

Practical determination and validation of instrument detection limit for the Thermo Scientific ISQ 7000 Single Quadrupole GC-MS system with Advanced Electron Ionization source

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Keywords

Gas chromatography-mass spectrometry (GC-MS), advanced electron ionization, AEI, instrument detection limit (IDL), ISQ 7000 GC-MS system

Goal

The experiments performed in this work aimed to assess the sensitivity performance of the Thermo Scientific™ ISQ™ 7000 GC-MS system equipped with Advanced Electron Ionization (AEI) source.

Introduction

With the introduction of the Thermo Scientific™ ISQ™ 7000 GC-MS system, the highly sensitive Advanced Electron Ionization (AEI) source is available to face the most challenging applications when ultra-trace concentration levels need to be detected. The design and geometry of the AEI source is different from the traditionally designed electron ionization (EI) sources for GC-MS applications. The conventional design of the EI source, like the Thermo Scientific™ ExtractaBrite™ source, consists of a filament orthogonal to the ion beam, whereas in the AEI source the filament is placed in-line with the ion beam. The difference between the two sources is shown in Figure 1. The design of the AEI source is similar to the Very High Yield Electron Impact (VHY-EI) ion source described by Koontz.¹ Key technical elements of the AEI source are a cyclotroning magnet (placed in line with the filament) and the voltage applied to the downstream lens enabling electrostatic mirroring of the electrons. These features, combined with an increased path length for electron interactions, lead to a true increase in the yield of the ionized species. The enhanced ionization efficiency leads to a significant sensitivity increase in both SIM and full scan acquisition modes (Figure 2), opening up to highly confident analytical results even for the most challenging applications. The estimation of performance commonly used to assess the sensitivity of a GC-MS system is the instrument detection limit (IDL). This term reflects the lowest amount of an analyte that the instrument can reliably ascertain and

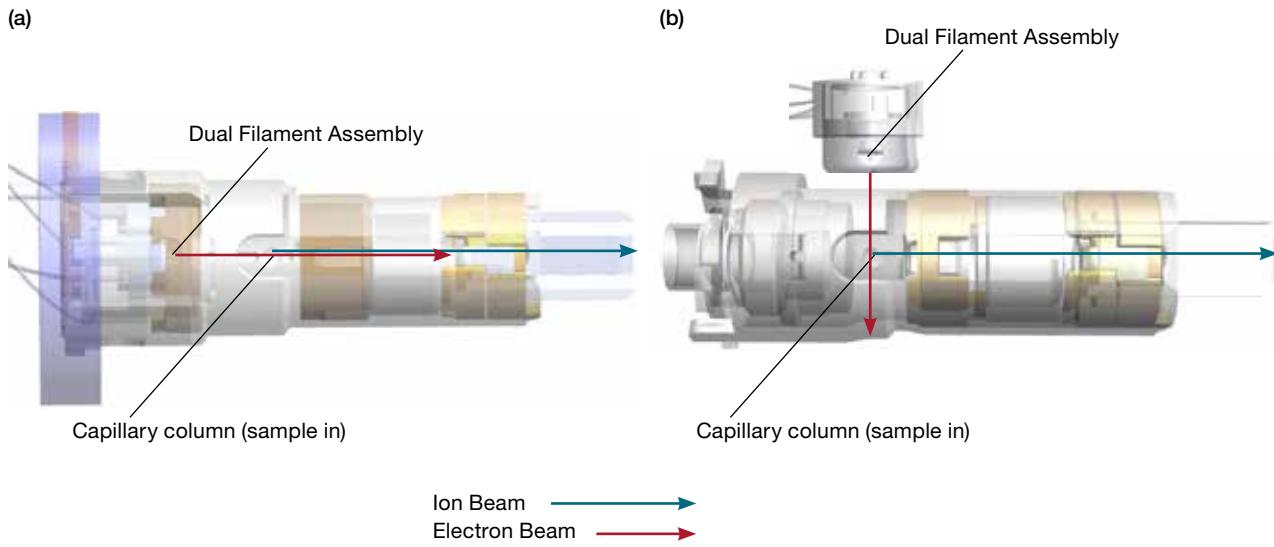


Figure 1. Comparison of the AEI source (a) with the conventional EI source (ExtractaBrite source) (b).

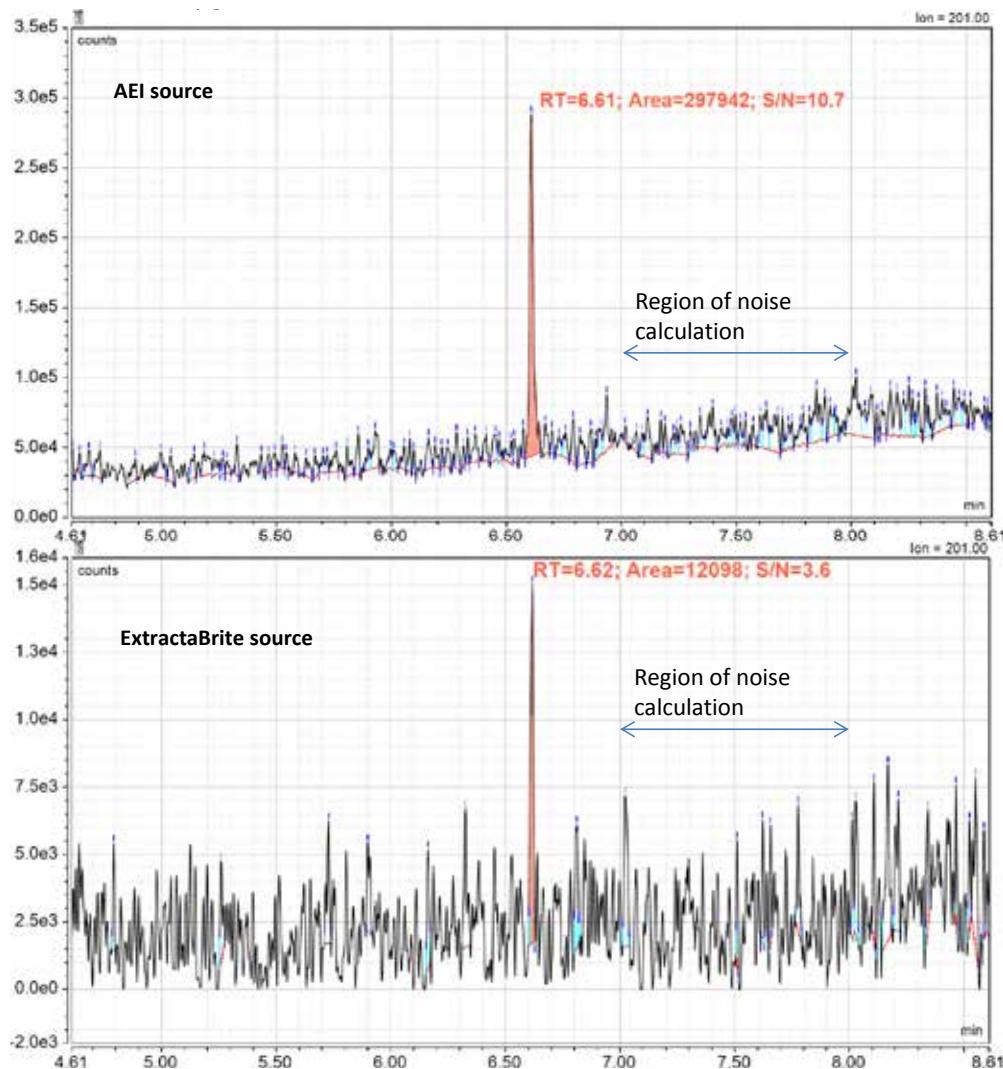


Figure 2. Comparison of extracted ion chromatogram of m/z 201 for hexachloroethane on the AEI source and the ExtractaBrite source at 2 pg on-column.

report as greater than zero.² It is the smallest analyte signal that is distinguishable from the background noise for a given analytical instrument. The IDL is calculated statistically by using precision (absolute peak area response over a statistically relevant number of repeat injections) at low analyte levels and is defined as:³

$$\text{IDL} = t \times \text{Amount} \times \% \text{RSD}$$

Where,

t = student test value at the 99% confidence interval

Amount = amount of analyte (on-column)

%RSD = percentage relative standard deviation for peak areas

S/N is another way to determine the limit of detection, but with modern instrumentation one can have low (or no) noise and this can make S/N calculations very subjective. Octafluoronaphthalene (OFN) is an inert, non-polar compound, ideal for GC-MS performance assessment. It has been utilized in this study since it is the de facto standard when comparing systems from various vendors.

Experimental

A 5 fg/ μL OFN in iso-octane solvent standard (P/N 1R76310-0105) was used for all experiments. This concentration level is close to the limit of quantification, and thus the results provide a more accurate estimation of the limit of detection.

A Thermo Scientific™ TRACE™ 1310 GC system equipped with a Thermo Scientific™ AS 1310 autosampler were connected to the ISQ 7000 mass spectrometer configured with the AEI source. Table 1 shows the autosampler, GC, and MS parameters used in this experiment. Nine instruments in various laboratories around the world were assessed using the same analytical conditions.

Table 1. Autosampler, GC, and MS parameters used for the experiments.

AS 1310 Autosampler

Syringe:	10 μL , 25 gauge, 50 mm length, cone tip (P/N 36500525)
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Injection volume:	1 μL
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Pre-injection solvent and cycles:	None
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Sample rinses:	3
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Post-injection solvent and cycles:	None
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TRACE 1310 GC system

Column:	Thermo Scientific™ TraceGOLD™ TG-SQC GC, 15 m \times 0.25 mm \times 0.25 μm
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Liner:	Splitless Liner single taper with wool, 4 mm ID, 78.5 mm length (P/N 453A0924)
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SSL mode:	Splitless
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Inlet temperature:	220 °C
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Split flow:	50 mL/min
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Splitless time:	0.5 min
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Septum purge flow:	Constant flow of 5.0 mL/min
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Carrier flow:	Constant He flow of 1.2 mL/min
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Oven program:	45 °C (0.5 min), 40 °C/min to 190 °C (0 min)
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ISQ 7000 GC-MS system

MS transfer line temperature:	250 °C
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Ion source temperature:	200 °C
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Ionization mode, Electron energy:	El, 45 eV
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Emission current:	50 μA
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Scan start:	2.4 min
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Scan mode:	SIM m/z 272
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Dwell time:	0.1 s
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Detector gain:	1.0E+06
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Results and discussion

ISQ 7000 AEI source IDL

A minimum of five injections are required in order to estimate the IDL of an instrument. In the experiments described here, 16 consecutive injections were performed. Figure 3 shows the area counts trend of all 16 injections. One can observe a stable area count over these injections. Figure 4 shows the SIM chromatogram of OFN (m/z 272) overlay of eight sequential injections resulting in IDL of 0.46 fg.

Figure 5 shows the IDLs for the nine instruments assessed. These instruments were located in various laboratories in the US, Europe and Asia, thus demonstrating IDL consistency

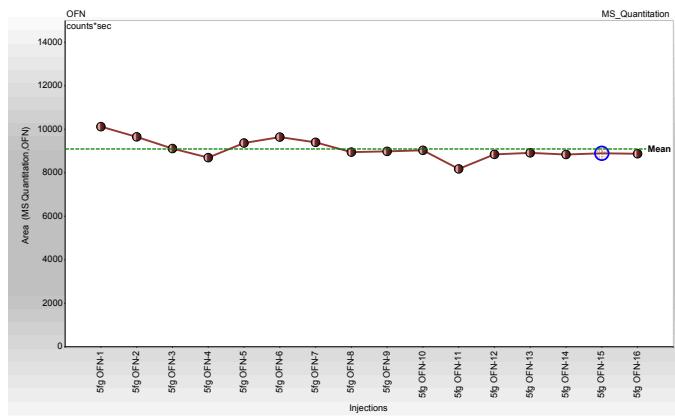


Figure 3. Absolute peak area response for $n = 16$ consecutive injections of 5 fg OFN on-column solvent standard.

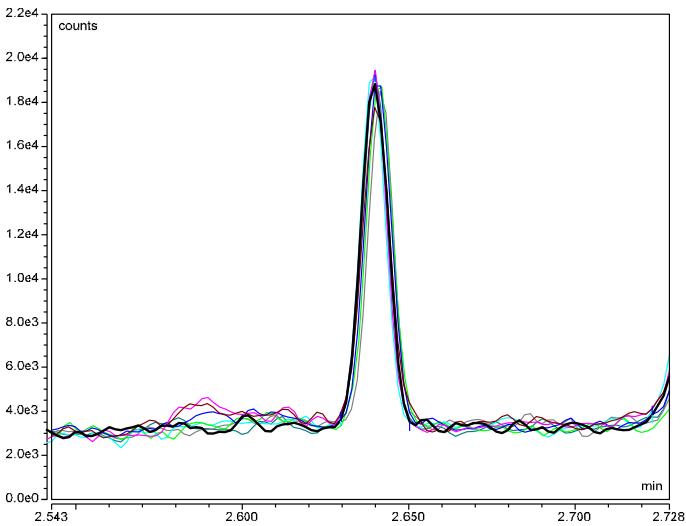


Figure 4. SIM overlay of OFN mass m/z 272 from $n = 8$ consecutive injections of 5 fg OFN on-column. Calculated IDL = 0.46 fg.

in varying environmental settings. IDLs of less than 1 fg OFN were easily achieved by all systems. The average IDL obtained from these systems was 0.64 fg with maximum and minimum IDLs of 0.87 fg and 0.46 fg, respectively.

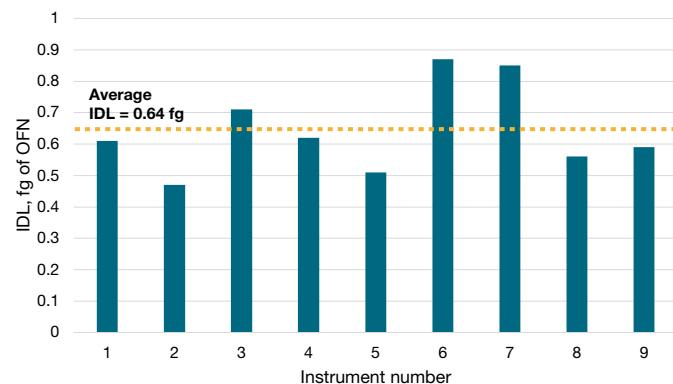


Figure 5. Calculated IDLs from $n = 9$ individual ISQ 7000 instruments configured with the AEI source.

Conclusions

The ISQ 7000 GC-MS system configured with the AEI source delivers detection limits consistently <1 fg as determined by repeated injections of 5 fg OFN on multiple systems.

The level of IDLs achieved with the AEI source is groundbreaking for a single quadrupole GC-MS system, and opens up new possibilities for applications, especially for those that require screening for analytes present at ultra-trace levels.

The enhanced sensitivity demonstrated is critical when addressing the most demanding analytical requirements, in both routine and research environments, making the ISQ 7000 GC-MS system equipped with the AEI source the ideal solution for laboratories using a quadrupole GC-MS system for applications that require very low levels of detection.

References

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2. *Analytical Detection and Quantification Limits: Survey of State and Federal Approaches*; Publication No. 4721; American Petroleum Institute, June 2002.
3. Technical Note 10494: Practical Determination and Validation of Instrument Detection Limit of Thermo Scientific™ ISQ™ 7000 Single Quadrupole GC-MS with ExtractaBrite EI ionization source; Thermo Fisher Scientific, 2018.

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