Better, Smarter GC

The Agilent Intuvo System is designed to help you work smarter - find out how it can boost efficiency in your GC lab.

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What is Intuvo?

Intuvo is a new system comprising capillary gas chromatographs, consumables and supplies. Based on numerous patented innovations designed to streamline GC, the Agilent Intuvo System greatly improves operational efficiency in the GC laboratory.

What is unique in the Intuvo GC System?

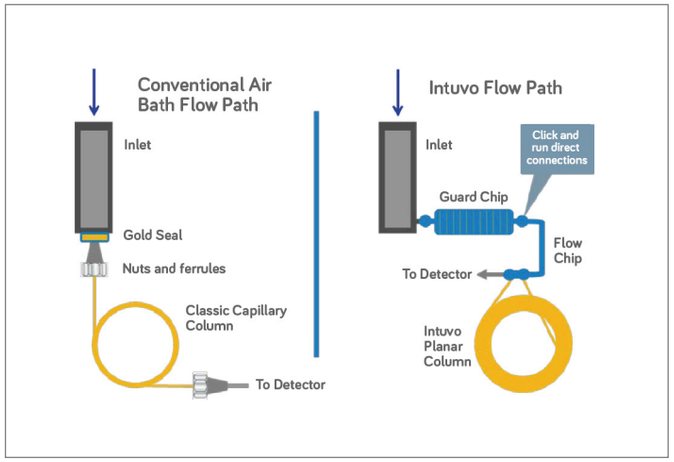
Intuvo is built on three transformational innovations:

1. Direct Heating

Unlike conventional GC systems, which use a convection air bath oven, Intuvo uses direct conductive heating to temperature program the entire flow path and analytical column. Direct heating uses less than half the power of a conventional oven, takes about half the bench space, and can be heated and cooled much faster, improving throughput.

2. Intuvo Click and Run Connections

Cumbersome ferrules used in conventional gas chromatographs are eliminated. Instead, advanced direct face seal connections are made with an audible and tactile 'click', telling the user a correct connection has been made. This eliminates leaks from incorrectly fitted ferrules, minimizing unplanned downtime and associated business disruption.



3. Intuvo Guard Chip and Trim-free Column

Intuvo is designed with a simple, disposable Guard Chip, which serves as a pre-column retention gap. The Guard Chip prevents unwanted material from being depositing on - and damaging - the head of the column. The need to trim columns is eliminated altogether, greatly enhancing productivity, while removing much of the art from GC operation.

What is the benefit of Intuvo for GC operators?

Intuvo is easy to manage and provides streamlined and dependable operation of the GC laboratory. This is achieved by:

Delivering better results

- Intuvo direct heating is fast, reducing sample cycle time
- Changing an Intuvo column is over 10 times faster, improving uptime
- Eliminating column trimming and the associated recalibration and requalification time increases the instrument's productive uptime, which in many cases can be used to run higher-value priority samples sooner

Eliminating mistakes

Leaks and improper installation associated with improperly fitted ferrules and improperly trimmed columns are a major source of downtime, leading to costly reruns and delays, production hold ups, and even the loss of valuable (sometimes irreplaceable) customer samples. With Intuvo, mistakes are avoided by eliminating ferrules and the need to trim columns.

Improving operations

The result? Intuvo helps to drive sustainable operational improvement such as:

- On-time delivery
- Number of priority samples
- Cost per billable sample
- Resource and asset management

How does this compare with traditional GCs?

Dimensions, materials of construction, and separation phases of the column are analogous to existing capillary GC. This all means that GC methods already developed for conventional air bath ovens are transferrable to the Intuvo system.

However, Intuvo comes with a number of features and benefits not seen with GCs on the market today. Intuvo shines most anywhere routine high-throughput GC analyses are carried out. By streamlining the GC experience, including installation, setup, operation and maintenance, Intuvo transforms the way GC is done.

How does Intuvo change the role of the GC operator?

Intuvo greatly reduces complexity and makes accessible what used to be only within the realm of experts. Intuvo changes the way GC operators work by minimizing the time spent on routine tasks. It also minimizes risks associated with maintenance as well as the time spent maintaining the GC, which in the past has been a worry for GC operators, due to the potential for lost productivity.

LINKS

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Quick and Smart

For many years, gas chromatography saw only incremental development in instrumentation, but the Agilent Intuvo 9000 GC system represents a gear-change in ease-of-use and efficiency. How can the productivity and economics of the GC lab be improved by Intuvo?

Leak-Free Connections

Challenge
Leaks resulting from faulty GC connections are a major source of unplanned downtime and productivity loss.

Innovation
Intuvo does not require nuts and ferrules to make flow path connections. Instead, it uses direct, face-to-face, click-and-run connections. The audible and tactile click tells operators they have successfully made a leak-free connection. Intuvo's unique automatic leak detection provides continuous assurance.

Saving: Cut downtime by 24 hours per year, based on 8 leaks per year.

Fast Direct Heating and Cooling

Challenge
Minutes matter to many GC labs. Police labs need fast results to support cases. Service labs can charge premium rates for faster analyses. QA labs must maintain production flow. Even small reductions in cycle time can result in big savings.

Innovation
Intuvo achieves faster throughput with an innovative direct heating and cooling system. Unlike conventional air bath oven GC systems, an Intuvo column can be heated at rates as high as 250 °/min, and cooled 1–2 minutes faster, while using half the power consumption, half the space, and placing half the demand on the lab HVAC system.

Saving: Just one extra sample per day could increase revenue by over $30,000/year.

Elimination of Column Trimming

Challenge
Trimming capillary columns to remove contamination by sample matrix is a skilled and time-consuming maintenance task. Over time, trimming causes the performance of the column to degrade, and the change in column length requires time-consuming recalibration.

Innovation
Intuvo eliminates the need for trimming by using a disposable Guard Chip between the inlet and the column. The Chip acts as a retention gap to trap unwanted high-molecular-weight material, keeping it from depositing on the column, and prolonging its life. The Guard Chip can be changed in just 3–5 minutes, compared to 20–30 minutes to trim and reinstall a conventional column. It also avoids the downtime caused by improper trimming. There is no change in column length and no shift in retention time.

Saving: In a typical food or environmental lab, eliminating column trimming could save over $7,500/year, while reducing re-calibration could save over $9,000.

Incremental Economic Value of Agilent Intuvo

These examples illustrate how the transformational innovations of Intuvo could provide a much better return on innovation compared to conventional GC systems.

One of the biggest values of Intuvo is the reduction in business uncertainty resulting from unplanned downtime. Providing more consistent and predictable business results, day to day, especially between operators or between operational sites across the globe, can be one of the most important returns on an Intuvo investment.

Read the full white paper to find out more about the return on investment of Intuvo and details of how we estimated cost savings.

LINKS

White Paper
Improving ROI

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Application Note: Mineral Oil Total Petroleum Hydrocarbon

Introduction

The analysis of hydrocarbon contaminants in environmental samples is commonly called TPH, or total petroleum hydrocarbon determination. For TPH analysis, chromatographic separation of individual compounds is not required. Instead, the entire sample can elute as a largely unresolved mass for quantification. This fact permits the use of ultra-fast GC techniques; namely, short columns, fast temperature program ramps, and high carrier gas flow rates. The short run times typical of ultra-fast GC allow many more analyses in a typical workday, an important consideration for labs struggling to keep up with large sample backlogs. In addition to ultra-fast GC, Intuvo provides unrivalled chromatographic reliability.

The Agilent Intuvo 9000 GC is designed to enable ultra-fast GC separations with a high degree of precision and reliability.

Experimental

For this work a Certified Reference Material (BAM-U021) composed of TPH contaminated soil was obtained from the Bundesanstalt für Materialforschung und Prüfung in Germany. The sample was certified by consensus analysis to contain 3,560 mg/kg of mineral oil hydrocarbons with an uncertainty of 260 mg/kg. Prior to GC analysis, the sample was prepared in duplicate using the extraction and cleanup procedure described in ISO Method 167031.

Results and Discussion

Five injections of each BAM-U021 duplicate sample were run on the Agilent Intuvo 9000 GC. Figure 1 shows an overlay of a single run from each duplicate sample. Each analysis is completed in about three minutes using Ultra-Fast GC conditions. The C10 and C44 peaks are added to the sample to serve as integration markers

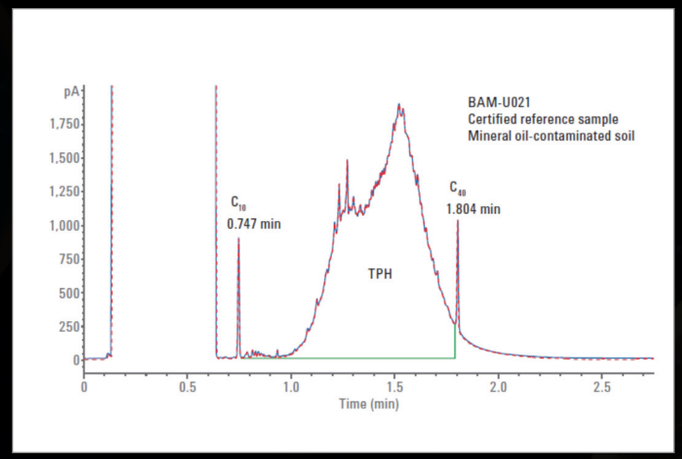


Figure 1. Overlay of duplicate TPH soil extracts

for the total TPH sample response. Each run has nearly identical chromatographic response and retention time. Table 1 shows the quantitative results.

Three analysis performance measures are illustrated with this data. First, the method accuracy is shown by comparing the mean results to the BAM certified values. Each duplicate analysis agrees with the 3,560 mg/kg certified value. Next, the instrument precision is shown by the RSDs calculated for each duplicate. Five runs of each duplicate sample have a quantitate RSD of less than one percent. Finally, the single lab method precision is measured using the single lab repeatability (r) test described in the ISO 16703 method. The experimental repeatability of 59 mg/kg is well below the required value of 139 mg/kg.

Conclusion

An ultra-fast GC method was developed for the analysis of TPH in environmental samples. The Agilent Intuvo 9000 GC has the instrument performance to deliver rapid column heating, rapid cool down, and high flow rates using Agilent Intuvo GC columns to run this method with a high degree of method accuracy and precision.

Run	*U021 A (mg/kg)*	*U021 B (mg/kg)*
1	3,462	3,480
2	3,487	3,485
3	3,502	3,482
4	3,513	3,479
5	3,538	3,492
Mean	3,500	3,484
Cert. value	3,560 ± 260	
Std Dev	28.547	5.234
RSD	0.82 %	0.15 %
r (exp)	59	
r* (ref)	136	

Table 2. Repeatability for duplicate analyses of TPH soil extracts

Application Note: USP 467: Analysis of Residual Solvents

Introduction

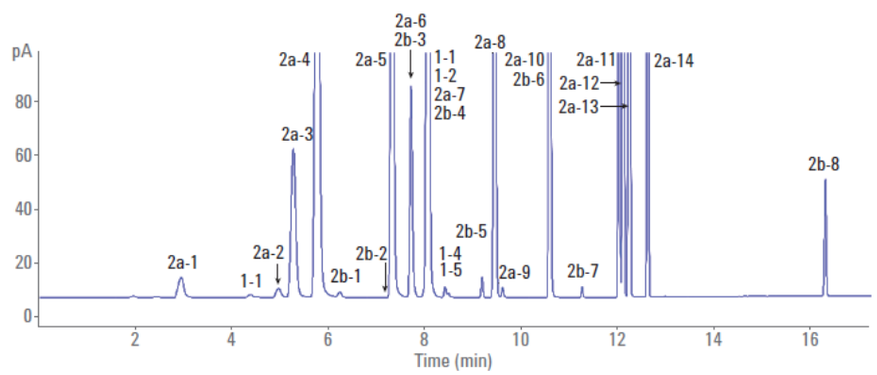
Analysis of residual solvent is a critical application in the pharmaceutical industry. The choice of solvent during manufacturing can improve yield or affect the properties of the product synthesized. However, solvents do not enhance the product's efficacy and must be removed as completely as possible to meet product specifications and good manufacturing practices. As a result, testing for residual solvents during production or purification processes is a necessary aspect of manufacturing.

Analysis of residual solvents according to USP 467 was evaluated on an Agilent Intuvo system equipped with a headspace sampler.

The Agilent Intuvo 9000 Gas Chromatograph yields advantages over conventional GC systems:

- Modular flow path for simplified sample splitting to two columns
- Quick column changes for easier method development
- Smaller footprint

Figure 1. GC analysis of a mixture of three standard solutions.



Class 1	RSD %
1,1-Dichloroethane (1-1)	2.7
1,1,1-Trichlorethane (1-2)	2.1
Carbon tetrachloride (1-3)	4.5
Benzene (1-4)	1.9
1,2-Dichlorobenzene (1-5)	0.93
1,1-Dichloroethane (1-6)	2.7

Table 1. Class 1 solvent standard repeatability

Experimental

An Intuvo 9000 GC was equipped with an Agilent 7697A Headspace Sampler. Class 1, Class 2a, and Class 2b standard solutions were prepared and evaluated according to USP 467 methodology (see Tables 1, 2 and 3).

Results and Discussion

Eight headspace vials were prepared for each solvent standard (Classes 1, 2a, and 2b), and repeatability was evaluated. Repeatability was very good, with all but one compound yielding RSDs of less than five percent (Tables 1–3). While USP 467 does not have specific RSD requirements, five percent RSD is acceptable for most laboratories.

The three standard solutions were then mixed to evaluate the three classes in a single run. Figure 1 shows the resulting chromatogram. The differences in concentration and coelutions of multiple compounds demonstrate the need to run these as separate mixes, or use additional analytical techniques

Conclusion

The Agilent Intuvo 9000 GC equipped with the Agilent 7697A Headspace Sampler delivers excellent repeatability performance for USP 467 Class 1, 2a, and 2b solvent standards. However, when attempting to analyze the three mixes together, difficulties arise due to differences in concentration and coeluting analytes. Additional analytical techniques such as splitting to two columns for dual detector analysis or using a mass spectrometer as a detector would improve detection and identification of the analytes in a single mix (from application 5991-8032EN).

Class 2a	RSD %
Methanol	1.3
Acetonitrile	0.98
Dichloromethane	1.3
trans-1,2-Dichloroethene	2.4
cis-1,2-Dichloroethene	1.7
Tetrahydrofuran	0.69
Cyclohexane	2.5
Methylcyclohexane	2.7
1,4-Dioxane	1.1
Toluene	2.1
Chlorobenzene	1.8
Ethylbenzene	2.3
m,p-Xylene	2.3
o-Xylene	2.1

Table 2. Class 2a solvent standard repeatability

Class 2b	RSD %
Hexane	4.6
Nitromethane	6.7
Chloroform	4.2
1,2-Dimethoxyethane	3.7
Trichloroethylene	4.6
Pyridine	2.8
2-Hexanone	2.9
Tetralin	3.6

Table 3. Class 2b solvent standard repeatability

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Application Note: Analysis of Semivolatile Organic Compounds By Intuvo GC/MSD

By Matthew Giardina

Introduction

GC/MS is regarded as the analytical technique of choice for the analysis of semivolatile organic compounds (SVOC). A number of compounds within the class of (SVOCs). are considered environmental pollutants. Government agencies with regulatory authority have established methods and set performance criteria for the measurement SVOCs in a number of environmental and industrial matrices. For example, United States Environmental Protection Agency (USEPA) method 8270D contains a list of 243 compounds that are suitable for analysis by GC/MS in solid waste, soil, air and water extracts detailing specifications and requirements for quantitative analysis.

The durability and data quality from the Agilent 7890 GC has set the standard for the analysis of SVOCs and these instruments are used in environmental testing laboratories throughout the world. With the development of new technology, it is critical there is no negative impact on robustness or performance.

The Intuvo 9000 GC includes a number of design innovations making it ideally suited for SVOC analysis. Intuvo Flow Technology (IFT) incorporates the use of an easy to install and replace Intuvo Column and Guard Chip to act as a precolumn to prevent particulate and nonvolatile contamination of the column and flow path. Direct heating technology reduces power requirements, and facilitates more rapid column cooling for faster cycle time.

The system durability is demonstrated by the repetitive injection of a composite soil extract.

Experimental

A stock standard containing 77 target compounds and surrogates was purchased from AccuStandard (New Haven, CT). The standard was selected to provide a representative mixture of acids, bases, and neutrals. The stock standard was diluted in dichloromethane containing six internal standards. A composite mixture of soils extracted with dichloromethane prepared for method 8270 was donated from ESC Lab Sciences (Mt. Juliet, TN). The extracts selected for the composite mixture contained the heaviest matrix residue typically encountered in their laboratory.

To demonstrate equivalency, a standard prepared with a target and surrogate concentration of 20 µg/mL, and an internal standard concentration of 40 µg/mL was injected on a 7890 GC coupled to a 5977B MSD and a 30 m Agilent J&W DB-5ms Ultra Inert column, and an Intuvo 9000 GC coupled to a 5977B MSD and a 30 m Intuvo DB-5ms Ultra Inert column. The same column temperature program and detector conditions were used for analysis. Figure 1 shows a normalized total ion chromatogram obtained on both systems.

The relative responses of the last eluting polyaromatic hydrocarbons (PAHs) indeno[1,2,3-cd] pyrene, dibenzo[a,h] anthracene, and benzo[g,h,i]perylene were slightly greater on the Intuvo 9000 GC compared to the 7890 GC. This demonstrates that the thermal profile across the Intuvo 9000 GC flow path is consistent, allowing the higher boiling point PAHs to pass through the flow path, and maintain recovery and peak shape.

Tailing factor (TF) was used as the determinant of acid/base activity of pentachlorophenol and benzidine. Based upon method 8270D requirements, the TF measured at 10 percent peak height for the extracted quantitation ion should be no greater than two. For pentachlorophenol and benzidine, the measured tailing factors were 1.0 and 0.8, respectively (Figure 2).

Conclusion

This study demonstrates the suitability of the Agilent Intuvo 9000 GC for the analysis of SVOCs. The Intuvo 9000 GC can easily meet the performance requirements as specified by USEPA method 8270D. Compared to the Agilent 7890 GC, the Intuvo 9000 GC provided equivalent results in terms of relative retention time and relative response. In addition, repetitive injections of a soil extract illustrated the resilience of the Intuvo 9000 GC to a substantial matrix challenge, and it was easier to maintain compared to the 7890 GC. Intuvo Guard Chip replacement was more expedient than column trimming in terms of maintenance time, and did not require retention time adjustment.

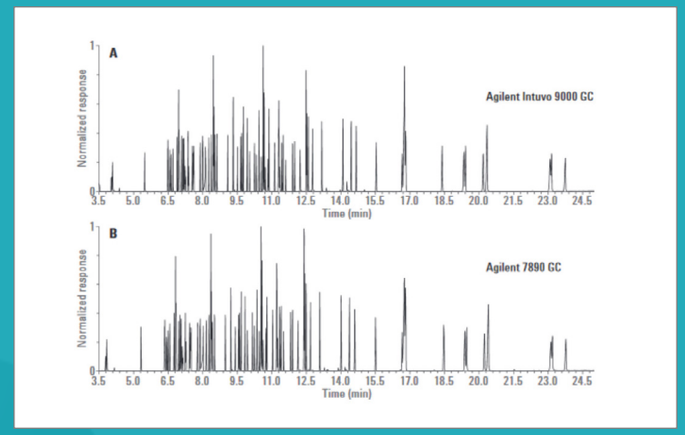

Figure 1. Comparison of SVOC chromatograms generated with an Agilent Intuvo 9000 GC (A) and an Agilent 7890 GC (B).

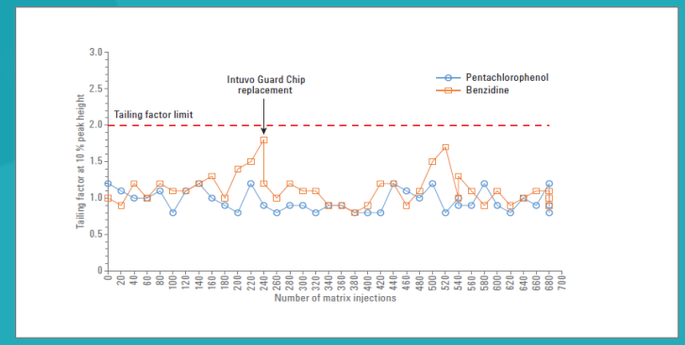

Figure 2. Tailing factor measurements after liner replacement for pentachlorophenol and benzidine.

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Application Note: Dioxins Analysis in Food and Feed by Intuvo 9000/7010 GC-QQQ System

By Stefano Mazzotta, Studio Effemme Chimica Applicata, and Anna Cali and Rebecca Veeneman, Agilent Technologies

Introduction

Dioxins and furans are persistent environmental pollutants that have been extensively studied and shown to bioaccumulate in the environment. More than 90 percent of human exposure is through the consumption of food. Dioxins are toxic, and their impact to the environment and human health via exposure have been studied and documented for decades. Dioxins and furans have shown toxic effects in animal studies and damage to reproductive and immune systems and are carcinogenic.

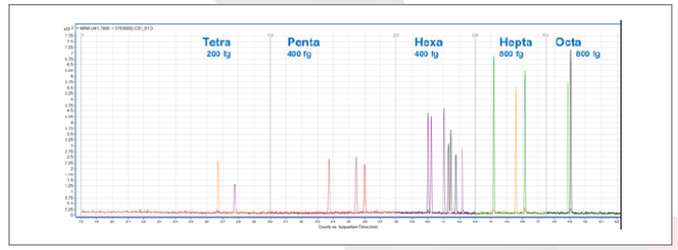
Historically, high resolution mass spectrometry (HRMS) was needed to confirm and quantify trace levels of dioxins. However, since 2014, the European Union (EU) has instituted a regulation (709/2014) governing the levels of PCDDs, PCDFs, dioxin-like PCBs, and non-dioxin-like (NDL) PCBs in food and feed that enables the use of tandem mass spectrometry (GC/MS/MS) systems in confirmatory testing for compliance with EU MLs. This change followed evidence that triple quadrupole mass spectrometers could provide sensitivity concomitant with HRMS systems.

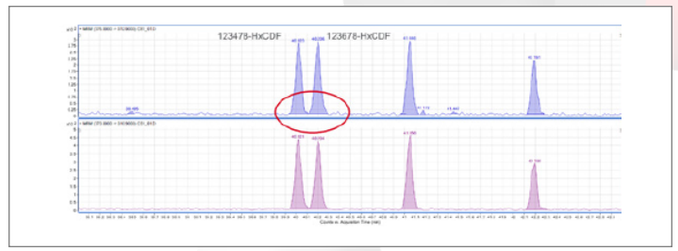
Experimental

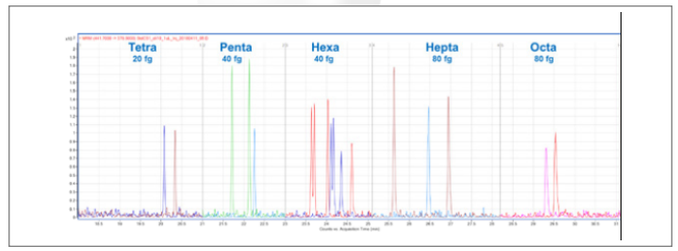
Experiments were performed using an Agilent Intuvo GC coupled to a 7010 Tandem MS with High Efficiency Ion Source (HES). Two methods were developed to meet various needs. The first method meets the minimum retention times for the internal standards and the relative retention times for the CDDs/CDFs reported in 1613 method.

The second method is targeted for speed with a 31-minute run time. Obviously this ignores the retention time requirements of 1613 method.

Name	RT	Transition	conc RSD	MDL	LOQ	LOD	Noise	S/N
2378-TCDF	26.64	303.9 -> 240.9	2.6	3.8184	13.5334	4.06	1.92	18.54
2378-TCDD	27.835	319.9 -> 256.9	3.9	5.7684	20.445	6.1335	1.61	14.62
12378-PCDF	33.638	339.8 -> 277.0	4.9	14.8674	52.6945	15.8083	2.02	17.6
23478-PCDF	35.359	339.8 -> 277.0	6.4	20.1087	71.271	21.3813	1.99	21.43
12378-PCDD	35.921	355.9 -> 292.9	6.5	20.0992	71.2375	21.3713	1.48	19.69
123478-HxCDF	39.96	373.8 -> 310.9	6.4	19.4782	69.0366	20.711	1.55	24.45
123678-HxCDF	40.141	373.8 -> 310.9	4.6	14.2778	50.6047	15.1814	1.55	25.08
234678-HxCDF	41.148	373.8 -> 310.9	3.4	10.0481	35.6132	10.684	1.96	22.2
123478-HxCDD	41.237	389.8 -> 326.9	4.2	12.5373	44.4357	13.3307	1.51	23.82
123678-HxCDD	41.533	389.8 -> 326.9	8.1	24.7315	87.6558	26.2967	1.45	25.78
123789-HxCDD	41.737	389.8 -> 326.9	7.3	21.5291	76.3055	22.8916	1.55	26.1
123789-HxCDF	42.135	373.8 -> 310.9	4.9	14.8626	52.6774	15.8032	1.39	30.68
1234678-HpCDF	44.133	407.8 -> 344.8	4	23.4349	83.06	24.918	1.76	46.85
1234678-HpCDD	45.674	423.8 -> 360.8	3.5	20.7641	73.594	22.0782	1.9	34.19
1234789-HpCDF	46.105	407.8 -> 344.8	8.7	52.986	187.7979	56.3394	1.52	52.61
OCDD	48.83	457.7 -> 394.8	2.8	16.7276	59.2874	17.7862	1.33	44.22
OCDF	48.995	441.7 -> 378.8	2.5	14.9213	52.8855	15.8656	1.35	49.63







Results

Method 1 meets minimum retention time and resolution parameters outlined in EPA 1613 methodology. For the internal standards and the relative retention times for the CDDs/CDFs, the height of the valley between the most closely eluted isomers and the 2,3,7,8-substituted isomers is less than 25 percent as reported in 1613 method (Figure 1) and gas chromatographic separation of isomers shall be sufficient.

The second method demonstrates required isomer resolution in a method run time of some 31 minutes

Conclusion

The Intuvo GC/7010 triple quadrupole MS/MS system has been tested for the analysis of PCDDs, PCDFs in food and feed.
Two methods were developed to meet various needs.

This is compliant with new European Union Commission Regulations No 589/2014 and No 709/2014, which allow GC/MS/MS use as a confirmatory method for analysis of certain foodstuffs.

Agilent has developed specific software scripts and a customized report for the analysis of dioxin and dioxin-like PCBs in feed and food, in compliance with EU regulations.

Application Note: Rapid Separation of Fatty Acid Methyl Esters

by Yun Zou

Abstract

The analysis of fatty acid methyl esters (FAMEs) for the characterization of the lipid content fraction in foods is an important application in food analysis.

This Application Note explores rapid separation of FAME mixtures using an Intuvo 9000 GC system with an Agilent J&W DB-FastFAME Intuvo column.

Introduction

The analysis of oils, fat, and fat-containing food is a common task in food-testing laboratories. The GC analysis of fatty acids as their FAME derivatives is an important tool in the characterization of fats in the determination of total fat and trans-fat content in foods. Many regulatory methods for testing foods, such as edible oils, require separation of specific cis/trans fatty acid isomers using a capillary column coated with a cyanopropyl stationary phase when determining fatty acid composition.

Often 100 m columns and long analysis times (>70 minutes) are required to achieve good FAME separations. This analytical run time means low productivity and efficiency. The DB-FastFAME Intuvo GC column with a cyanopropyl phase was specifically engineered for the fast separation of FAME mixtures, including some key cis/trans separations, to meet the requirements of regulatory methods. When coupled with the rapid heating and cooling of the direct heating Intuvo GC, productivity increases by orders of magnitude.

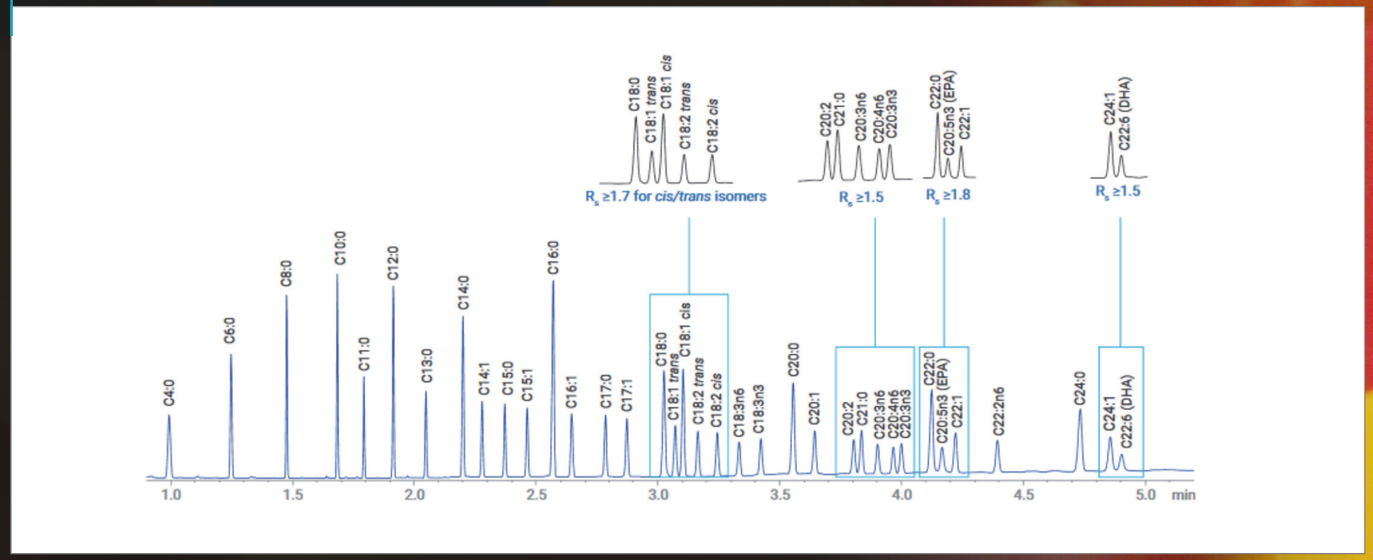


Figure 1. GC/FID chromatogram of the FAME 36 component mixture on a 20 m x 0.18 mm, 0.20 µm DB-FastFAME Intuvo column using Method 1 with helium as carrier gas.

Results and discussion

The FAME 36 component standard mix is designed to mimic the fatty acid composition of many food samples; it can be used to identify key FAMEs in many foods. This mix contains FAMEs ranging from C4:0 to C24:1, including most of the important saturated, monounsaturated, and polyunsaturated FAMEs.

Figure 1 shows the separation of the FAME 36 component mixture on the 20 m × 0.18 mm, 0.20 µm DB-FastFAME Intuvo GC column. The method with helium as the carrier gas completely resolves all compounds within five minutes, including AOAC critical pairs Rs>1.5.

Figure 2 shows the typical GC/FID chromatogram of the FAME mix with rapeseed oil. Using this method, good peak shape and resolution was obtained, with an analysis time of five minutes. Using hydrogen as carrier gas, the C4–C24 even carbon saturated FAME mixture and the FAME 36 component mixture can be well separated in less than four minutes (Figures 3). This indicates that fast sample throughput can be achieved with the column without compromising resolution. The full application note demonstrates 37 component FAME mix fully resolved with a total run time of under 6.5 minutes.

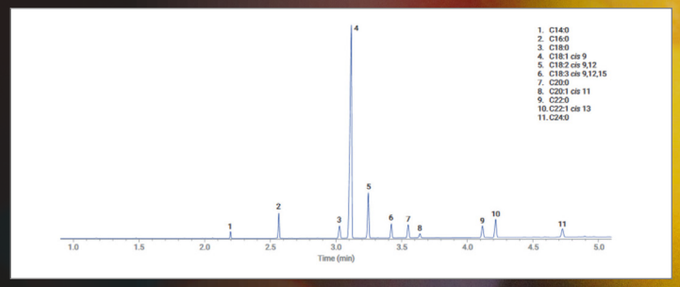


Figure 2. GC/FID chromatogram of rapeseed oil on a 20 m x 0.18 mm, 0.20 µm DB-FastFAME Intuvo column using Method 1 with helium as carrier gas (see Table 1).

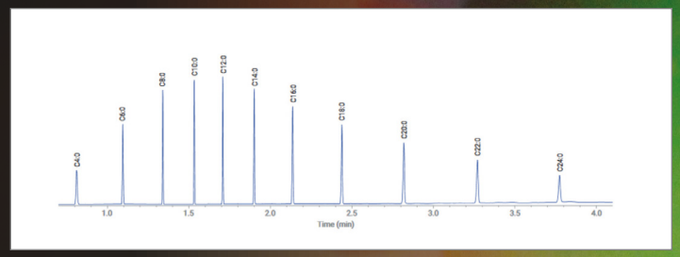


Figure 3. GC/FID chromatogram of the C4-C24 even carbon saturated FAME mixture on a 20 m x 0.18 mm, 0.20 µm DB-FastFAME Intuvo column using Method 2 with hydrogen as carrier gas (see Table 2).

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Application Note: Phthalates Analysis

Introduction

The need for a robust analytical method for the identification and quantitation of phthalate esters (phthalates) has increased over the past several years. Their common use in plasticizers has increased potential exposure from food, household materials, and children's toys. This drives the need for a reliable method of analysis.

Phthalates can be difficult to analyze using GC/MS for a variety of reasons, including poor peak shape, loss of sensitivity, and signal loss over time.

Issues with analyses such as these can be mitigated with a conventional gas chromatograph/mass spectrometer system, but the Agilent Intuvo 9000 GC, coupled to a mass spectrometer equipped with a high efficiency source (HES) provides additional advantages:

- Simplified column installation
- Innovative inert flow path

A redesigned modular flow path simplifies column installation, while the innovative inert flow path maintains chromatographic integrity through the course of the analysis.

Experimental

An Intuvo 9000 GC was coupled to an MS-HES. A 30 m Intuvo HP-5ms Ultra Inert column was installed and run at 1 mL/min. A variable oven program was used to separate 14 phthalates in a standard obtained from Ultra Scientific (Figure 1). The standard was diluted to 200 ppb in isooctane, and analyzed in Selected Ion Monitoring mode (SIM).

A consistent response was achieved, and the peak shape was unchanged over the course of the analysis. Sharp, symmetric peaks were maintained over 117 injections (Figure 2).

Conclusion

The data demonstrates a robust and reliable method analyzing a range of phthalates at low concentration. The innovative flow path simplifies method development and analysis with the inertness required to maintain.

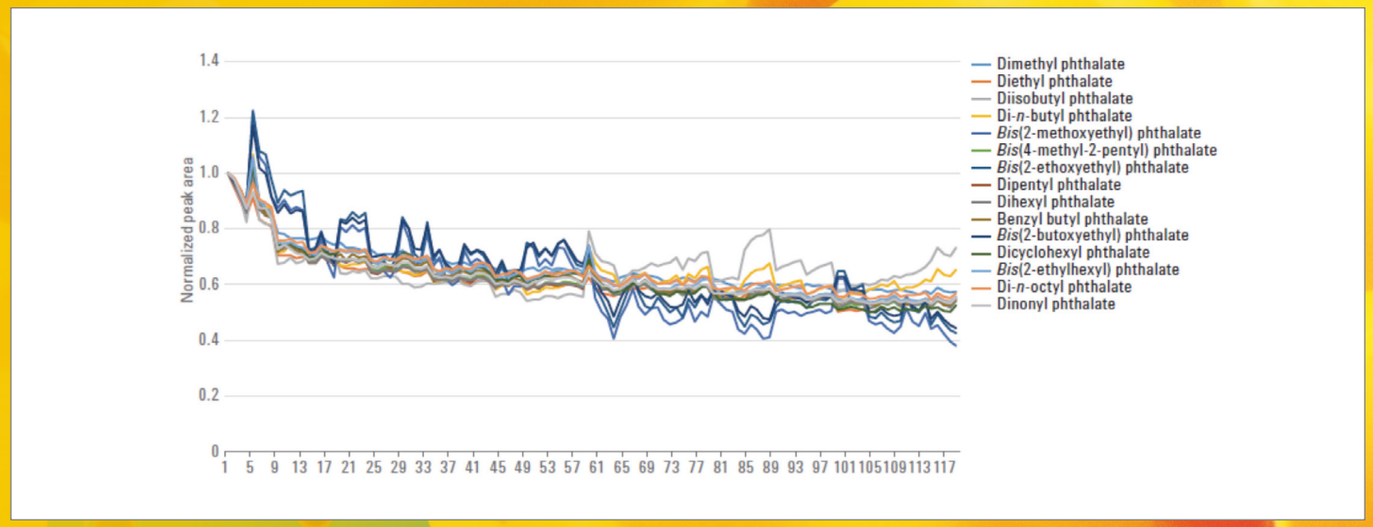

Figure 1. Phthalate normalized area response for 117 injections.

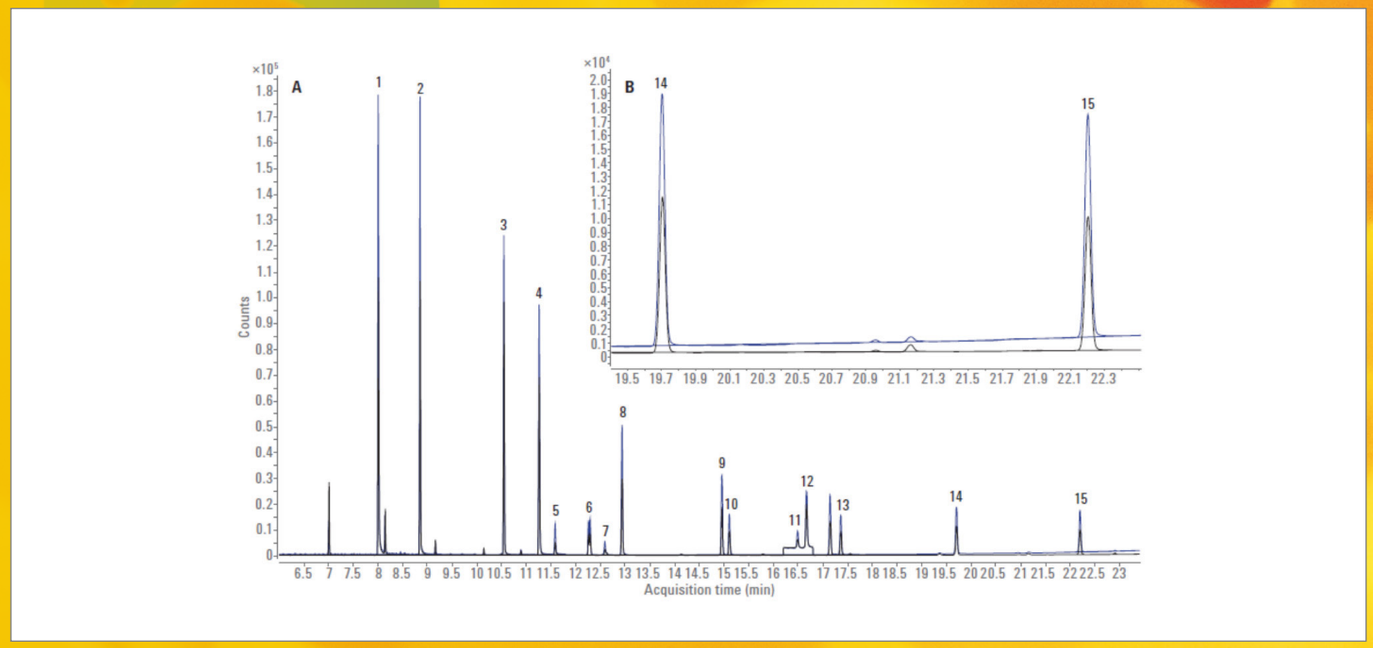

Figure 2. The phthalate standard diluted to 200 ppb at initial injection (blue) and after 117 injections (black). Aside from the difference in response due to initial column bleed, there is no change in peak shape over time.

Application Note: Multiresidue Pesticide Analysis using Agilent Intuvo 9000 GC and 7000 Series Mass Spectrometer

Abstract

This Application Note shows an evaluation of pesticides in seven different matrices for the Agilent Intuvo 9000 GC and 7000 Series Mass Spectrometer. Calibration curves for 21 pesticides showed excellent linearity for concentrations ranging from 1 ng/mL to 1,000 ng/mL. Excellent response and peak shape consistency was obtained with the implementation of the Agilent Intuvo Guard Chip, which protects downstream components and eliminates the need to trim the column after matrix evaluation. Average recovery for a 50 ng/mL sample across 60 food extract injections was over 80 percent with an RSD of less than 10 percent. With regular maintenance, including liner and Intuvo Guard Chip replacements, peak shape and recoveries were found to be unchanged for over 500 injections.

Introduction

As pesticide use has increased, so has the level of concern among environmentalists, regulators, and consumers. Regulations regarding the maximum residue limit (MRL) of pesticides that can be found in or on food have been established in Europe to a default value of 0.01 ppm.

To analyze pesticide residues in foods, some level of sample preparation must be done. At a minimum, the sample must be homogenized and extracted into a solvent suitable for chromatography. The QuEChERS extraction method is widely accepted for these analyses.

While using multiple reaction monitoring (MRM) MS/MS can reduce visible matrix interferences, it does not remove the matrix from the sample. Injecting the matrix can result in loss of signal and peak tailing. This can be mitigated, to some extent, by using backflush. Maintenance, including liner replacements and column trims, are needed to fully maintain the system. The Agilent Intuvo 9000 GC uses a Guard Chip as part of the Intuvo inert flow path, eliminating column maintenance. By removing column trimming from the maintenance model, retention times are left unchanged while the column is protected from matrix contamination.

For this Application Note, an Agilent 7000 Series Triple Quadrupole GC/MS was coupled with an Intuvo 9000 GC with an Agilent Intuvo HP5-MS UI Column. Calibration curve linearity and analyte recovery over time was evaluated for seven different food matrices.

Conclusion

A calibration and matrix evaluation was performed on an Agilent Intuvo 9000 GC equipped with an Intuvo HP5-MS UI column and an Agilent 7000 Series Triple Quadrupole GC/MS.

Twenty-one pesticides were evaluated with seven matrices to represent a range of commodities, with varying levels of difficulty. The instrument showed excellent calibration linearity and recovery. With the implementation of the Agilent Intuvo Guard Chip, the following was observed:

- The need to trim the column to maintain peak shape and recovery was eliminated.
- Retention time locking was not required.
- The source did not require cleaning throughout the entire evaluation.
- Excellent peak shape and recovery was maintained, even without backflushing.
- Replacement of the Intuvo Guard Chip did not affect retention times.

In this evaluation, calibration curve coefficients were usually 0.995 percent or better, regardless of matrix. Average recovery for a 50 ng/mL sample across 60 food extract injections was approximately 100 percent for all seven matrices. This demonstrates consistent responses over the course of a batch analysis. Peak shapes and retention times were also exceptionally consistent both before matrix, after matrix exposure, and after maintenance. By preemptively replacing the Intuvo Guard Chip after approximately 100 injections, the system was well maintained for over 500 injections with minimal intervention.

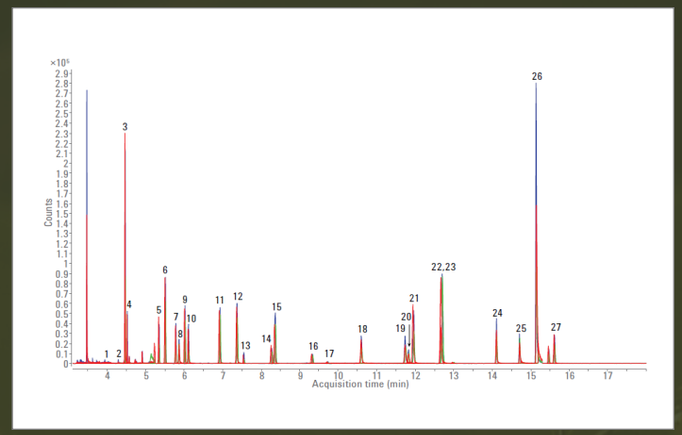

Figure 1. Overlaid chromatograms for the 50 ng/mL calibration check (blue) after 60 honey extract injections (red), and after liner and Agilent Intuvo Guard Chip replacement (green), show very good consistency.

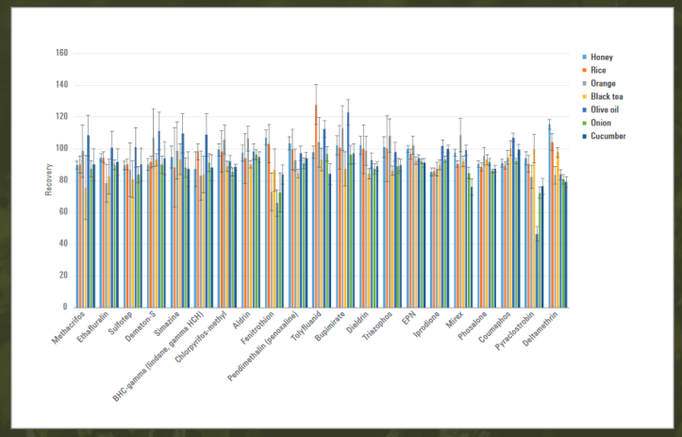

Figure 2. Average recoveries for 60 injections for seven different matrix types are nearly 100 % for a majority of the target analytes. Error bars denote the standard deviation of the measurement.

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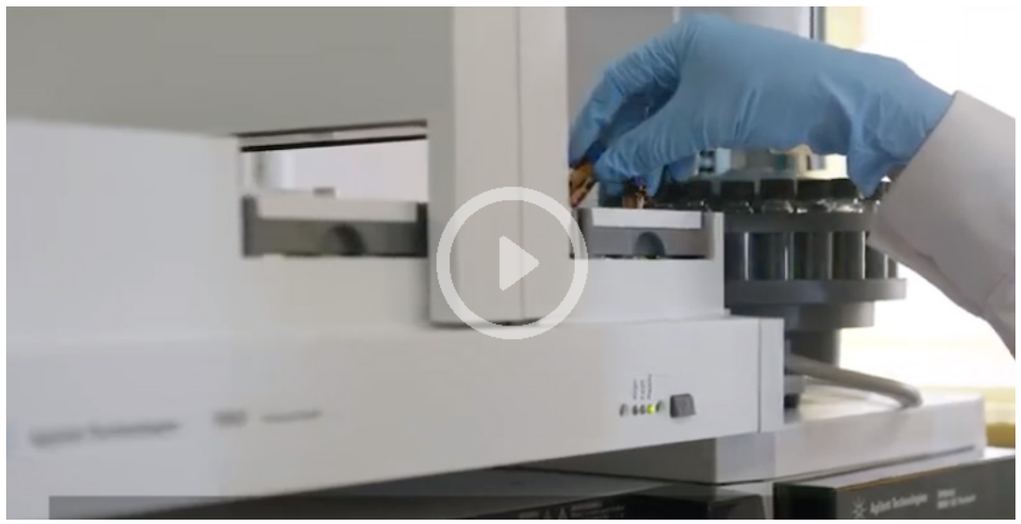

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- Reliably analyze a wide array of pesticides and environmental pollutants with a Retention Time Locked MRM database with over 1,100 compounds developed specifically for the Intuvo flowpath.
- Save time, improve productivity, and halve required bench footprint using the novel Intuvo 9000 GC.
- Eliminate complex column trimming using Intuvo click and run, ferrule-free connections.
- Extend column life with the Intuvo Guard Chip and simple-to-configure backflush.

Analyzing pesticide residues in food can quickly become complex with increasing target compound and commodity lists and decreasing detection limit requirements. Having a robust method on an easy-to-use platform that integrates seamlessly to a large database is desired to facilitate this analysis. Sometimes, however, you do not have the time or capability to develop such tools—fortunately Agilent has done the work for you!

The Agilent Pesticides & Environmental Pollutants Intuvo MRM database and optimized analytical methods for the Intuvo-GC/TQ help laboratories get on the fast track to success. Start analyzing samples immediately using provided industry-standard methods and optimized MRM transitions.

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