GC-MS Troubleshooting: Avoid Poor Performance from Slow Acquisition Speeds

Clearly Define Your Mass Peaks by Optimizing Your MS Data Acquisition Rate

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Abstract
Data acquisition rate is an essential component to achieving accurate and precise chromatographic results. A data acquisition rate that is too slow of a speed often leads to loss of sensitivity, precision, and accuracy while an acquisition speed that is too fast may cause excessive noise and decreased sensitivity. In this article, we discuss how to select the ideal MS data acquisition rate to achieve optimal integration and reliably calculate peak areas.

Related Products

- *GC_MS CLeaning Kit*
- *ETP Electron Multipliers for Mass Spectrometry*
- *Inland 45 Pump Oil*
- *Ion Source Cleaning Powder*

Introduction

The foundation for quantitative chromatography is the relationship between peak area and compound concentration. The ability to define the shape of the peak is the cornerstone for reliably calculating peak area, but there are method parameters that can hamper our ability to get a well-defined peak, undermining our ability to achieve accurate and precise results.

To avoid poorly defined peaks, it is important to make sure your mass spectrometer's data acquisition rate is matched to chromatographic performance. By comparing the width of the chromatographic peak with the MS data acquisition rate, you can verify that a sufficient amount of data is being collected to accurately define the shape of the chromatographic peak.

In addition to the shape of the peak itself, data acquisition rates can affect how well the mass spectrometer is able to accurately determine the spectral composition of a given peak, which can affect your ability to properly identify a compound.

In this article, we'll explore examples of deformed peak shapes and skewed mass spectra caused by improperly set data acquisition rates and discuss how to correct data acquisition issues.

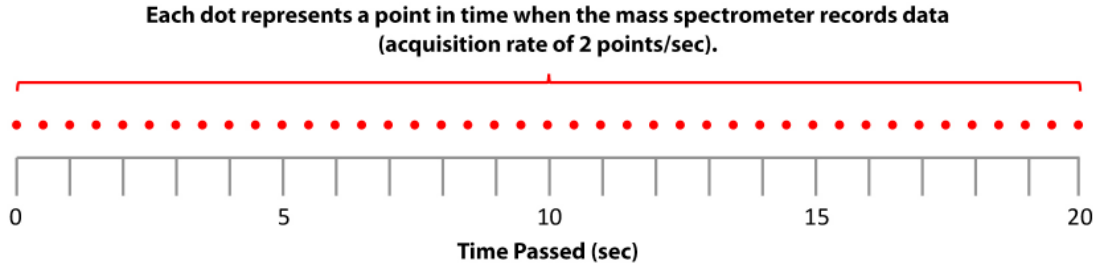
How are Peak Shape and MS Data Acquisition Rate Related?

Since most mass spectrometers coupled to chromatographs rely on quadrupole mass analyzers (single or triple quad), we'll use a single-quad MS for this example.

A Data Acquisition Rate that is Too Slow

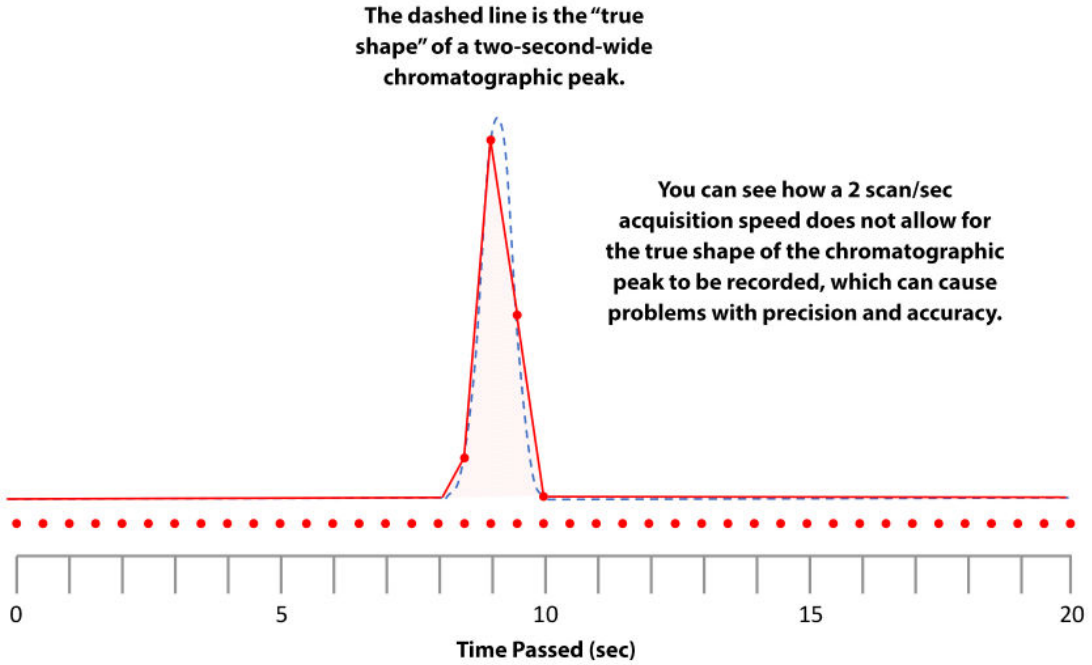
Let's consider a data acquisition rate that is too slow as our first example. In this case, the MS is reporting data twice every second, or at 2 Hz (Figure 1).

Figure 1: Points Recorded over Time at a Data Acquisition Rate of 2 Hz



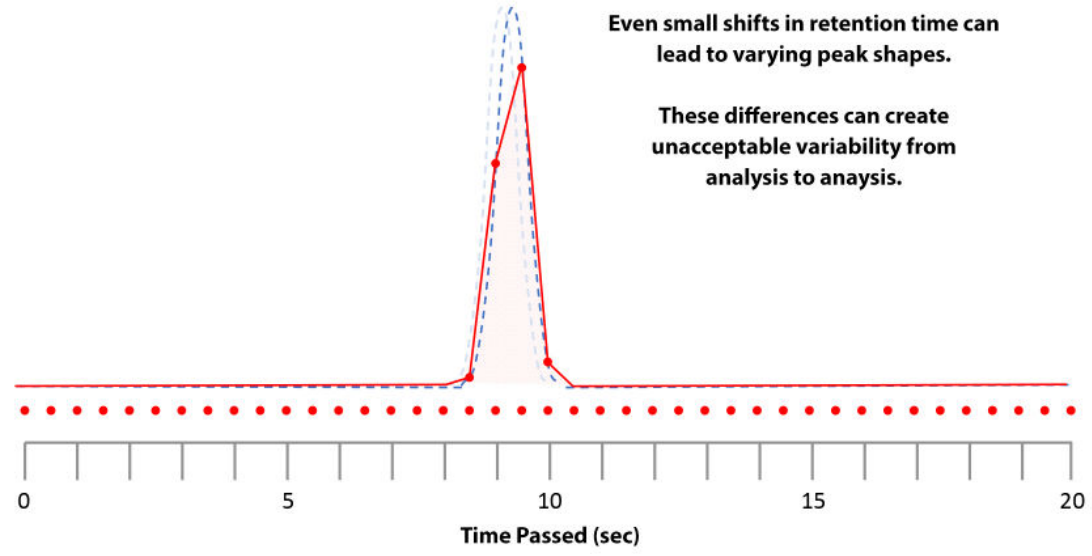
If a 2-second-wide chromatographic peak enters the mass spectrometer, a 2 Hz acquisition speed will only record a few data points across its width. As shown in Figure 2, this results in a jagged-looking peak that cannot capture the complete peak area. At best, this results in some degree of a loss in sensitivity since the available peak area is not fully captured due to the slow acquisition speed. Quantitation accuracy may suffer as well, but since the instrument was likely calibrated under the same low acquisition speed conditions, it may not be off as much as if the instrument had been calibrated using an ideal data acquisition rate.

Figure 2: A data acquisition rate that is too slow does not capture the true peak shape.



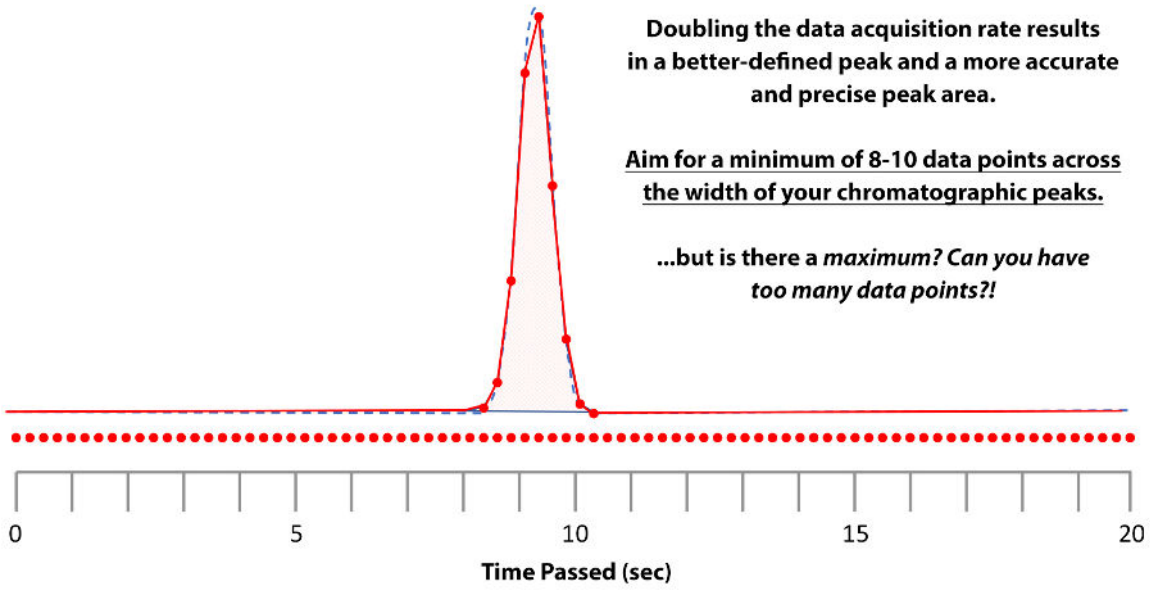
While calibrating under the slow acquisition rate mitigates some of the accuracy error, it does not account for the degradation in precision that will likely also occur. Even a small shift in retention time from analysis to analysis can affect how much of the peak is recorded with a slow acquisition rate (Figure 3).

Figure 3: Varying Retention Times and a Slow Data Acquisition Rate



By doubling the data acquisition speed to 4 Hz, we can see that the true shape of the chromatographic peak is better defined, allowing for better accuracy, precision, and sensitivity (Figure 4).

Figure 4: Peak Area after Doubling the Data Acquisition Rate



A Data Acquisition Speed that is Too Fast

If acquiring data too slowly leads to a poorly defined peak shape that reduces sensitivity, precision, and, in some cases, accuracy, it would be easy to think that collecting data as fast as the instrument will allow is a good solution. An infinite number of points across the peaks would perfectly define the peak shape, right? In reality, though, there is a "too fast" just as there is a "too slow" when it comes to data acquisition rate.

To explain how a mass spectrometer data acquisition rate can be too fast, we must first understand how data is collected. A quadrupole mass spectrometer often has a balance to strike when acquiring data. As the MS scans the mass range, it does not simply collect a single data point at a given m/z ratio value. Rather, it collects multiple "samples" and then averages them together. And, as is typical with averages, the greater the sample size, the better a representation the average will be, leading to an improved signal to noise ratio. Collecting all of those samples takes time, though. If you set an overall data acquisition rate that is too fast, you may end up acquiring more points across the chromatographic curve, but the data will be noisier, and the signal to noise will be reduced considerably. The result is a peak that no longer looks "blocky" but one that looks jagged.

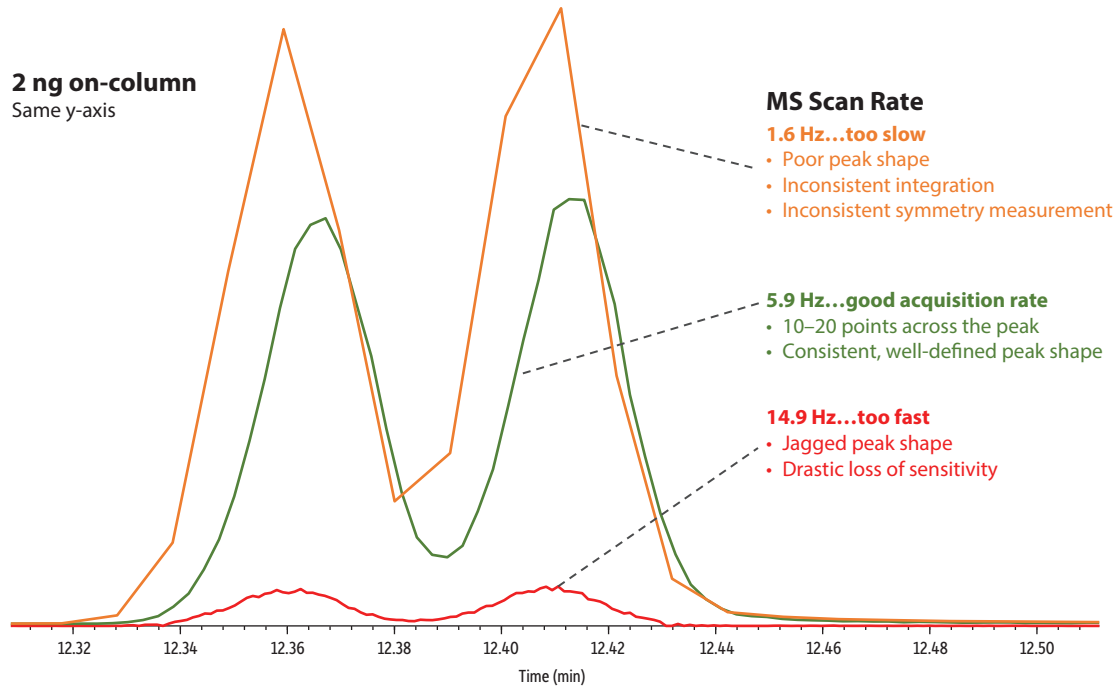
Poor peak shape caused by a data acquisition rate that is too fast can pose a challenge to the processing software's auto-integration, resulting in the need to do more manual data review. Additionally, a scan rate that is too fast can result in significantly lower abundances of the ions detected, which leads to a decrease in sensitivity.

What MS Data Acquisition Speed is "Just Right"?

How do you avoid the pitfalls of a nonoptimal acquisition rate and select conditions that are "just right?" Thankfully, there is a helpful rule of thumb. In general, aim to have 10-20 points across the width of the chromatographic peak (8-10 at minimum) to ensure an accurate peak shape for quantitation. Exactly how to set your specific instrument to achieve this goal will depend on the mass spectrometer itself as well as the chromatography. For instance, a peak that is 2 seconds wide will require a data acquisition rate that is twice as fast than a peak that is 4 seconds wide. Tailoring MS conditions as much as possible to match the chromatographic performance is ideal.

Figure 5 illustrates how chromatographic peak shape and overall intensity changes with different scan speeds, highlighting the value of making sure your scan speed is tailored to your chromatography.

Figure 5: The effects of scan rate on peak shape.



Also, check to see if your instrument allows you to adjust the data acquisition parameters during the course of an analysis for additional optimization. Scanning a narrower, lower range of ions earlier in the chromatographic analysis when lower molecular weight compounds are likely to elute can provide a faster acquisition rate when the peaks are also likely to be the narrowest.

If you have any questions about MS data acquisition rates, analysis, or your chromatography, don't hesitate to contact Restek or check out our other resources at www.restek.com.

24819

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Inland 45 Pump Oil

Recommended for most mass spectrometers.

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- Low vapor pressure 10^{-7} torr.
- Nontoxic and noncorrosive.
- Compatible with Buna-N, neoprene, and Viton seals.
- Optimum vacuum pump performance.
- Lowest mass spectrometer background.
- Recommended for optimum mass spec performance.

Description	qty.	Similar to Part #	cat.#
Inland 45 Pump Oil	1 liter	Agilent 6040-0798, 6040-0834	24819

GC-MS Cleaning Kit

Poor sensitivity, loss of sensitivity at high masses, or high multiplier gain during an auto-tune are all indicators that your mass spectrometer source may need to be cleaned. Restek has assembled all of the necessary components for cleaning and polishing your ion source.

27194

The Restek GC-MS Cleaning Kit (cat.#s 27194, 27195) Includes:

- Lint-free nylon gloves (small, 2 pair)
- Lint-free nylon gloves (large, 2 pair)
- Lint-free cotton cloth, 9 x 9 (10-pk.)
- Micro mesh 4 x 6 sheet (4-pk.)
- Aluminum oxide (1-kg jar)
- Cotton tip applicators
- Tweezers, large
- Tweezers, small
- Septum puller
- Rotary tool, battery operated (optional, 27194)
- Tool kit bag

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- Lint-free nylon gloves (large, 2 pair)
- Lint-free cotton cloth, 9 x 9 (10-pk.)
- Micro mesh 4 x 6 sheet (4-pk.)

Description	qty.	cat.#
Mass Spec Cleaning Kit with Rotary Tool	kit	27194
Mass Spec Cleaning Kit without Rotary Tool	kit	27195

Note: cat.# 27194 contains a rotary tool with a rechargeable Li-ion battery, that requires a 110 V power supply and a US-style (Type A) outlet to charge.

Ion Source Cleaning Powder

Use this aluminum oxide powder to clean surfaces that contact the sample or ion beam when you encounter poor sensitivity and inadequate abundances at high masses.

22685

Description	qty.	Similar to Part #	cat.#
Ion Source Cleaning Powder	1 kg	Agilent 8660-0791	22685

ETP Electron Multipliers for Mass Spectrometry

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- Two-year shelf life guarantee.
- Discrete dynode design results in extended operating life.
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The multi-dynode approach of all ETP electron multipliers results in longer lifetimes—and better sensitivity—compared with channel electron multipliers (CEM) or continuous-dynode multipliers.

The electron multipliers manufactured by ETP use a proprietary dynode material. This material has a number of properties that make it very suitable for use in an electron multiplier. It has very high secondary electron emission, which allows exceptional gain to be achieved from each dynode. This material is also very stable in air. In fact, an ETP multiplier can be stored for years before being used. As a direct result of the high stability of the active materials used in ETP multipliers, they come with a two-year shelf life warranty (stored in original, sealed package). Many testing laboratories take advantage of this long shelf life by keeping a replacement ETP multiplier on hand, ready for immediate installation. This keeps instrument downtime to a minimum.

For a typical ETP electron multiplier for GC-MS, the total active dynode surface area is ~1000 mm^2. This can be compared to a standard continuous dynode multiplier that has a total channel surface area of only around 160 mm^2 (for a channel with 1 mm diameter and 50 mm length). This increased surface area spreads out the workload of the electron multiplication process over a larger area, effectively slowing the aging process and improving operating life and gain stability.

Description	Instrument	qty.	cat.#
ETP Electron Multiplier for Mass Spectrometry	for Agilent 5973 & 5975 GC-MS (includes mount for initial installation)*†	ea.	23074
	for Agilent 5973 & 5975 GC-MS and LC-MSD (Replacement Multiplier)*†	ea.	23075

*Note: The electron multipliers have been specifically developed to retrofit the original manufacturer's equipment. The detector incorporates a modular design to facilitate ease of replacement and additional innovations intended to enhance performance. First-time installation requires a mount that includes the mechanical housing. After initial installation, only the replacement electron multiplier is required.
†This unit is designed for use in the 5975, 5973 GC, and the LC-MSD (not for 5975C Triple Axis Detector).

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