

ISQ 7610

# Extended linear dynamic range with the XLXR detector system on the Thermo Scientific ISQ 7610 single quadrupole GC-MS

## Authors

Amit Gujar<sup>1</sup>, Tim Anderson<sup>1</sup>,  
Kenneth M. Free<sup>1</sup>, and Adam Ladak<sup>2</sup>

<sup>1</sup>Thermo Fisher Scientific, USA

<sup>2</sup>Thermo Fisher Scientific, UK

## Keywords

Detector linear dynamic range,  
spectral fidelity, method consolidation

## Introduction

Analytical laboratories that are analyzing food, environmental, pharma, petrochemical, or clinical samples utilize GC-MS or GC-MS/MS for both targeted and known unknowns analysis and employ commercially available libraries such as NIST or Wiley. These laboratories often face challenges when analyzing compounds at both high and low concentrations in a single run. When analytes of interest are present in widely varying concentrations, it can be difficult to accurately quantify the compounds due to the limits of the dynamic range of the mass spectrometer detector system. This leads to the analysis being split into class- or compound-specific methods, which impedes the sample throughput in a laboratory. Even single methods are subject to issues when a very high concentration sample is analyzed on the system, as it cannot be quantified accurately and a repeat analysis must be performed with a diluted sample to bring the concentration of the analyte within the calibration range. This adds further time to the sample analysis and can delay important results getting to clients.

The issues with limited detector dynamic range are not isolated to quantification. When performing sample screening, for example in drugs of abuse analysis, if a compound saturates the detection system due to high concentration, the mass spectral fidelity is affected. This poor spectral quality can lead to poor library matches and potential misidentification of the compound of interest or even a false negative. If the saturation of the detector is identified, the samples can be diluted and re-run; however, this adds to sample processing time and can potentially cause low-level contaminants to be diluted below detection limits and not reported.

Here, the new Thermo Scientific™ XLXR™ detector system is compared with the previous-generation electron multiplier detection system on the Thermo Scientific™ ISQ™ 7610 GC-MS. The two main advantages of the XLXR detector system are the greater linear dynamic range and the extended lifetime compared to the previous generation. These improvements have been made possible because of a complete redesign of the electron multiplier system, better material technology of the multiplier dynodes, and an improved electrometer that can manage the increased current output from the multiplier. The XLXR detector comes as a standard offering on all ISQ 7610 and Thermo Scientific™ TSQ™ 9610 GC-MS/MS systems.

## Experimental

The ISQ 7610 single quadrupole mass spectrometer coupled with a Thermo Scientific™ Trace™ 1610 GC and AI/AS 1610 autosampler was used for all the experiments. Initially, the system was run using the previous electron multiplier detection system to perform analysis of an octafluoronaphthalene (OFN) standard calibration curve between 4.3 fg and 43 ng on-column using selected ion monitoring (SIM) acquisition. The detection system was then replaced with the XLXR detector and the experiment repeated. No other conditions were modified on the systems to ensure the comparison was being made on the detectors only. The purpose of this experiment was to go from very low levels (near LOD) to very high levels (near expected detector saturation) to gauge the linear dynamic range of the detector. Table 1 shows the conditions used for all the experiments.

**Table 1. Experimental conditions for the linear dynamic range studies**

| AS 1610 Autosampler               |  |
|-----------------------------------|--|
| Syringe                           | 10 µL, 25 gauge, 50 mm length, cone tip ( <a href="#">P/N 36500525</a> )       |
| Injection volume                  | 1 µL   |
| Pre-injection solvent and cycles  | None   |
| Sample rinses                     | 3  |
| Post-injection solvent and cycles | None   |
| TRACE 1610 GC system              |  |
| Column                            | Thermo Scientific™ TraceGOLD™ TG-SQC, 15 m × 0.25 mm × 0.25 µm                 |
| Liner                             | Splitless Liner single taper with wool, 4 mm ID, 78.5 mm length (P/N 453A0924) |
| SSL mode                          | Splitless  |
| Inlet temperature                 | 220 °C   |
| Split flow                        | 50 mL/min  |
| Splitless time                    | 0.8 min  |
| Septum purge flow                 | Constant flow of 5.0 mL/min  |
| Carrier flow                      | Constant He flow of 1.2 mL/min   |
| Oven program                      | 40 °C (1.0 min), 30 °C/min to 250 °C (2 min)                                   |
| ISQ 7610 GC-MS system             |  |
| MS transfer line temperature      | 250 °C   |
| Ion source temperature            | 200 °C   |
| Source type                       | Thermo Scientific™ ExtractaBrite™  |
| Ionization mode, electron energy  | EI, 70 eV  |
| Emission current                  | 50 µA  |
| Scan range (Da)                   | SIM <i>m/z</i> 272   |
| Dwell time                        | 0.2 s  |
| Detector gain                     | 3E+05  |

A second experiment, with slightly different experimental conditions was conducted near the upper range of the detector system. Here OFN was injected from 0.43 ng to 43 ng and the data acquired in Full Scan mode. The purpose of this experiment was to compare the mass spectral fragmentation pattern and the spectral fidelity of the two detectors at high concentrations. Table 2 gives the experimental conditions for this experiment.

**Table 2. Experiment conditions for mass spectral fragmentation and spectral fidelity experiments**

| <b>AS 1610 Autosampler</b>        |  |
|-----------------------------------|--|
| Syringe                           | 10 $\mu$ L, 25 gauge, 50 mm length, cone tip ( <a href="#">P/N 36500525</a> )  |
| Injection volume                  | 1 $\mu$ L  |
| Pre-injection solvent and cycles  | None   |
| Sample rinses                     | 3  |
| Post-injection solvent and cycles | None   |
| <b>TRACE 1610 GC system</b>       |  |
| Column                            | Thermo Scientific™ TraceGOLD™ TG-5ms, 30 m $\times$ 0.25 mm $\times$ 1 $\mu$ m |
| Liner                             | Splitless Liner single taper with wool, 4 mm ID, 78.5 mm length (P/N 453A0924) |
| SSL mode                          | Splitless  |
| Inlet temperature                 | 220 $^{\circ}$ C   |
| Split flow                        | 50 mL/min  |
| Splitless time                    | 0.8 min  |
| Septum purge flow                 | Constant flow of 5.0 mL/min  |
| Carrier flow                      | Constant He flow of 1.2 mL/min   |
| Oven program                      | 40 $^{\circ}$ C (1.0 min), 30 $^{\circ}$ C/min to 250 $^{\circ}$ C (0 min)     |
| <b>ISQ 7610 GC-MS system</b>      |  |
| MS transfer line temperature      | 250 $^{\circ}$ C   |
| Ion source temperature            | 300 $^{\circ}$ C   |
| Source type                       | ExtractaBrite  |
| Ionization mode, electron energy  | EI, 70 eV  |
| Emission current                  | 50 $\mu$ A   |
| Scan range (Da)                   | 50–300   |
| Dwell time                        | 0.2 s  |
| Detector gain                     | 3E+05  |

## Results and discussion

### Linearity and quantification

The analysis of the OFN calibration curve from 4.3 fg to 43 ng on both systems showed clear improvements using the XLXR detector. Seven orders of linear dynamic range were achieved on the system. On the ISQ 7610 GC-MS, the linearity on the previous electron multiplier detection system gives a reasonable  $R^2$  value of 0.994. However, the % RSD of the residual values is 17.9, and it is

clear that the data at 4.3 ng does not fit on the linear curve. If this curve were used to quantify samples, the results would be inaccurate. With the XLXR detector, the linearity is improved with an  $R^2$  value of 0.9995 and a % RSD of the residual values is 5.4. This curve would allow accurate quantification over the whole range. Figure 1 shows a comparison of the linearity on the ISQ 7610 GC-MS using each detector.

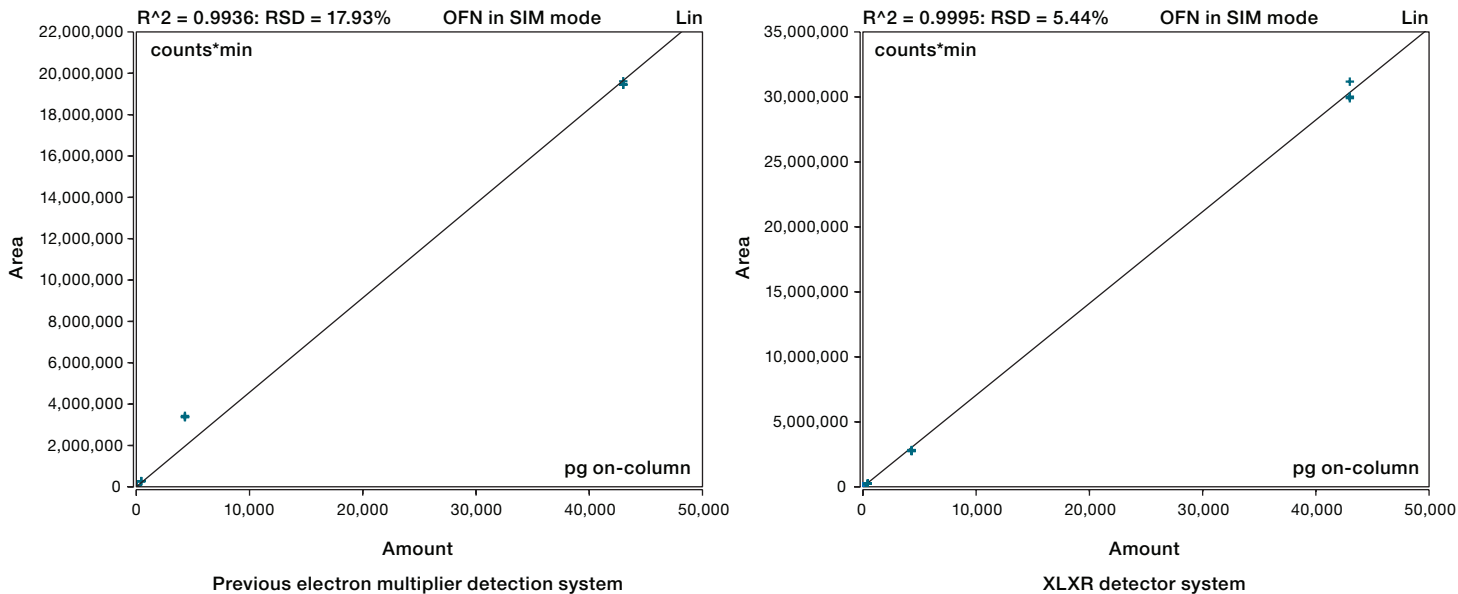


Figure 1. Comparison of the previous electron multiplier detection system and the XLXR detector on the ISQ 7610 GC-MS using OFN over a range of 4.3 fg to 43 ng on-column

The benefits of the XLXR detector for linearity over a large dynamic range are even clearer when reviewing the data for the second set of experiments conducted at higher concentration range. Using the previous electron multiplier detection system, it is impossible to use a linear regression on the curve and a quadratic curve must be used. This is due to the calibration points of 20 ng and 43 ng saturating the detector. If a sample at high concentration was run on this detector, it would not

be accurately quantified and therefore requiring a reanalysis. In comparison, the calibration curve on the ISQ 7610 GC-MS equipped with the XLXR detector gives a linear regression with an  $R^2$  value of 0.9997 and a percentage RSD of 2.5%. Figure 2 shows the comparison of the linearity on the ISQ 7610 GC-MS using each detector. This extended dynamic range allows for methods to be consolidated and accurate quantification of trace and high-level compounds to be performed in a single run.

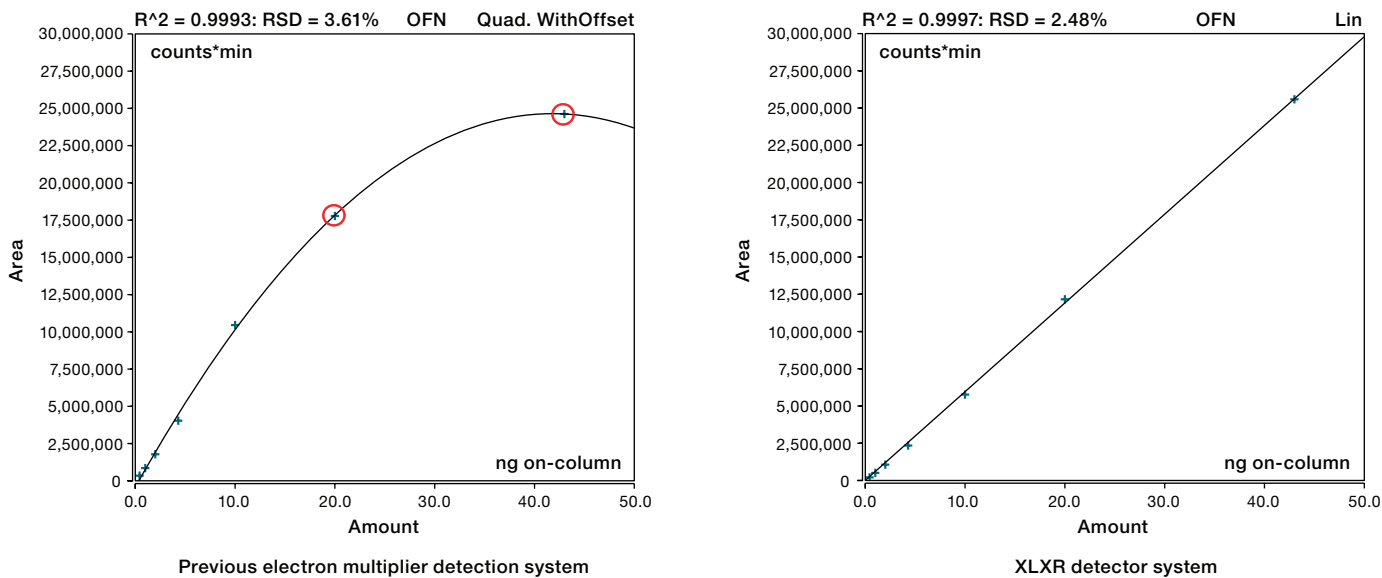


Figure 2. Comparison of the standard electron multiplier and the XLXR detector on the ISQ 7610 GC-MS using OFN over a range of 0.43 ng to 43 ng on-column. The points circled in red show detector saturation.

## Spectral fidelity

Utilizing the full scan data from the ISQ 7610 GC-MS, the spectral quality of OFN was assessed at the highest concentration of 43 ng of OFN on column. On the previous electron multiplier detection system, the spectral fidelity of the acquired data is compromised due to saturation. The increased abundance of  $m/z$  222, 241, and 273 gives a relatively poor spectral match to the library spectrum. In comparison the data on the ISQ 7610 GC-MS equipped with the XLXR detector gives an almost identical ion abundances to the library spectrum. This spectral quality aids in the determination of the presence of compounds in samples and gives confidence in data analysis. The higher dynamic range also allows for better confirmation of targeted compounds that use ion ratio as confirming criteria. Figure 3 shows the comparison of spectral quality on the ISQ 7610 GC-MS on the standard detector and the XLXR detector.

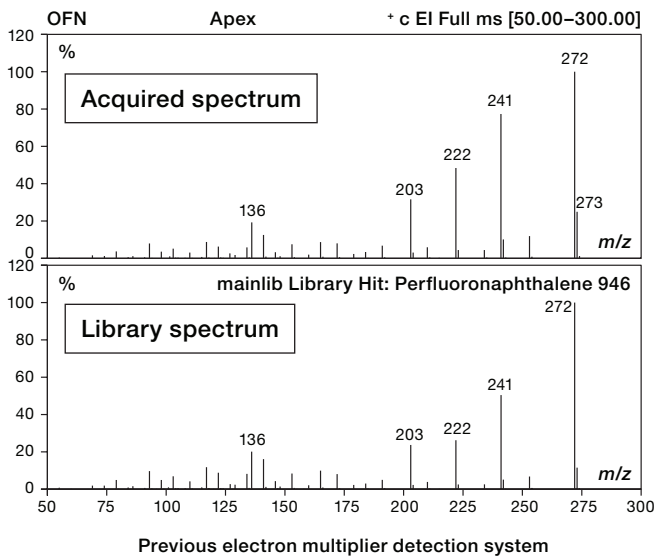


Figure 4 compares the isotopic fidelity of OFN by comparing the relative intensity of  $m/z$  273 to  $m/z$  272 at various on-column concentrations. One can clearly observe that for the previous electron multiplier detection system, the relative intensity deviates significantly from the theoretically predicted isotopic ratio of 10.82% at 20 ng and 43 ng on-column concentration, while the XLXR detector system deviates slightly, about 2.6 percentage points, at 43 ng on-column. Based on this and aforementioned results, the XLXR detector system is expected to have about two to four times higher dynamic range than the previous electron multiplier detection system.

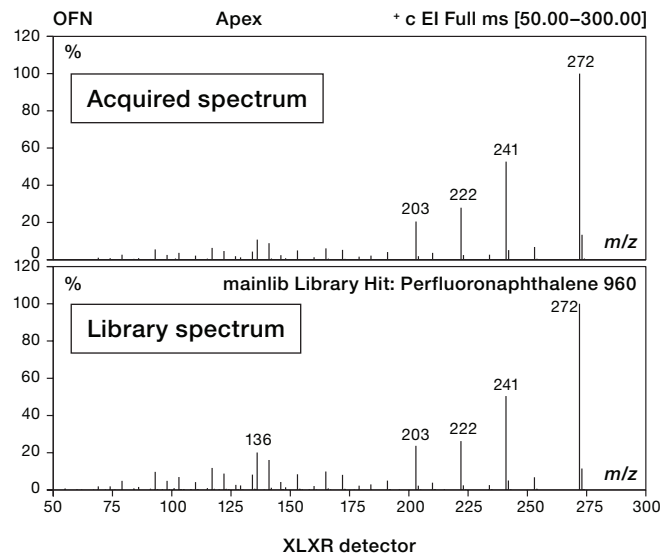


Figure 3. Mass spectral comparison on the ISQ 7610 GC-MS equipped with the previous electron multiplier detection system and the XLXR detector analyzing OFN at 43 ng

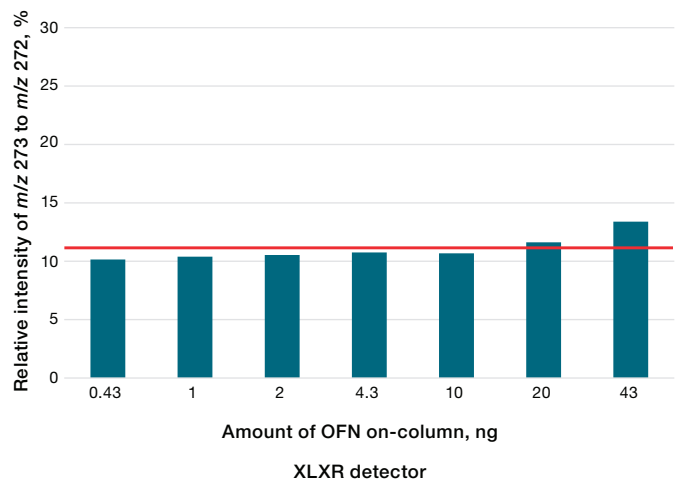
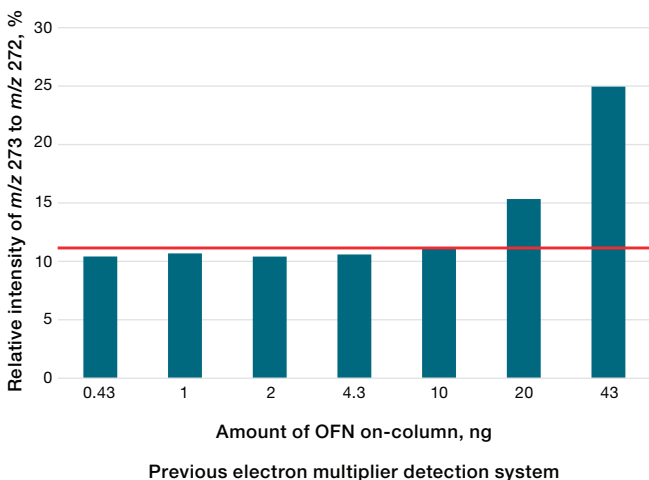


Figure 4. Chart comparing the relative intensity of  $m/z$  273 to  $m/z$  272 for various OFN on-column amounts for the previous electron multiplier detection system and the XLXR detector system. The theoretically predicted relative intensity is 10.82% and is indicated by the red horizontal line in the chart.

## Conclusion

The XLXR detector has been shown to have increased dynamic range over the previous-generation detector systems. This addresses the challenges faced by analytical testing laboratories and offers several advantages:

- Ability to consolidate methods and quantify low and high concentrations in a single method
- Reduction of the need to rerun high concentration samples to make them fit into the dynamic range of the system
- Utilization of linear regression for data analysis without the need to use quadratic fit
- Increase in spectral fidelity at high concentration, producing quality spectral matches with accurate isotopic patterns

These advantages, coupled with the extended lifetime of the XLXR detector, allow the ISQ 7610 GC-MS and TSQ 9610 GC-MS help analytical laboratories to maximize instrument utilization and increase sample throughput.

 Learn more at [thermofisher.com](https://thermofisher.com)