Dioxin quantification using TargetLynx™

Over the past decade, regulatory environmental agencies and industry have worked together to dramatically reduce dioxin emissions. The detection and quantification of dioxins is a particularly demanding analysis due to the low level of regulatory exposure limits and the variety of complex sample matrices encountered.

As standard, TargetLynx is the integral part of the core AutoSpec Premier package. TargetLynx supports all of the major dioxin and furan protocols, including US, Canadian (EPA 1997B), European (CEN 1948), US EPA Method 1613 and 8290, method 23 and IN831, along with user-defined methods. EPA Method 1668 for the quantification of PCBs (Polychlorinated Biphenyls) is also supported.

Dioxin quantification is simplified by the provision of acquisition methods, including GC methods, for standard protocols such as EPA Method 1613. The results from TargetLynx data processing are displayed in the TargetLynx browser to allow for convenient data review and is enhanced with several dioxin-dedicated features. Features include Congener Selection from a pull down list to quickly view congener-specific quantification data and similarly sample select to locate samples from large batches with ease.

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Dioxin quantification using targetLynx
Worldwide services and support
Waters Connections® Programs provide the solutions you need to maintain system across your Waters systems.

• Analytical Instrumentation and Software Services include Total Assurance Plans and Workarounds that extend and enhance the original warranty you receive when you buy a Waters product. These plans maximize the level of insurance investment and deliver the value you need to avoid costly and time-consuming system downtime.

• Connections Compliance Services provide you with timely and efficient solutions for your regulatory compliance challenges. You can use Waters Compliance Services to verify proper equipment operation for CGMP/GLP compliance, significantly reducing operating costs.

• Connections University is the center of our Educational Services, providing extensive HPLC and MS training and education at your site, at our corporate headquarters or at our local offices around the world.

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Typical dioxin chromatogram (US EPA 1613) as viewed in the TargetLynx browser.

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Sales Offices:

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AutoSpec Premier

The AutoSpec Premier™ is the latest development in magnetic sector instrumentation from Waters. It incorporates the Waters® Micromass® unique tri-sector (EBE), double-focusing geometry with the wide gap magvet of the proven Ultima NT system, providing a unique combination of high sensitivity, high resolution and low background noise. The AutoSpec Premier is the optional choice for ultra low-level trace detection applications, such as monitoring for dioxins and related compounds like Polychlorinated Biphenyls (PCB's) and Polybrominated Diphenyl Ethers (PBDE's), or drugs of abuse, by high resolution/ selected ion recording (HR/SIR) gas chromatography/mass spectrometry (GC/MS).

Looking for sensitivity, selectivity and dynamic range? Are you?

• Analyzing dioxins and related compounds?
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**EEB Geometry**

The Autospec Premier incorporates unique twin-sector (EBE) geometry ion optics, designed to give maximum sensitivity in all modes at both high and low resolution. The EEB design has one electric sector positioned before and one after the magnetic sector. The first electric sector has demagnifying optics to give a high dispersion to magnification ratio, allowing high resolutions to be obtained with a wider source slit and higher sensitivity. The mass resolution is continuously variable to 80,000 (10% valley definition). The second electric sector reduces background noise, improves abundance sensitivity and automatically rejects metastable ion interferences.

**Premier Magnet**

The Autospec Premier is fitted with a lineially laminated, gradient orientated, rolled steel sheet with high permeability and low hysteresis. The extraneous gap magnet design allows use of a wider source slit, giving higher transmission and reducing susceptibility to contamination. The patented post-acceleration photomultiplier detection system enables detection of positive or negative ions without switchingodynamic voltages. This detector has a single ion detector capability, and the long time stability of the photomultiplier ensures excellent and reproducible long-term performance.

**Ion Optics and Pumping**

The ion optics are supported by a pneumatic suspension beam to isolate the analyzer from vibrations and maximize instrument performance. The instrument is pumped with low maintenance, high capacity diffusion pumps for optional turbomolecular pump, with the analyzer doubly differentially pumped to ensure high abundance sensitivity. The vacuum system pumping, ionization and protection is fully automated.

**Sources and Options**

Plug in source are provided for Electrospray (ES) and Chemical Ionization (CI), allowing rapid and convenient source change without venting the instrument. GC is most often the sample introduction mode of choice, but it is not necessary to physically remove the GC to utilize the solids probe or Desorption Chemical Ionization probe (DCI).

**Sensitivity**

To achieve the highest sensitivity, HR/MS acquisition is used. In the mode, or 10,000 resolution (10% valley definition), an ionization of 100 Hz of 2,3,7,8-TCDD will give a signal-to-noise ratio of 221:10. Of a 125:1 or raw data line smoothing applied. Noise, in this case, is taken to be equivalent to 2 standard deviations (+/- 2). It is important to note that some other protocols for data analysis, quite similar to those with the noise defined as 2 standard deviations (+/- 2). For this definition of noise, the equivalent noise value is > 250:1.

**Stability**

The Autospec Premier gives expert stability of response over an extended period of analysis. This reduces the frequency of tuning and calibration necessary and increases productivity by reducing instrument downtime for routine maintenance.

**Mass accuracy**

Masses can be measured accurately to +/- 2 parts in 10^5.

**Dynamic range**

The Autospec Premier offers >5 orders of linear dynamic range enabling quantification over a wide concentration range and analysis of mixture containing components at both high and low concentrations in one analytical run.

**Acquisition Modes**

The following acquisition modes are supported:

- Multiple Reaction Monitoring (MRM)
- MIKES
- B/E Product Ion Scanning
- B/E Precursor Ion Scanning
- Constant Neutral Loss Scanning
- MRMES
- Multiple Reaction Monitoring (MRM)

**AutoTune**

The optimization of instrument tuning is made rapid and easy with the AutoTune facility. A sophisticated tuning algorithm combined with automated ion optics gives optimum tuning in a matter of minutes. The duration of the AutoTune routine is minimized by ‘fast’ slits, which have a response time of < 0.2 seconds regardless of the positional change, this allows for extremely rapid manipulation of the source and collector slits for optimum resolution and transmission.

**Automated resolution verification**

Regulatory requirements for some analytes require instrument resolution to be checked at regular intervals. Some US EPA Methods, for example, specify resolution checks every 24 hours during analysis. Mass selective software can automatically check instrument resolution after any sample in the Sample List, and will verify the resolution of all reference peaks in the mass window of the experiment. A history of these results can be provided, showing the peaks checked and the resolution measured for each. If the resolution of any of the reference peaks is outside user-specified tolerances, the sample list will be halted, preventing the loss of valuable samples.

**Unmatched performance**

The Autospec Premier gives unmatched performance in a compact footprint—the ultimate in high resolution MS. With the Autospec Premier, you will benefit from:

- Fast control with Masslynx™ Software in a Microsoft® Windows environment
- Target™ Application Manager included as standard
- Unique trigger (ISE) ion optics
- Extra wide gap magnesium for higher transmission
- Ultra high resolution > 80,000
- 20° flux dynamic range
- Constant gain, high performance photomultiplier detection system
- Flag B and C1 options for rapid changer
- Dual GC Interface, column change without venting instrument
- Low maintenance high capacity diffusion pumps as standard. Optional turbomolecular pumping configurations.
- Integrated GC (highest 605M) and autosampler (Autospec 376S, CTCPal and CombiPal)
- AutoTune and automated resolution checking
- Optional novel split detector for the abatement of column interferences.
AutoSpec Premier New Technology

The AutoSpec Premier has a dual GC interface to allow the use of two GC injectors with two GC columns installed for alternate or dual simultaneous injection. The pumping configuration and capacity allows change of the GC column without venting the instrument, as well as giving an improved thermal variation profile. The AutoSpec Premier gives the user the ability to immediately switch between course and fine control to satisfy calibration and sample analysis requirements.

Stability

The AutoSpec Premier gives the user the ability to immediately switch between course and fine control to satisfy calibration and sample analysis requirements. Dual GC interface with low maintenance, high capacity diffusion pumps (or optional turbomolecular pumps), with the analyzer double differentially pumped to ensure high abundance sensitivity.

 autoTune

The optimization of instrument tuning is made rapid and easy with the AutoTune facility. A sophisticated tuning algorithm combined with automated ion optics gives optimum tuning in a matter of minutes. The duration of the autoTune routine is minimized by fast slits, which have a response time of <0.2 seconds regardless of the positional change; this allows for extremely rapid manipulation of the source and collector slits for optimum resolution and sensitivity. A sophisticated tuning algorithm combined with automated ion optics gives optimum tuning in a matter of minutes. The duration of the autoTune routine is minimized by fast slits, which have a response time of <0.2 seconds regardless of the positional change; this allows for extremely rapid manipulation of the source and collector slits for optimum resolution and sensitivity.

AutoTune and automated resolution checking

Automated resolution verification

Regulatory requirements for some analyses require instrument resolution to be checked at regular intervals. Some US EPA Methods, for example, specify resolution checks every 12 hours during analysis. MassLynx software can automatically check instrument resolution with any sample in the Sample list, and will verify the resolution of all reference peaks in the mass window of the report. A landscape of these results can be provided, showing the peaks checked and the resolution measured for each. If the resolution of any of the reference peaks is outside the specified tolerance, the sample list will be halted, providing the loss of volatile samples.

Sensitivity

To achieve the highest sensitivity, HR/SIR acquisition is used. In this mode, at 20,000 resolution (10% valley definition), an ion of 100 Da of 2,3,7,8-TCDD will give a signal-tonoise ratio of 221:1050 Da of 1.25:1 or raw data [smoothed/normalized]. Notice, in this case, is taken to be equivalent to 1 standard deviation (7/26). It is important to note that some other protocols for dioxin analysis quite significantly with the noise defined as 2 standard deviations (7/26). For this definition of noise, the equivalent noise value is < 300.

AutoSpec Premier’s patented double-focusing, tri-sector geometry.

EBe Geometry

The AutoSpec Premier incorporates unique trapezoidal EBe geometry ion optics, designed to give maximum sensitivity in all modes at both high and low resolution.

The EBe design has one electric sector positioned before and one after the magnetic sector. The first electric sector has demagnifying optics to give a high dispersion to magnification ratio, allowing high resolutions to be obtained with a wider source slit and higher sensitivity. The second electric sector reduces background noise, improves abundance sensitivity and automatically rejects metastable ion interferences.

Options and Pumping

The ion optics is supported by a pneumatic suspension beam to isolate the analyser from vibration and maintain instrument performance. The instrument is pumped with low maintenance, high capacity diffusion pumps for optimal turbomolecular pump, with the analyzer double differentially pumped to ensure high abundance sensitivity. The vacuum system pumping, ionizing and protection is fully automated.

Sources and Options

Mass ions are produced for Electron impact (EI) and Chemical ionization (CI), allowing rapid and convenient source change without venting the instrument. GC is most often the sample introduction mode of choice, but it is not necessary to physically remove the GC to utilize the solids probe or Desorption Chemical Ionization probe (DCI).
The AutoSpec Premier has a dual GC interface to allow the use of two GC injectors with two GC columns installed for alternate or dual simultaneous injection. The pumping configuration and capacity allows change of the GC column without venting the instrument, reducing instrument downtime and increasing productivity. The dual GC interface of the AutoSpec Premier enables direct line-of-sight GC column installation into the source region, as well as giving an improved thermal variation profile.

The AutoSpec Premier incorporates unique ion optics, designed to give maximum sensitivity in all modes at both high and low resolution. The EBE design has one electric sector positioned before and one after the magnetic sector. The first electric sector has demagnifying ion optics and pumping. The second electric sector reduces background noise, improves abundance sensitivity and automatically rejects metastable ion interferences.

The extra-wide gap magnet design allows use of a wider source slit and increases sensitivity. The detector has single ion detection capability; and the long-term stability of the photomultiplier gain is improved by the patented post-acceleration photomultiplier detection system. This reduces the frequency of tuning and calibration necessary by reducing instrument downtime.

Sources and Options
Plug-in ion sources are provided for Electron Impact (EI) and Chemical Ionization (CI), allowing rapid and convenient source change without venting the instrument. The instrument is pumped with low maintenance, high capacity diffusion pumps (or optional turbomolecular pumps), with the analyzer double differentially pumped to ensure high abundance sensitivity. The vacuum system pumping, insulating and protection is fully automatic.

Mass accuracy
Masses can be measured accurately to < 2 mDa or < 5 ppm RMS.

Dynamic range
The AutoSpec Premier offers > 10 orders of linear dynamic range enabling quantification over a wide concentration range and analysis of mixtures containing components at both high and low concentrations in one analytical run.

Sensitivity
To achieve the highest sensitivity, HR/SIR acquisition is used. In this mode, an injection of 10,000 resolution (10% valley definition) on an injector of 100 µg of 2,3,7,8-TCDD will give a signal-to-noise ratio of > 125:1 on raw data (base smoothed applied). Notice, in this case, is taken to be equivalent to 3 standard deviations. It is important to note that some other protocols for dioxin analysis require the signal-to-noise ratio of > 7. This definition of noise, the equivalent noise level is > 250.1.

Unmatched HR/SIR sensitivity on raw, unsmoothed data.

Acquisition Modes
The following acquisition modes are supported:
• Multiple Reaction Monitoring (MRM)
• Constant Neutral Loss Scanning
• Voltage Scanning
• Voltage Selected Ion Recording (VSIR)
• MIKES
• Multiple Reaction Monitoring (WMW)

Unmatched performances
The AutoSpec Premier gives unmatched performance in a compact instrument at a high-resolution MS. With the AutoSpec Premier, you will benefit from:
• Full control with MassLynx™ Software in a Microsoft Windows environment
• Targetmax™ Application Manager included as standard
• Unique triple quadrupole (TQ) ion optics
• Extra wide gap magnet for higher transmission
• Ultra high resolution > 80,000
• > 10² linear dynamic range
• Constant gain, long-life photomultiplier detection system
• Flag E and CI sources for rapid changers
• Dual GC Interface, column change without venting instrument
• Linearity/resolution high capacity diffusion pumps as standard. Optional turbo/molecular pumping configurations.
• Integrated GC (highest ESI) and autosampler (Aplio 769, CT605GC and Conodona)
• AutoTune and automated resolution checking
• Optional novel split detector for the elucidation of isotopic interferences

The optimization of instrument tuning is made rapid and easy with the AutoTune facility. A sophisticated tuning algorithm combined with automated ion optics gives optimum tuning in a matter of minutes. The duration of the AutoTune routine is minimized by ‘fast’ slits, which have a response time of <0.2 seconds regardless of the positional change, this allows for extremely rapid manipulation of the source and collector slits for optimum resolution and transmission.

The AutoSpec Premier New Technology
The AutoSpec Premier has a dual GC interface to allow the use of two GC injectors with two GC columns installed for alternate or dual simultaneous injection. This pumping configuration and capacity allows change of the GC columns without venting the instrument, reducing instrument downtime and increasing productivity. The dual GC interface of the AutoSpec Premier enables direct line-of-sight GC column installation into the source region, as well as giving an improved thermal variation profile.

Electronic development has enhanced the precision with which the accelerating voltage can be set during voltage selected ion recording (VSIR). This ultimately improves mass precision, which can lead to superior data quality, especially in circumstances where a background signal is close to the desired mass.

A cold reference probe is included with the system enabling liquid introduction at room temperature. This gives superior lock mass recording (VSIR). This ultimately improves mass precision, which can lead to superior data quality, especially in circumstances where a background signal is close to the desired mass.

Stability
The AutoSpec Premier gives expert stability of response over an extended period of time. This reduces the frequency of tuning and calibration necessary by reducing instrument downtime for routine maintenance.

The instrument is pumped with low maintenance, high capacity diffusion pumps for optional turbomolecular pump, with the analyzer double differentially pumped to ensure high abundance sensitivity. The vacuum system pumping, insulating and protection is fully automatic.
Dioxin quantification using TargetLynx™

Over the past decade, environmental regulatory agencies and industry have worked together to dramatically reduce dioxin emissions. The detection and quantification of dioxins is a particularly demanding analysis due to the low level of regulatory exposure limits and the variety of complex sample matrices encountered.

As standard, TargetLynx is an integral part of the core AutoSpec Premier package. TargetLynx supports all of the major dioxin and furan protocols, including Asian, Canadian (1/80), European (1/40), US EPA Method 1613 and 8290, method 23 and NCEA, along with user-defined methods. EPA Method 1668 for the quantification of PCBs (Polychlorinated Biphenyls) is also supported.

Dioxin quantification is simplified by the provision of acquisition methods, including GC methods, for standard protocols such as EPA Method 1613. The results from TargetLynx data processing are displayed in the TargetLynx browser to allow for convenient review of data.

Typical dioxin chromatogram (US EPA 1613) as viewed in the TargetLynx browser.

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• Representatives of Waters Global Customer Assurance Organization, trained and certified in all Waters products and current in LC and MS applications, are available to verify proper equipment operation for CGMP/GLP compliance, significantly reducing operating costs. The AutoSpec Premier is the optimal choice for ultra low-level trace detection applications, such as monitoring for dioxins and related compounds like Polychlorinated Biphenyls (PCB's) and Polybrominated Diphenyl Ethers (PBDE's), or drugs of abuse, by high resolution/selected ion recording (HR/SIR) gas chromatography/mass spectrometry (GC/MS). It incorporates the Waters® AutoSpec Premier, the latest development in magnetic sector instrumentation from Waters. The AutoSpec Premier is the latest development in magnetic sector instrumentation from Waters. It incorporates the Waters® Micro Mass®, unique tri-sector (EBE), double-focusing geometry with the wide gap magnet of the proven Ultima NT system, providing a unique combination of high sensitivity, high resolution and low background noise. The AutoSpec Premier is the optimal choice for ultra low-level trace detection applications, such as monitoring for dioxins and related compounds like Polychlorinated Biphenyls (PCB's) and Polybrominated Diphenyl Ethers (PBDE's), or drugs of abuse, by high resolution/selected ion recording (HR/SIR) gas chromatography/mass spectrometry (GC/MS).
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The result file is complete with an audit log showing all processes, both manual and automatic, performed on a batch of samples or standards. Reports are customizable for printing and the data can also be exported as a text file or in XML format (for RTMQC methods. The result file is complete with an audit log showing all processes, both manual and automatic, performed on a batch of samples or standards. Reports are customizable for printing and the data can also be exported as a text file or in XML format (for RTMQC). The TargetLynx browser.

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