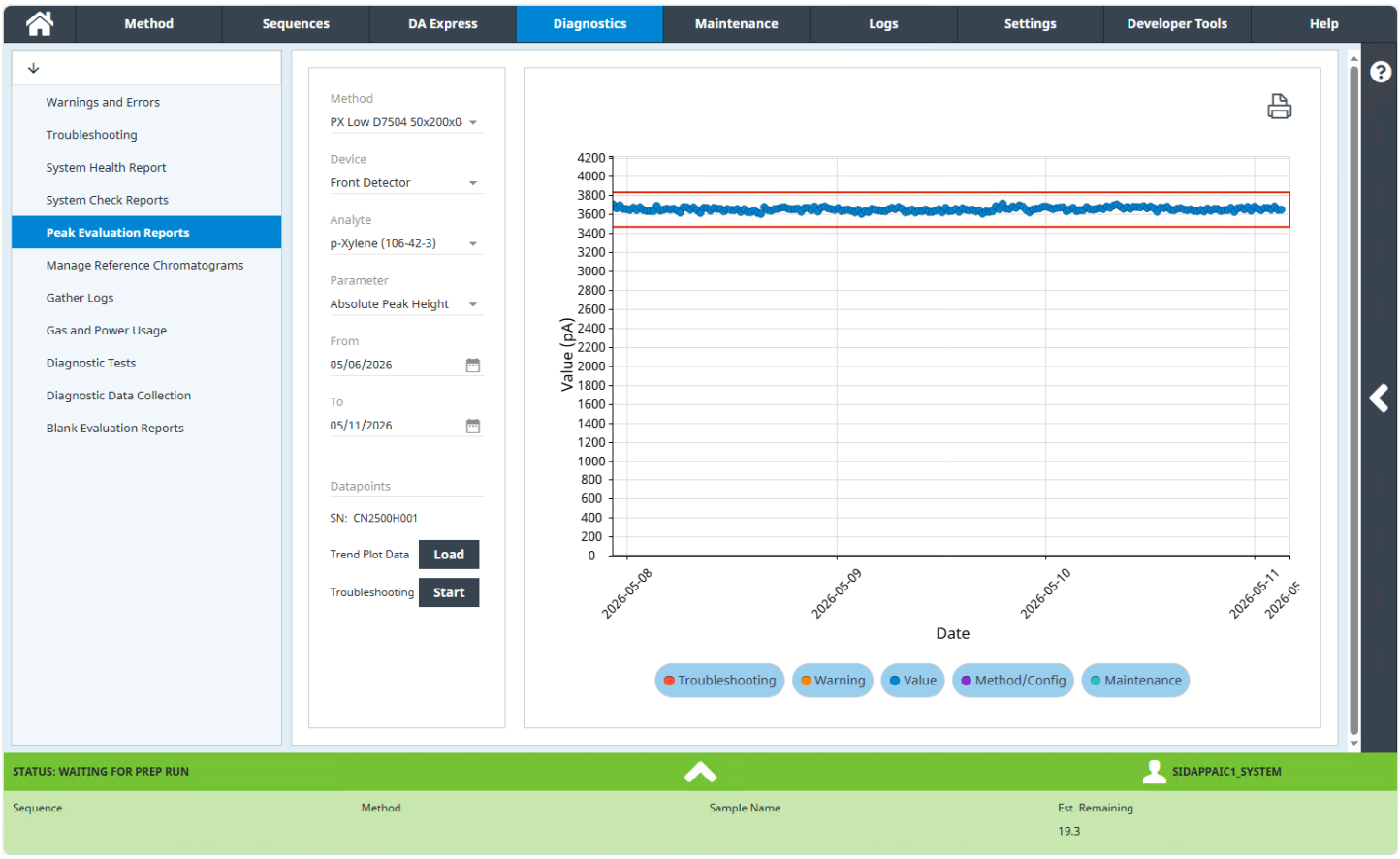

Figure 10. Peak evaluation report showing the trend plot for PX peak height with 5% threshold (red lines) in the Agilent GC Assist interface.

Conclusion

The Agilent 8890B GC system—powered by the highest-quality Agilent columns and supplies—delivers a robust, sensitive solution for measuring trace-level *m*-xylene (MX) in *p*-xylene (PX). The Agilent J&W HP-INNOWax column produced the highest-resolution separation and the best retention time stability, with an S/N ratio of 98.5 for 50 ppm MX. The narrower Agilent J&W DB-WAX FF column yielded a 40% faster run time at the cost of lower resolution and retention time stability, with an S/N ratio of 27.7 for 50 ppm MX. The integrated Agilent GC Assist features, peak evaluation and early maintenance feedback (EMF) counters, provide intelligent, automated monitoring of system and separation health, maximizing instrument uptime and supporting long-term success with routine trace analysis.

References

1. ASTM International. Standard Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography and Effective Carbon Number; ASTM D7504-23; ASTM International: West Conshohocken, PA, **2023**. https://www.astm.org/d7504-23.html
2. ASTM International. Standard Practice for Testing Fixed-Wavelength Photometric Detectors Used in Liquid Chromatography; ASTM E685-93(2021); ASTM International: West Conshohocken, PA, **2021**. https://www.astm.org/e0685-93r21.html

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