thermo scientific

Simplify

the complexity of dioxin analysis

Thermo Scientific Dioxin Analyzer

ThermoFisher SCIENTIFIC

Dioxin Analyzer: compliance, productivity and robustness

The Thermo Scientific™ Dioxin Analyzer is an integrated, sample-to-result GC-MS/MS based, easy to implement, analytical workflow developed to deliver robust and sensitive quantitation of polychlorinated dibenzo-p-dioxins (PCDD), polychlorinated dibenzo furans (PCDF) and dioxin-like polychlorinated biphenyls (dl-PCBs), in food and feed samples in compliance with the latest EU regulations (EU Regulation 664/2017).

Recent advances in GC-MS/MS technology have enabled lower detection limits and improved automated selected reaction monitoring (SRM) performance. This, in turn, has led the European Commission to update regulations to permit the use of GC-MS/MS, in addition to magnetic sector technology, as a confirmatory method for enforcing maximum levels (ML) and action levels (AL) for dioxins/furans and dl-PCBs in foodstuffs and animal feeds. The Dioxin Analyzer not only takes advantage of the new GC-MS/MS technical advancements, but significantly improves the ease of use and accelerates the implementation in routine laboratories. The Dioxin Analyzer includes a comprehensive user deployment guide, quality check standards, a pre-loaded acquisition method, as well as data processing and reporting templates—all in a single analytical package from a single vendor.

The immediate benefits for the laboratories are:

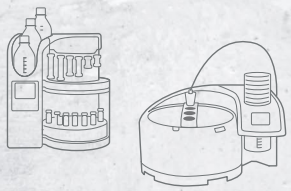
- **Compliance** with EU requirements for dioxin/furan and dl-PCBs in food and feed samples and ultimate confidence in results.
- **Productivity** ensured by out-of-box implementation and operational simplicity, supported by a comprehensive suite of Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) tools, tailored to dioxin analysis. These are aimed at reducing training needs and increasing productivity immediately after installation.
- **Robustness** of the instrumentation for a large variety of sample types and extended uptimes (less unscheduled downtime) producing consistent high quality results.

DIOXIN ANALYZER

SAMPLE PREPARATION

Tools to accelerate sample preparation

Automated micro-sampling and injection of small volumes of extracts



SEPARATION

TG-Dioxin column with unique selectivity for toxic dioxin and furan congeners

Separation of critical pairs

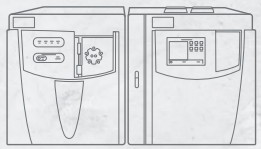
Quality check standards included to verify performance criteria

DETECTION

TSQ 9000 with AEI source

Pre-optimized SRM transitions

Segmented or time acquisition method



DATA MANAGEMENT

Compliant-ready data processing

Automated calculation of TEQ results built into Chromeleon software

Reporting templates with isotope standards recovery, ion ratio, LOQ flagging and other extensive functionality

TSQ 9000 triple quadrupole GC-MS/MS system coupled with Thermo Scientific™ TRACE™ 1310 Gas Chromatograph

Full compliance with EU criteria at the lowest LOQ level

Gain cost-effective high sample-throughput for monitoring maximum levels and action levels of dioxins/furans and dl-PCBs in foodstuffs and animal feeds. To support the validity of data reported within the framework of official controls, testing laboratories are required for each analytical sequence, to demonstrate compliance with method performance criteria, ranging from sensitivity to accuracy. In particular, the limit of quantitation (LOQ) for each of the most toxic congeners needs to be confirmed by checking the consistency of the response at the lowest concentration level of a calibration curve. Leveraging benchmark sensitivity of the Thermo Scientific™ TSQ™ 9000 triple quadrupole GC-MS/MS system with the Advanced Electron Ionization (AEI) source, the Dioxin Analyzer provides the confidence to meet all EU compliance criteria at the lowest LOQ levels.

PERFORMANCE CRITERIA

Separation of isomers shall be <25% peak to peak between **1,2,3,4,7,8-HxCDF** and **1,2,3,6,7,8-HxCDF**

Resolution for each quadrupole to be set equal to or better than unit mass resolution (sufficient resolution to separate two peaks one mass unit apart)

Maximum permitted tolerance of relative ion intensities of ±15 % for selected transition product ions in comparison to theoretical or measured values

Response Factor deviation of the lowest concentration shall be <30% from average value throughout the sequence, in order to use this point to calculate LOQ

Limit of Quantification (LOQ) shall be 20% of the maximum level in the corresponding food and feed stuffs

EU quality performance criteria for routine use of GC-MS/MS for confirmational analysis

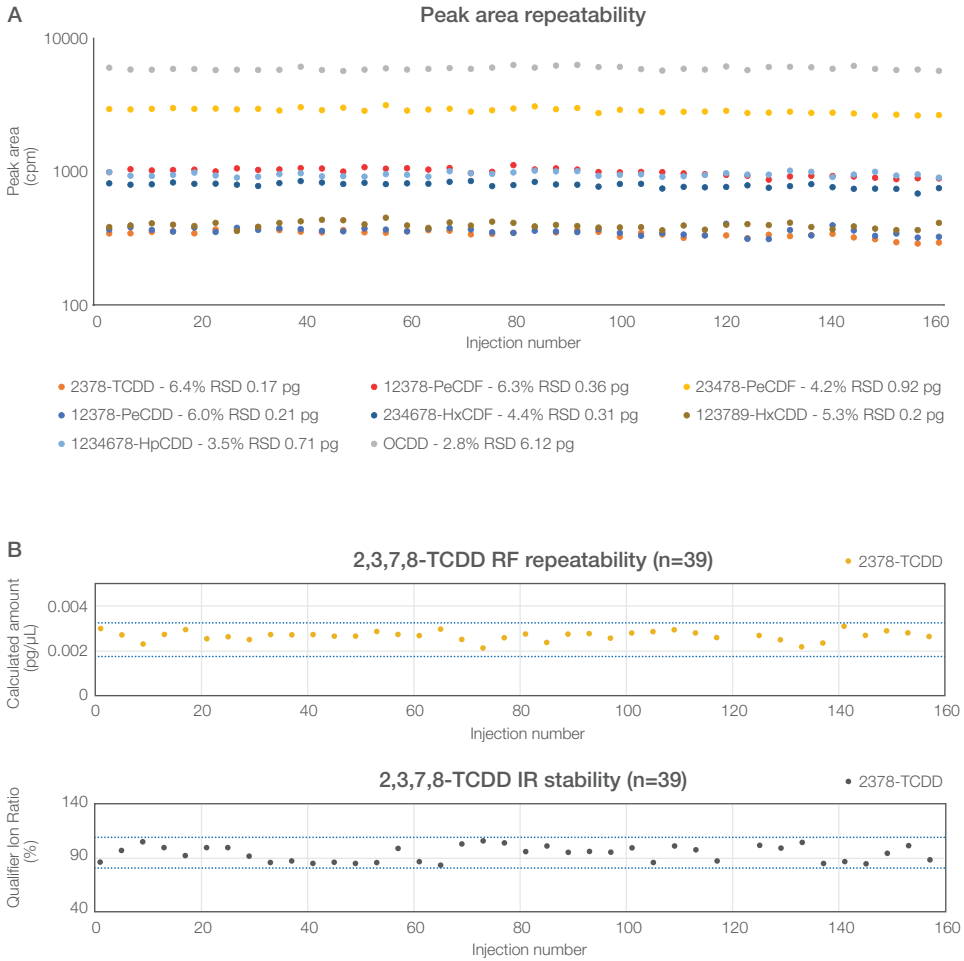
Enhanced productivity from day one

All instrument components, capillary GC column, system suitability check standard solutions, software and comprehensive user guidelines are included for seamless implementation and enablement of ongoing optimum performance immediately after installation. Complex data calculation and automatic generation of results is made easier through the Chromeleon CDS productivity tools. This suite of software tools supports and facilitates all steps, from creating the analytical sequence with all quality checks and performance criteria, to the interactive monitoring of the analysis progress, automatic calculation of concentrations based on isotope dilution calculations and reporting the Toxic Equivalent (TEQs) results in compliance with the EU Regulation. Thus, giving back to the user, valuable time to be invested in other more profitable activities.

Robust workflow for unstoppable uptime

Take advantage of the robust, productive and reliable quantitation, in compliance with EU method performance criteria, in every analysis sequence. The new ultra-robust AEI source design of TSQ 9000 GC-MS/MS system provides extremely high tolerance to the matrix. This enables the user to set up complete sample sequences with hundreds of injections—alternating samples with blanks and multiple level standard solutions—with the confidence to deliver consistent and trusted results. As an example, Figures A and B show the excellent robustness of an injection sequence over a period of two weeks, with no maintenance such as: source cleaning, liner replacement, tuning or analytical column trimming, performed during the sequence despite the high number of injections and matrix complexity.

Consistent results over continuous analysis



(A) Absolute peak area repeatability over two weeks of analysis, for selected PCDD/F congeners in pooled matrix sample. Relative standard deviations and amounts on-column (pg) are annotated for each selected congener. (B) LOQ Response Factor (RF) deviation (upper plot, calculated as deviation from target amount) and Ion Ratio, IR, (lower plot) for the 10 fg on-column 2,3,7,8-TCDD congener (2.5 fg/µL, 4 µL injection).

Increase your laboratory productivity with the Dioxin Analyzer

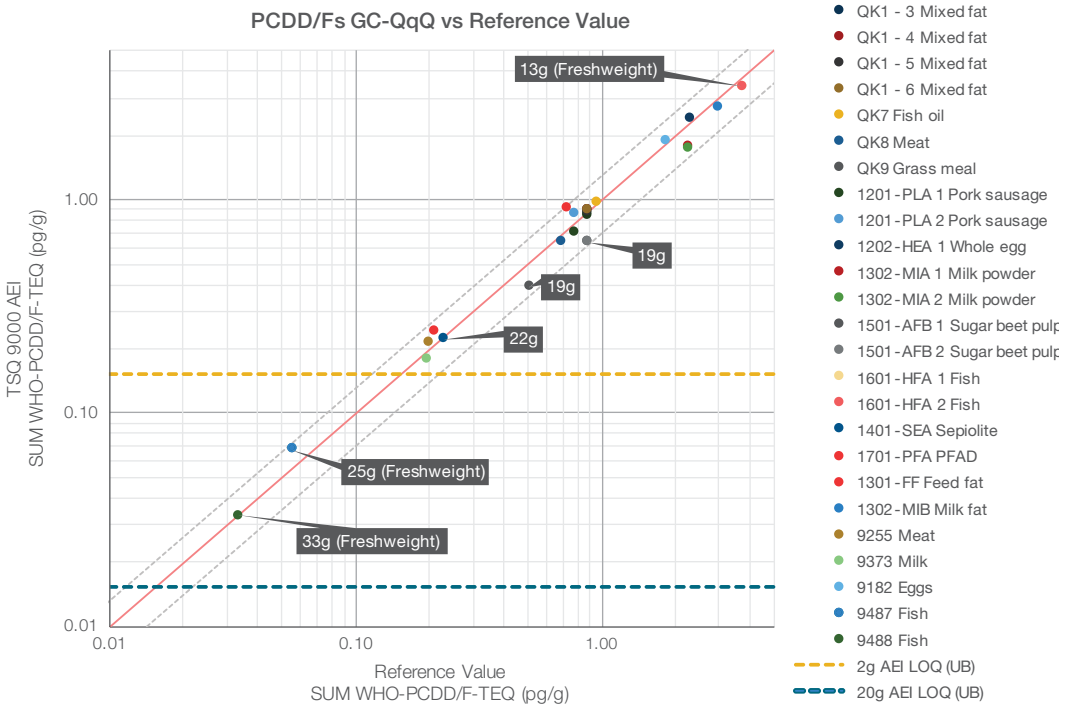
The Dioxin Analyzer addresses the high cost and complexity faced by scientists testing food and animal feeds for low levels of dioxins/furans and dl-PCBs. This analyzer includes all components needed—consumables, hardware, software and built-in instrument and data processing methods. All components are pre-configured and tested from your single trusted supplier, Thermo Fisher Scientific.

Lower cost per sample

The TSQ 9000 triple quadrupole GC-MS/MS system, with fully automated SRM transitions optimization, allows the method to be easily implemented into any laboratory. The system delivers reproducible data at high sensitivity to meet all compliance criteria at the lowest LOQ level. This level of sensitivity enables users to lower sample weights, reducing both the cost of sample preparation and system maintenance.

Comparison of results obtained using the GC-MS/MS and the EURL reference values. The center red line represents 100% agreement with the reference value and the upper and lower greyed lines represent a ±30% deviation from this value. Unless specified, sample intake weight was 2 g; amount scales are logarithmic to aid comparison (see Application note 10703 for further data).

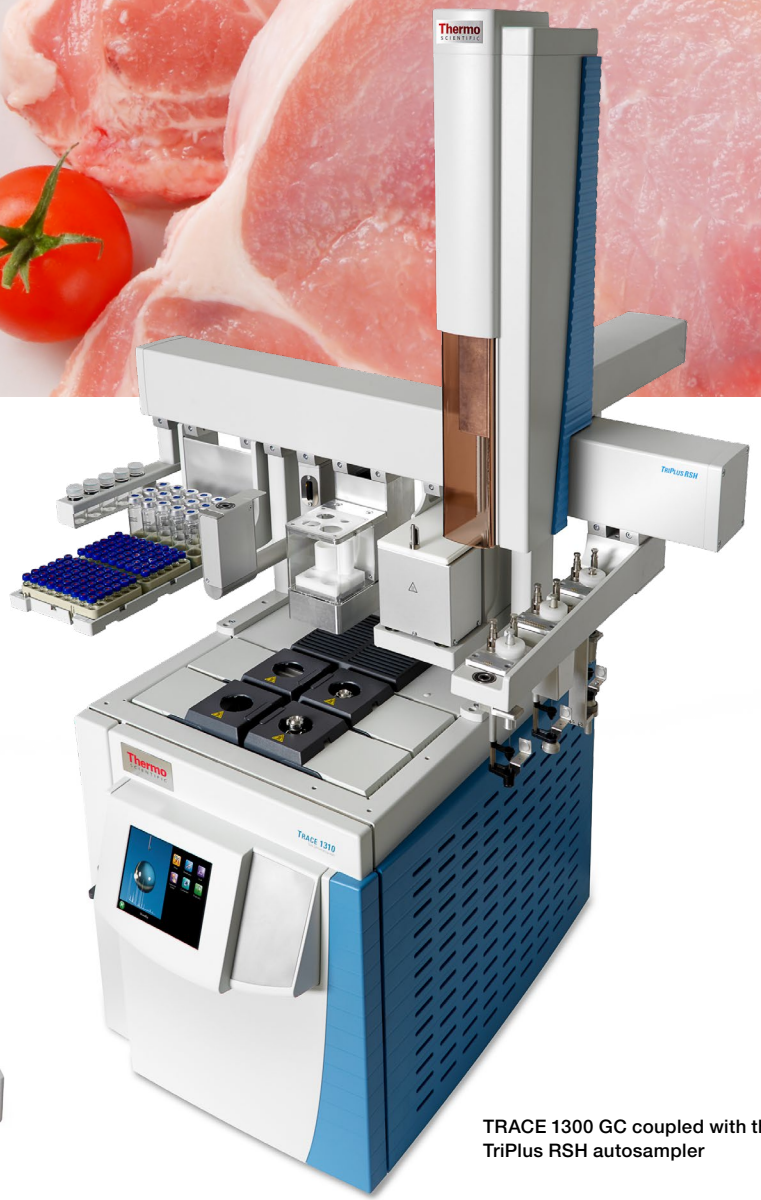
Excellent match with EURL results on supplied samples



Quality results with a large variety of sample types and lower sample weights

Impeccable results start with correct GC injection and chromatographic separation. High quality PTV and SSL injectors, available with the TRACE 1300 GC Series, ensure a high quality start to the chromatographic separation process even with splitless injection of several microliters. Best-in-class serviceability and ease of operational use add unmatched high lab productivity to the delivery of quality results. The Thermo Scientific™ TriPlus™ RSH Autosampler offers the unique capability for accurate micro-sampling and injection of extracts (few hundred of microliters) resulting from extraction, clean-up and fractionation, thus maximizing results with your most precious samples.

Accurate micro-sampling and injection



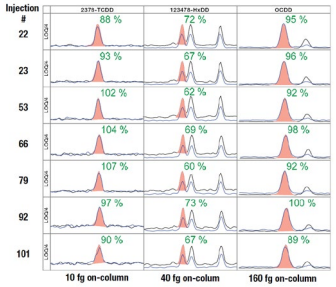
TRACE 1300 GC coupled with the TriPlus RSH autosampler

Rapid and consistent implementation of the analyzer

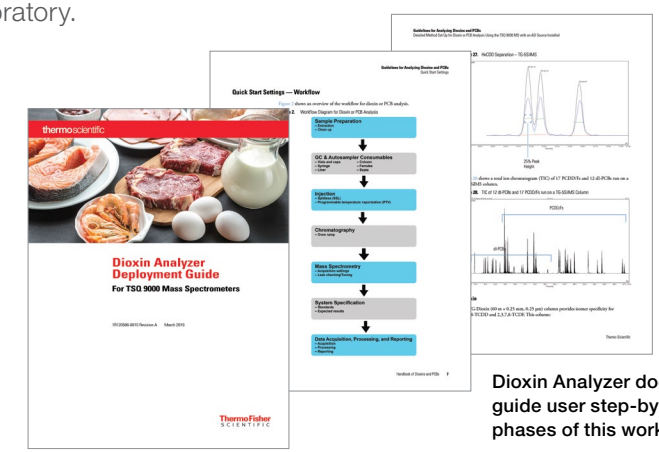
Included with the Dioxin Analyzer are documents and quality check standards to assist the rapid and consistent implementation of this workflow in the lab. The document guides the user step by step through all phases to implement a pre-configured method or to customize a new user-defined method.

The deployment guide is based on an extended validation undertaken to test the suitability of GC-MS/MS, especially for the long-term routine analysis of hundreds of samples. Successful validation of method performance criteria (LOQ, precision, accuracy and calibration) was carried out using three different TSQ 9000 AEI GC-MS systems AEI source, in three geo-locations, providing perfectly aligned results. This validation also demonstrated that implementation of the method could be deployed with ease in any laboratory across the world—giving piece of mind to analysts wanting to quickly integrate the Dioxin Analyzer into their laboratory.

LOQ repeatability during the UK-based validation



Overlaid extracted ion chromatograms are displayed (quantification and confirmation ions) for selected TCDD, HxCDD and OCDD congeners. IRs (as displayed in green) and RFs were within the allowable tolerances.



Dioxin Analyzer documentation to guide user step-by-step though all phases of this workflow

Excellent chromatographic separation for dioxin analysis

The proprietary phase of the Thermo Scientific™ TraceGOLD™ TG-Dioxin Capillary GC Column provides excellent separation of toxic dioxin, furan and PCB congeners within 45 minutes. This is critical to eliminate any quantitation errors resulting from co-elution of critical pairs of congeners. The TG-Dioxin column also facilitates compliance with all required chromatographic quality performance criteria.

Tools to accelerate extraction and concentration

Two optional tools are available to accelerate the sample extraction and subsequent concentration of extracts.

The Thermo Scientific™ Dionex™ ASE™ 350 Accelerated Solvent Extractor

- Provides walk-away automation
- Fast extractions
- Reduced solvent usage

The Thermo Scientific™ Rocket™ Evaporator System

- Concentrates or dries large-volume samples rapidly and in parallel
- Evaporates directly into the autosampler vials
- Minimizes cross-contamination and sample loss

ASE 350 Accelerated Solvent Extractor and the Rocket Evaporator System

Powerful tools to simplify complexity of dioxin analysis

Chromeleon software is fully scalable, from a single workstation to an enterprise-wide installation, and can provide control of more than 500 modules from Thermo Fisher Scientific and many other vendors, using the same intuitive user interface. With the Dioxin Analyzer, Chromeleon CDS simplifies all data processing and reporting using a comprehensive suite of software tools and pre-loaded acquisition, calculation and reporting templates designed to meet EU requirements for confirmatory analysis of dioxin/furan and PCBs—all with ultimate confidence of the highest data integrity and compliance-ready data processing.

Error-free execution of routine analysis

The eWorkflow™ procedures provide a pre-loaded template that captures the unique aspects of a chromatography workflow and guides the operator through a minimal number of choices needed to create a finalized sequence with predefined files and a well-defined method structure. The Dioxin Analyzer eWorkflow includes all pre-optimized SRM transitions, isotope dilution calculation and reporting templates for an error-free execution of the analysis to meet compliance requirements.

Interactive results pane with real-time updates

Interactive results pane showing ISTD recovery and ion ratio deviation (flagged red if outside limits) and upper, middle and lower-bound congener specific result. Sum WHO-PCDD/F-TEQ result and flag to indicate above/below maximum limit (top right). Ion ratio deviation and congener specific contribution to the WHO-PCDD/F-TEQ (bottom right).

Compliance control at a glance

Compliance tools are available in the results pane and dynamically updated during the data acquisition for easy and immediate checking of results, thus saving time. This template shows for instance, internal standard (ISTD) recovery and ion ratio deviation, using a color-coded flag to visually highlight compliance/noncompliance throughout the sequence. Dynamically updated calculation results for sample Toxicity Equivalent (TEQ) are also shown and color flagged for faster action in case the results are outside of the acceptable limits.

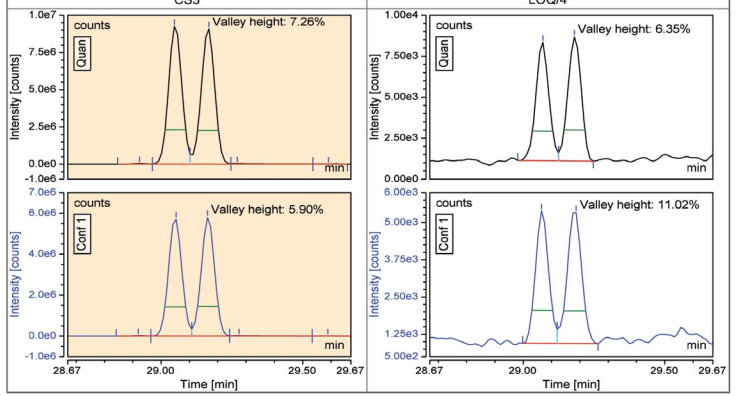
System suitability test report

Chromatogram and SST Results

Injection Details

Injection Name:	**LOQ/4**	*Run Time (min):*	**44.700**
Vial Number:	**28**	*Injection Volume:*	**4.00**
Injection Type:	**Check Standard**		
Calibration Level:	**PCDD/F LOQ/4**		
Instrument Method:	**Non-ortho PCBs and PCDD-Fs - PTV**		
Processing Method:	**non-ortho PCBs and PCDD-Fs**	*Dilution Factor:*	**1.0000**
Injection Date/Time:	**25/Sep/18 14:10**	*Sample Weight:*	**1.0000**

Chromatogram



SST Results

No.	Name	Inj.Condition	Result	Inj. Eval. Result	Injection
1	HxCDF separation	(injection.calLevel="PCDD/F CS3")	Not Executed	6.348	LOQ/4

Number of executed test cases: 1 **Total Result: Passed**

System suitability results built into the method allow for intelligent run control, ensuring samples are only analyzed if the system passes specification, saving repeat analysis time, acquisition and processing of non-compliant data

Built-in reporting tool

To simplify data reporting, the Dioxin Analyzer eWorkflow offers a comprehensive template that includes the required results and calculations to meet all quality and compliance requirements. This includes recovery for ^{13}C-labeled standards, ion ratio, sum parameters, and LOQ flagging amongst other features. If needed, the Report Designer enables further customization to meet all reporting and charting requirements.

Comprehensive calibration report

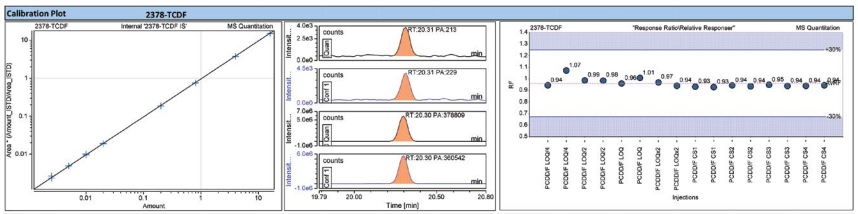
A

Calibration

Calibration Details 2378-TCDF

Calibration Type	AvgCalFact	*Offset (C0)*	0.0000
Evaluation Type	Area	*Average Response Factor*	0.9608
Number of Calibration Points	16	*Curve (C2)*	0.0000
Number of disabled Calibration Points	0	*R-Square*	0.99946

Calibration Plot 2378-TCDF



Calibration Results 2378-TCDF

No.	Injection Name	Level	Quan SRM m/z 2378-TCDF	Qual SRM m/z 2378-TCDF	Quan Area counts*min 2378-TCDF	Qual Area counts*min 2378-TCDF	Target Ratio % 2378-TCDF	IR deviation % 2378-TCDF	X value 2378-TCDF	Y value 2378-TCDF	Residual for Cal. Point X 2378-TCDF	RF 2378-TCDF	RF deviation % 2378-TCDF
3	LOQ/4	PCDD/F LOQ/4	303.9 / 240.9	305.9 / 242.9	238.8	247.903	98.40	5.5	0.0025	0.0024	-1.77	0.944	1.7%
4	LOQ/4	PCDD/F LOQ/4	303.9 / 240.9	305.9 / 242.9	251.8	226.625	98.40	-8.5	0.0025	0.0027	11.55	1.072	-11.1%
5	LOQ/2	PCDD/F LOQ/2	303.9 / 240.9	305.9 / 242.9	482.3	469.772	98.40	-1.0	0.0050	0.0049	2.67	0.985	-2.6%
6	LOQ/2	PCDD/F LOQ/2	303.9 / 240.9	305.9 / 242.9	425.7	416.241	98.40	-0.2	0.0050	0.0049	2.44	0.984	-2.3%
7	LOQ	PCDD/F LOQ	303.9 / 240.9	305.9 / 242.9	889.0	853.900	98.40	-2.4	0.0100	0.0096	-0.12	0.960	0.1%
8	LOQ	PCDD/F LOQ	303.9 / 240.9	305.9 / 242.9	859.4	772.505	98.40	-8.6	0.0100	0.0101	4.94	1.008	-4.8%
9	LOQ*2	PCDD/F LOQx2	303.9 / 240.9	305.9 / 242.9	1642.9	1515.245	98.40	-6.3	0.0200	0.0194	0.87	0.969	-0.8%
10	LOQ*2	PCDD/F LOQx2	303.9 / 240.9	305.9 / 242.9	1497.5	1423.660	98.40	-3.4	0.0200	0.0188	-2.15	0.940	2.1%
11	CS1	PCDD/F CS1	303.9 / 240.9	305.9 / 242.9	16378.1	15503.763	98.40	-3.8	0.2000	0.1864	-2.99	0.932	2.9%
12	CS1	PCDD/F CS1	303.9 / 240.9	305.9 / 242.9	15694.6	14867.928	98.40	-3.6	0.2000	0.1857	-3.39	0.928	3.3%
13	CS2	PCDD/F CS2	303.9 / 240.9	305.9 / 242.9	70639.4	66960.188	98.40	-4.0	0.8000	0.7548	-1.81	0.943	1.7%
14	CS2	PCDD/F CS2	303.9 / 240.9	305.9 / 242.9	56854.8	54187.241	98.40	-3.1	0.8000	0.7487	-2.60	0.936	2.5%
15	CS3	PCDD/F CS3	303.9 / 240.9	305.9 / 242.9	292420.7	275292.884	98.40	-4.3	4.0000	3.7937	-1.29	0.948	1.2%
16	CS3	PCDD/F CS3	303.9 / 240.9	305.9 / 242.9	275790.2	261557.296	98.40	-3.6	4.0000	3.7546	-2.31	0.939	2.2%
17	CS4	PCDD/F CS4	303.9 / 240.9	305.9 / 242.9	1230656.9	1175701.454	98.40	-2.9	16.0000	15.0263	-2.26	0.939	2.2%
18	CS4	PCDD/F CS4	303.9 / 240.9	305.9 / 242.9	1189144.7	1120101.192	98.40	-4.3	16.0000	15.0970	-1.80	0.944	1.7%

Comprehensive sample report template

B

Injection results

Injection Details

Injection Name:	*OK1 1*	*Run Time (min):*	*44.700*
Vial Number:	*25*	*Injection Volume:*	*4.00*
Injection Type:	*Unknown*	*Comment:*	*Mixed Fat*
Calibration Level:		*Lipid content (%)*	*100.000*
Instrument Method:	*Non-ortho PCBs and PCDD-Fs - PTV*	*Dry Mass (%)*	*100.000*
Processing Method:	*non-ortho PCBs and PCDD-Fs*	*Dilution Factor:*	*20.0000*
Injection Date/Time:	*25/09/2018 11:38:01 +01:00*	*Sample Weight:*	*1.9870*

Peak Name	Retention Time min	Quantitation Ion	Qualification Ion	Quan Area counts*min	Qual Area counts*min	Target Ratio %	Cont. Ratio %	IR deviation %	ISTD recovery %	Sample LOQ pg/g	<LOQ?	WHO TEF	Upperbound WHO-PCDD/F-TEQ pg/g	Middlebound WHO-PCDD/F-TEQ pg/g	Lowerbound WHO-PCDD/F-TEQ pg/g
2378-TCDF	20.312	303.9 / 240.9	305.9 / 242.9	2765.9	2720.7	99.40	98.37	0.0	79.18	0.0018		0.1000	0.0483	0.0483	0.0483
12378-TCDD	20.882	319.9 / 256.9	321.9 / 258.9	276.5	273.8	94.75	99.02	4.5	86.15	0.0092		0.1000	0.0743	0.0743	0.0743
12378-PeCDF	24.347	339.9 / 276.9	337.9 / 274.9	752.3	652.3	73.28	86.71	10.8	94.21	0.0534		0.0300	0.0042	0.0042	0.0042
23478-PeCDF	25.720	339.9 / 276.9	337.9 / 274.9	1337.5	1090.9	77.94	78.57	0.8	94.82	0.0531		0.3000	0.0680	0.0680	0.0680
12378-PeCDD	25.969	355.9 / 292.9	353.9 / 290.9	1109.1	883.5	79.00	79.66	2.1	95.71	0.0505		1.0000	0.3386	0.3386	0.3386
123478-HxCDF	29.069	373.8 / 310.9	371.8 / 308.9	502.7	297.5	62.98	55.86	-11.3	81.18	0.0620		0.1000	0.0113	0.0113	0.0113
123678-HxCDF	28.180	373.8 / 310.9	371.8 / 308.9	576.5	345.7	62.97	59.95	-4.8	87.39	0.0576		0.1000	0.0113	0.0113	0.0113
234678-HxCDF	29.856	373.8 / 310.9	371.8 / 308.9	500.6	335.4	62.60	67.00	7.0	76.77	0.0656		0.1000	0.0115	0.0115	0.0115
123478-HxCDD	29.939	389.8 / 326.9	391.8 / 328.9	1489.3	976.7	63.89	65.58	2.6	88.39	0.1139		0.1000	0.0475	0.0475	0.0475
123678-HxCDD	30.039	389.8 / 326.9	391.8 / 328.9	3460.3	2269.8	64.93	65.59	1.0	86.91	0.1144		0.1000	0.1112	0.1112	0.1112
123789-HxCDD	30.361	389.8 / 326.9	391.8 / 328.9	1639.9	1212.4	64.24	65.90	2.6	86.91	0.1144		0.1000	0.0808	0.0808	0.0808
123789-HxCDF	30.713	373.8 / 310.9	371.8 / 308.9	240.2	172.3	63.89	71.73	12.8	71.23	0.0707	<LOQ	0.1000	0.0071	0.0035	0.0000
1234678-HpCDF	32.360	407.8 / 344.8	409.8 / 346.8	472.6	403.2	80.44	85.31	6.1	80.75	0.1807	<LOQ	0.0100	0.0017	0.0008	0.0000
1234678-HpCDD	33.792	423.8 / 360.8	425.8 / 362.8	17262.9	13832.3	80.10	80.13	-0.7	94.22	0.1068		0.0100	0.0688	0.0688	0.0688
1234789-HpCDF	34.519	407.8 / 344.8	409.8 / 346.8	220.4	172.5	79.90	78.28	-2.0	81.83	0.1230	<LOQ	0.0100	0.0012	0.0006	0.0000
OCDD	36.395	457.7 / 394.8	459.7 / 396.8	14186.5	13473.3	96.02	94.97	-1.1	91.29	0.4411		0.0003	0.0039	0.0039	0.0039
OCDF	38.668	441.8 / 378.8	443.8 / 380.8	296.1	288.3	94.10	97.37	3.5	83.02	0.4850	<LOQ	0.0003	0.0001	0.0001	0.0000
												SUM WHO-PCDD/F-TEQ (pg/g)	**0.869**	**0.864**	**0.859**

A. Key information includes average response factor, response factor deviation and Ion ratio
B. Key results include ^{13}C-labeled standards recovery, ion ratio, sum parameters, LOQ flagging

thermo scientific

The most comprehensive GC-MS portfolio for dioxins and POPs analysis

In addition to the Dioxin Analyzer, Thermo Fisher Scientific offers complementary technology for the analysis of dioxins/furans and persistent organic pollutants (POPs). Each solution has specific advantages and collectively offer the most comprehensive GC-MS portfolio available on the market.

Magnetic Sector GC-HRMS

The Thermo Scientific™ DFS™ Magnetic Sector GC-HRMS offers worldwide full compliance with any official Dioxin, PCB or PBDE method (e.g., EPA 1613, 1668, 1614). Laboratories measuring not only the maximum levels, but also low background levels in food and animal feed, can exploit the benefits of unmatched sensitivity for dioxin/furan and dl-PCB and robustness, delivered by the most established dioxin solution.

Orbitrap GC-MS/MS

The Thermo Scientific™ Q Exactive™ GC Orbitrap™ GC-MS/MS system is a full-scan, high-resolution, accurate-mass (HRAM) dedicated benchtop GC-MS system that provides the highest confidence for screening legacy and emerging contaminants, and subsequent identification and quantitation for a comprehensive characterization of samples. This performance is achieved through the superior resolving power, mass accuracy and sensitivity that only Orbitrap technology can deliver.

Find out more at **thermofisher.com/DioxinAnalyzer**

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