Zebron™
GC Columns

Sensitive, Reproducible Results for Food Testing

with Zebron GC Columns

- Easy column selection for food testing
- Explore high performance GC phases
- Get featured food testing applications

phenomenex®
...breaking with tradition℠

www.phenomenex.com/FoodGC

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You asked for a better online experience. One filled with technical resources, easy-to-find products, and useful tools to make your GC work and food testing easier. Good news...it's here!

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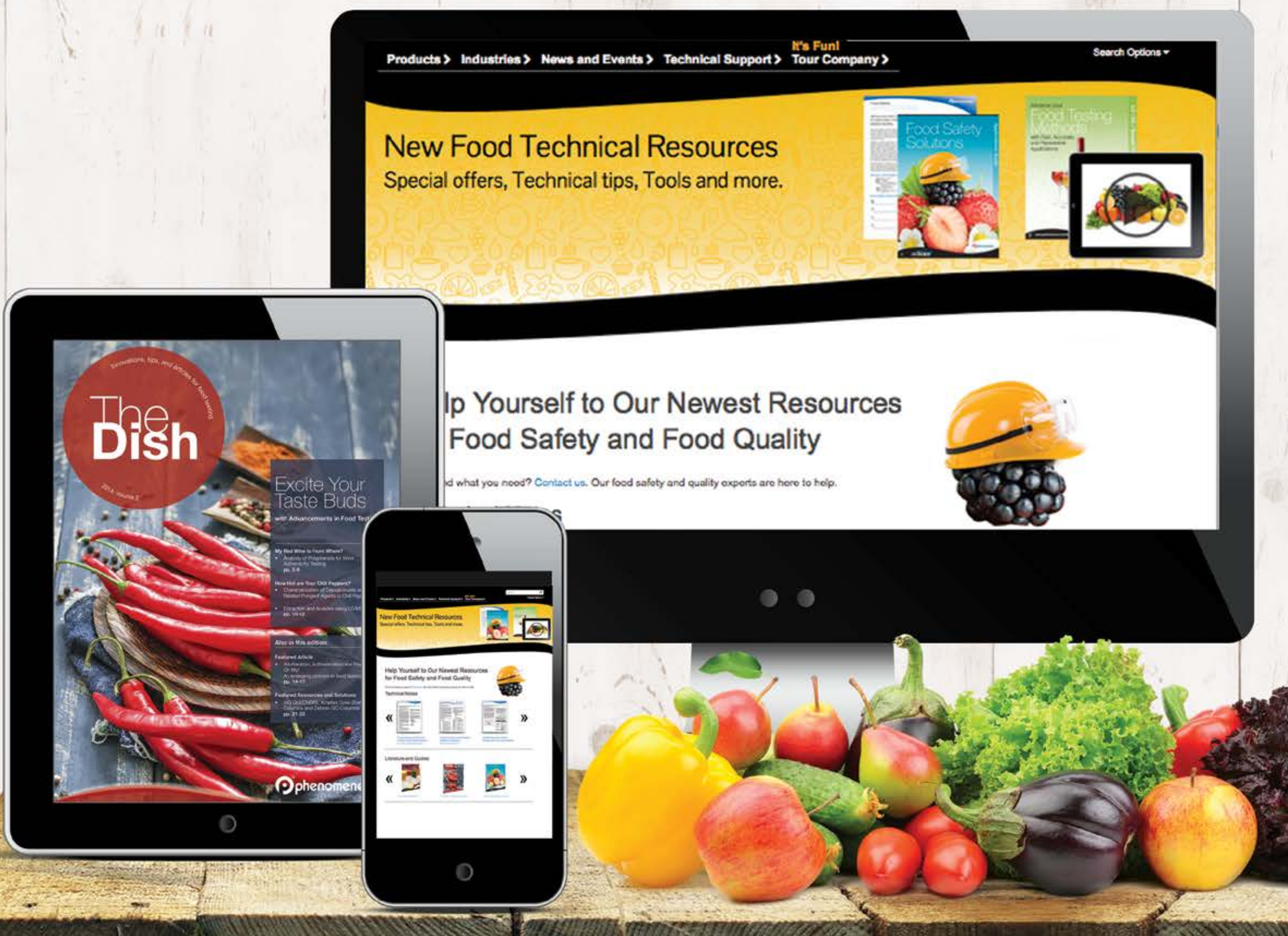
www.phenomenex.com/ResourcesGC



Better Food Testing Starts Here

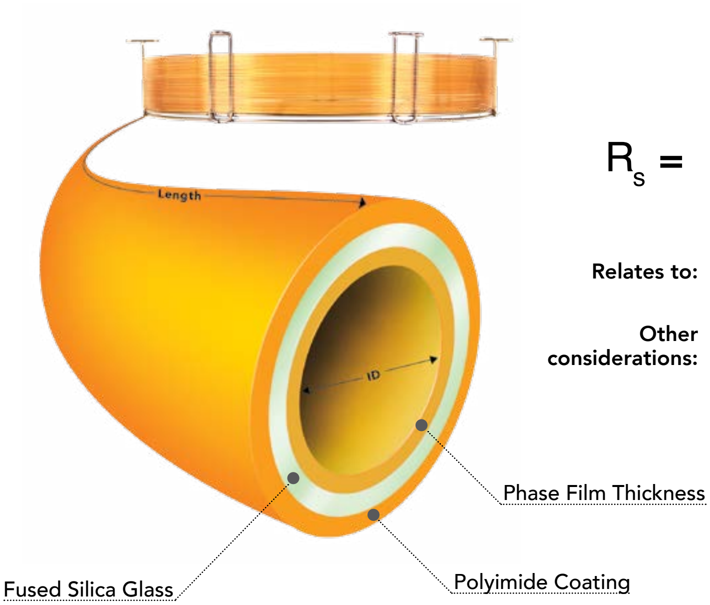
Zebron GC Columns

Serving Sensitive, Reproducible Performance Since 1998

Improve Column Selection

The Master Resolution Equation

How do you choose a column? Do you reach into a cabinet of mystery columns, look to your favorite 5 % phenyl phase, or borrow one from a colleague? Understanding how column paramaters impact key elements of the master resolution equation will help you quickly make the right column selection for successful separations.



$$R_s = \left[\frac{\sqrt{N}}{4}\right] \times \left[\frac{\alpha - 1}{\alpha}\right] \times \left[\frac{k}{k+1}\right]$$

	Efficiency Term	Selectivity Term	Retention Term
Relates to:	Column Length Column ID	Column Phase	Column ID Film Thickness
Other considerations:	Carrier Gas Linear Velocity	Temperature	Temperature

Try the NEW GC Column Finder!

Easily select a column by part number, manufacturer, industry, application or official method in under 1 minute.

www.phenomenex.com/FindGC

Selecting Your Dimensions

Length

Longer columns can improve resolution, but they will also increase run times. Under isothermal conditions, doubling column length only increases resolution by 41 %, but doubles the run time! Choose a column length that balances efficiency with acceptable run times.

Short — **Long**

15 m or less

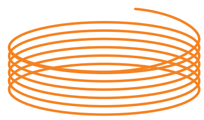
Applications
- High boilers
- GC/MS applications

Advantages
- Faster run times
- Higher temp. limits
- Lower bleed
- Higher efficiency

Disadvantages
- Less inert
- Limited retention

Good Starting Length

30 m



60 m or more

Applications
- Complex samples with closely eluting peaks
- Low boilers
- Less active samples
- Complex temperature ramps

Advantages
- Better resolution

Disadvantages
- Slow run times

Internal Diameter

Column internal diameter (ID) has a major impact on both resolution and sample capacity. Unlike column length, using smaller ID columns can actually lead to faster run times, because the column length required with a small ID is often shorter due to increased efficiency.

Narrow — **Wide**

0.10, 0.18, 0.20 mm

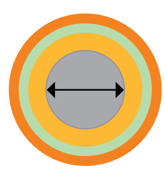
Applications
- Complex samples

Advantages
- Faster run times
- Better resolution

Disadvantages
- Lower sample capacity
- Easily overloaded

Good Starting ID

0.25 mm



0.32, 0.53 mm

Applications
- Dirty samples
- Highly concentrated samples

Advantages
- Increased sample capacity
- Increased sample

Disadvantages
- Decreased efficiency
- May need higher flow rates unsuitable for GC/MS

Film Thickness

Film thickness determines solute retention and plays an important role in column sample capacity. Thin film columns are faster and provide higher resolution, but lower sample capacity. In most instances, choose the thinnest film possible that still provides adequate retention. When working with active samples, using a slightly thicker film can significantly improve peak shape.

Thin — **Thick**

0.10, 0.18 µm

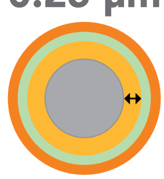
Applications
- High boilers
- GC/MS applications

Advantages
- Faster run times
- Higher temp. limits
- Lower bleed
- Higher efficiency

Disadvantages
- Less inert
- Limited retention

Good Starting Film

0.25 µm



0.50 µm or more

Applications
- Low boilers
- Gases, solvents, purgeables, volatiles
- Purity testing

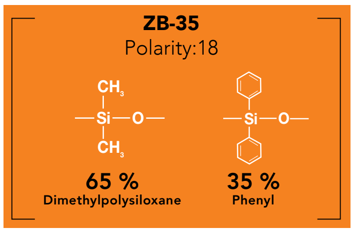
Advantages
- Better inertness
- Higher capacity
- Higher bleed

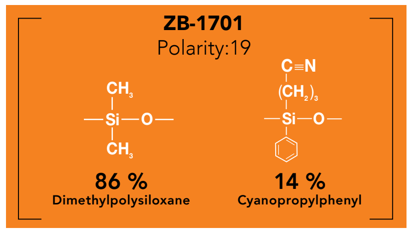
Disadvantages
- Slower run times
- Lower temp. limits

Selecting A Phase

Selectivity Has The Biggest Impact On Resolution

Resolution between two analytes is mainly determined by the selectivity of the stationary phase. By increasing resolution between two compounds, the total analysis time can often be reduced significantly. Phase polarity gives a general guideline for sample capacity and separation, which can affect peak shape and resolution. However, two columns may have similar polarity but show different separation profiles due to dissimilar selectivities. For example, ZB-35 and ZB-1701 are close in polarity, but the cyanopropyl group makes ZB-1701 different in terms of selectivity.





The 3 Most Prevalent GC Interactions

Dispersive Forces (Van der Waals Interactions)

- Weakest of all intermolecular forces and occurs between non-polar compounds
- Separation is based on boiling point (classic example – hydrocarbon separation in SimDist analysis)

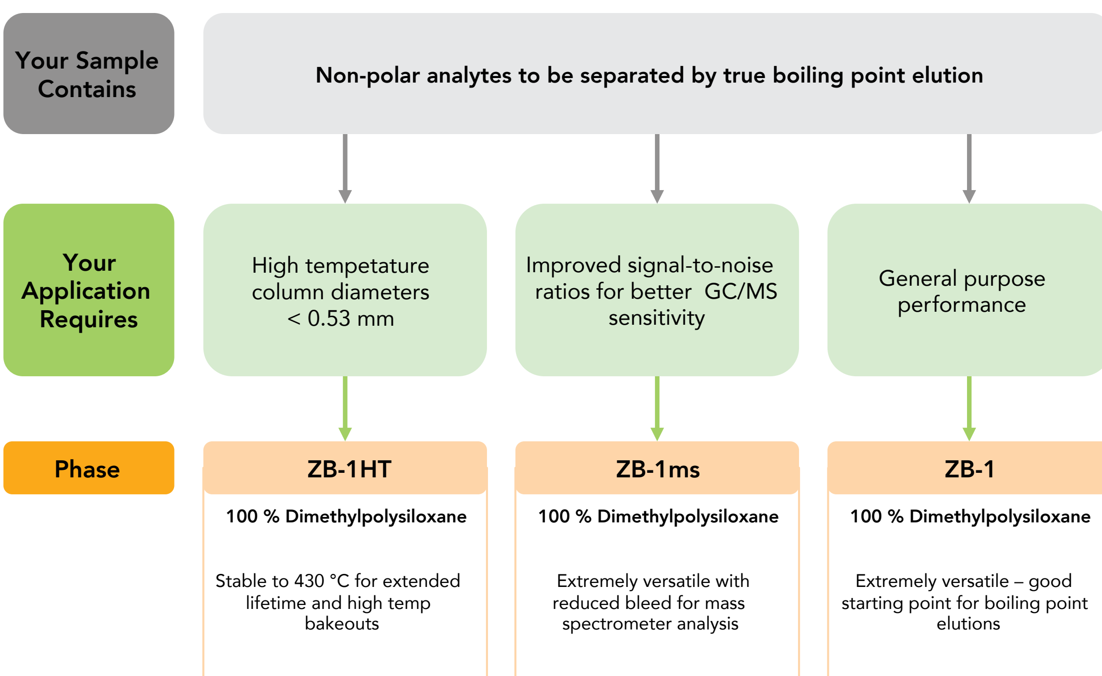
Dipole-Dipole Interactions

- Either permanently present or induced by analyte-stationary phase interactions
- Higher dipole-dipole interaction can help separate compounds with similar boiling points, but different chemical structures

Hydrogen Bonding (Acid-Base Interactions)

- Can cause poor peak shape or irreversible binding to the inlet liner or to the column itself
- Zebron columns are specially deactivated to minimize these interactions

Choosing A "1" Phase (e.g. ZB-1)



Selecting A Phase

Choosing A "5" Phase (e.g. ZB-5)

Your Sample Contains	A wide range of functional groups		Closely eluting aromatic analytes		Acids, Amines, Bases
Your Application Requires	High temp stability	0.53 mm ID or film > 0.50 µm	Versatility for multiple analyses	Good results for active compounds (food / environmental matrices)	Good results for active compounds (pharmaceutical/ toxicology matrices)
Phase	**ZB-5HT**	**ZB-5**	**ZB-5ms**	**ZB-SemiVolatiles**	**ZB-5MSi**
	5 % Phenyl	**5 % Phenyl**	**5 % Phenyl-Arylene**	**5 % Phenyl-Arylene**	**5 % Phenyl**
	Stable to 430 °C for extended lifetime and high temp bakeouts	Extremely versatile – good starting point for a typical 5 phase	Extremely versatile with better selectivity for aromatics	Highly inert with equivalent selectivity to ZB-5ms; recommended for SVOCs, PBDEs, and active analytes	Highly inert with equivalent selectivity to ZB-5; recommended for drug screens and active analytes

Note: "Closely eluting aromatic analytes" leads to both ZB-5ms and ZB-SemiVolatiles. "Acids, Amines, Bases" leads to both ZB-SemiVolatiles and ZB-5MSi.

Choosing A "PEG" Phase (e.g. ZB-WAX)

Your Sample Contains	FAMEs, Flavors, Essential Oils, Solvents, or BTEX Compounds		Free Fatty Acids or Organic Acids
Your Application Requires	Aqueous stability for polar compounds; low temperature volatiles	Improved signal-to-noise ratio for better GC/MS sensitivity	Efficiency and inertness for acidic analytes
Phase	**ZB-WAXPLUS™**	**ZB-WAX**	**ZB-FFAP**
	Polyethylene Glycol	**Polyethylene Glycol**	**Nitroterephthalic Acid Modified Polyethylene Glycol**
	100 % aqueous stability with excellent separations of polar complex mixtures; widely used for profiling and "fingerprinting"	General-purpose PEG phase for polar food samples	Excellent peak shape for underivatized acids, organic acids, free fatty acids, and alcohols

GC Column Selection by Application

Food Safety

Compound Class	Analysis	Recommended Columns
Pesticides & Antimicrobials	Multi-Residue Pesticide Screening	ZB-MultiResidue™-1 and 2
	Organochlorine Pesticides in Water	ZB-MultiResidue-1 and 2
	Organochlorine Pesticides in Foods of Plant Origin	ZB-MultiResidue-1 and 2
	Organophosphorus Pesticides in Foods of Plant Origin	ZB-MultiResidue-1 and 2
	Triazine Pesticides in Water	ZB-50
	Triazine Pesticides in Foods of Plant Origin	ZB-50
	Chloramphenicol in Foods of Animal Origin	ZB-1ms
Environmental Contaminants	Polybrominated Diphenyl Ethers (PBDEs) in Food	ZB-SemiVolatiles or ZB-35
	Polychlorinated Biphenyls (PCBs) in Water	ZB-MultiResidue-1 or ZB-XLB-HT Inferno™
	Polychlorinated Dibenzo-dioxins (PCDDs) in Food	ZB-5ms
	Polychlorinated Dibenzo-furans (PCDFs) in Food	ZB-5ms
	Polycyclic Aromatic Hydrocarbons (PAHs) in Water	ZB-SemiVolatiles or ZB-35
Food Contact Materials	Food Packaging Volatiles	ZB-624
	Melamine in Food	ZB-XLB-HT Inferno
	Cyanuric Acid in Food	ZB-XLB-HT Inferno
	Phthalates in Food	ZB-5ms
	Residual Solvents in Food	ZB-624 or ZB-WAXPLUS™
	Bisphenol A & F (BPA/BPF) in Food	ZB-5ms
Additives & Preservatives	Parabens in Food	ZB-5ms
	Chloropropanols (3-MCPD) in Food	ZB-5ms
	Flavor Additives (Borneol)	ZB-MultiResidue-1
	Phenolic Antioxidants (BHA & BHT) in Food	ZB-50
	Tocopherols in Food	ZB-5
Process Contaminants	Acrylamide in Foods	ZB-5HT Inferno
	Acrylamide, Acrylonitrile, and Acrolein in Water	ZB-624
	Benzene in Food	ZB-WAXPLUS
	Glycols in Food	ZB-WAXPLUS
Hormones	Steroid Hormones in Food	ZB-5ms or ZB-1ms

Food Quality

Compound Class	Analysis	Recommended Columns
Fatty Acids & FAMEs	Food Industry Fatty Acid Methyl Esters (FAMEs)	ZB-WAXPLUS
	Marine Oil Fatty Acid Methyl Esters (FAMEs)	ZB-WAXPLUS
	Saw Palmetto Fatty Acid Methyl Esters (FAMEs)	ZB-WAXPLUS
	Free Fatty Acids	ZB-FFAP
	Essential Fatty Acids (EFAs) Omega-3 and Omega-6	ZB-WAXPLUS
Triglycerides	Butter Triglycerides	ZB-5HT Inferno
	Canola Oil Triglycerides	ZB-5HT Inferno
	Olive Oil Triglycerides	ZB-5HT Inferno
	Peanut Oil Triglycerides	ZB-5HT Inferno
Alcoholic Beverages	Cognac Compounds	ZB-WAXPLUS
	Distilled Liquor Screen	ZB-FFAP
	Ethanol in Beer	ZB-Bioethanol
	Sulfur in Beer	ZB-1ms
	Whiskey Compounds	ZB-WAXPLUS
	Wine Compounds	ZB-WAX or ZB-WAXPLUS
Other Acids	Organic Acids	ZB-FFAP
	Amino Acids	ZB-50
Sterols	Sterols in Lard	ZB-5HT Inferno
	Sterols in Margarine	ZB-5HT Inferno
	Sterols in Olive Oil	ZB-5HT Inferno
	Sterols in Peanut Butter	ZB-5HT Inferno
Sugars	Alditol Acetates	ZB-5ms
	Trimethylsilyl (TMS) Sugars	ZB-MultiResidue-1

Flavors & Fragrances

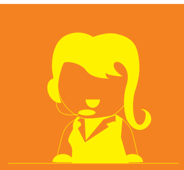
Compound Class	Analysis	Recommended Columns
Essential Oils	Cold-Pressed Orange Oil	ZB-WAXPLUS
	Ginkgo Biloba Oil	ZB-1ms
	Lavender Oil	ZB-1ms
	Peppermint Oil	ZB-WAX
	Rose Oil	ZB-XLB
	Spearmint Oil	ZB-5ms
	Ylang Ylang Oil	ZB-1ms
Flavors	Flavors Screening	ZB-FFAP
	Flavor Allergens	ZB-5ms
	Flavor Volatiles	ZB-1ms, ZB-WAXPLUS, or ZB-624
	Alcoholic Beverage Profile	ZB-FFAP
	Honey Profile	ZB-WAXPLUS
Fragrances	Fragrance Screening	ZB-WAXPLUS or ZB-624
	Fragrance Allergens	ZB-1ms

GC Column Selection by Manufacturer

Upgrade to Zebron™! Our commitment to quality and innovation is what makes Zebron GC columns well suited for any application. Performance is GUARANTEED*.

	Zebron Phase	Zebron Composition	Restek®	Agilent®	Supelco®	Alltech®	SGE®	OV®
	ZB-1	100% Dimethylpolysiloxane	Rtx®-1, Rtx-1PONA, Rtx-1 F&F	DB®-1, DB-2887, DB-1 EVDX, HP®-1, HP-101, HP-PONA, Ultra 1, CP-Sil 5 CB	SPB®-1, SPB-1 TG, SE-30, MET-1, SPB-1 Sulfur, SPB-HAP	AT-1, AT-Sulfur, EC-1	BP1, BP1-PONA, BPX1-SimD	OV-1
Featured on p. 11	ZB-1ms	100% Dimethylpolysiloxane	Rtx-1ms	DB-1ms, HP-1ms, CP-Sil 5 CB MS, VF-1ms	MDN®-1, Equity®-1	AT-1ms	SolGel-1ms™	
	ZB-1HT Inferno™	100% Dimethylpolysiloxane	Rxi®-1HT	DB-1ht, CP-SimDist	Petrocol 2887			
	ZB-1XT SimDist	100% Dimethylpolysiloxane	MXT®-1HT SimDist	CP-SimDist UltiMetal DB-HT SimDis				
	ZB-5	5% Phenyl 95% Dimethylpolysiloxane	Rtx-5	DB-5, HP-5, Ultra 2, HP-PAS-5, CP-Sil 8 CB	MDN-5, SPB-5, PTE-5, SE-54, PTA-5, Equity-5, Sac-5	AT-5, EC-5	BP5, BPX5	OV-5
	ZB-5MSi	5% Phenyl 95% Dimethylpolysiloxane	Rtx-5ms, Rxi-5ms, Rtx-5Amine	DB-5, HP-5ms, HP-5msi, HP-5ms Ultra Inert	MDN-5S			
Featured on p. 13	ZB-5HT Inferno	5% Phenyl 95% Dimethylpolysiloxane	Stx®-5HT, Rxi-5HT, XTI®-5HT, Rtx-5HT	DB-5ht, VF-5ht	HT-5			
	ZB-5ms	5% Phenyl-Arylene 95% Dimethylpolysiloxane	Rtx-5Sil MS, Rxi-5Sil MS	DB-5ms, DB-5.625, DB-5ms EVDX, VF-5ms, CP-Sil 8 CB MS				
Featured on p. 12	ZB-SemiVolatiles	5% Phenyl-Arylene 95% Dimethylpolysiloxane	Rxi-5Sil MS Rxi-5ms	DB-5ms Ultra Inert HP-5ms Ultra Inert	SLB®-5ms			
	ZB-35	35% Phenyl 65% Dimethylpolysiloxane	Rtx-35, Rtx-35ms	DB-35, DB-35ms, HP-35, HP-35ms	MDN-35, SPB-35, SPB-608	AT-35	BPX35, BPX608	OV-11
	ZB-35HT Inferno	35% Phenyl 65% Dimethylpolysiloxane			Phenomenex Exclusive			
	ZB-50	50% Phenyl 50% Dimethylpolysiloxane	Rtx-50	DB-17, DB-17HT, DB-17ms, DB-17 EVDX, HP-50+, CP-Sil 24 CB	SP-2250, SPB-17, SPB-50	AT-50	BPX50	OV-17
	ZB-624	6% Cyanopropylphenyl 94% Dimethylpolysiloxane	Rtx-1301, Rtx-624	DB-1301, DB-624, DB-VRX, HP-VOC, CP-1301, CP-Select 624 CB	SPB-1301, SPB-624	AT-624, AT-1301	BP624	OV-624
	ZB-1701	14% Cyanopropylphenyl 86% Dimethylpolysiloxane	Rtx-1701	DB-1701 , CP-Sil 19 CB	SPB-1701, Equity-1701	AT-1701	BP10	OV-1701
	ZB-1701P	14% Cyanopropylphenyl 86% Dimethylpolysiloxane		DB-1701P				
	ZB-WAX	Polyethylene glycol	Rtx-WAX, Famewax, Stabilwax-DB	DB-WAXetr, HP-INNOWax, CP-Wax 57 CB	Met-Wax, Omegawax	EC-Wax	SolGel-WAX™	
Featured on p. 15	ZB-WAXplus™	Polyethylene glycol	Stabilwax®	DB-WAX, CAM, HP-20M, Carbowax 20M, CP-Wax 52 CB	SUPELCOWAX® 10	AT-Wax, AT-AquaWax	BP20	Car-bowax 20M
	ZB-FFAP	Nitroterephthalic acid modified polyethylene glycol	Stabilwax-DA	DB-FFAP, HP-FFAP, CP-Wax 58 (FFAP) CB, CP-FFAP CB	Nukol, SPB-1000	AT-1000, EC-1000	BP21	OV-351
	ZB-CLPesticides-1	Proprietary	Rtx-CLPesticides, Stx-CLPesticides					
	ZB-CLPesticides-2	Proprietary	Rtx-CLPesticides2, Stx-CLPesticides2					
Featured on p. 14	ZB-MultiResidue™-1	Proprietary	Rtx-CLPesticides, Stx-CLPesticides					
	ZB-MultiResidue-2	Proprietary	Rtx-CLPesticides2, Stx-CLPesticides2					
	ZB-XLB	Proprietary	Rtx-XLB	DB-XLB, VF-XMS	MDN-12			
	ZB-XLB-HT Inferno	Proprietary			Phenomenex Exclusive			
	ZB-Drug-1	Proprietary			Phenomenex Exclusive			
	ZB-BAC-1	Proprietary	Rtx-BAC1	DB-ALC1				
	ZB-BAC-2	Proprietary	Rtx-BAC2	DB-ALC2				
	ZB-Bioethanol	Proprietary			Phenomenex Exclusive			

This section is, neither in terms of manufacturers nor in terms of their products, a complete list, and the accuracy of the data is not guaranteed. Small differences in dimensions or performance might be possible and slight adjustments to your application may be necessary.
*See p. 10



Looking For Another Phase?

We'll cross-reference your current column for you! Contact your local GC Specialist for additional information or visit **www.phenomenex.com/GC**

Explore Zebron™ Phases for Food Testing

Guaranteed Quality by Design

Our GC R&D and production team has on average 25+ years of GC experience, and many spent years creating keystone phases at J&W Scientific prior to joining the Phenomenex team. This expertise means Zebron products are designed to work out-of-the-box, headache free. We guarantee it.

- Stringent individual QC testing – no batch tests
- Excellent sensitivity and high temperature stability
- MS certified phases for low bleed
- Novel selectivities for better separations

Better Quality, Less Maintenance

“After most runs with Zebron, I didn't have to clean or trim the column. The separation was the same, but the Zebron lasted about 4-5 times longer, which for me means saving at least $5,000 a year.”

Sam Sabella
BASF USA

guarantee

If Zebron columns do not provide you with equivalent or better separations as compared to any other GC column of the same phase and comparable dimensions, send in your comparative data within 45 days and keep the column for FREE!

We're YouTube Stars!

We're so committed to quality, we made a video about it! Watch it at **www.phenomenex.com/InnovateGC**

ZB-1ms

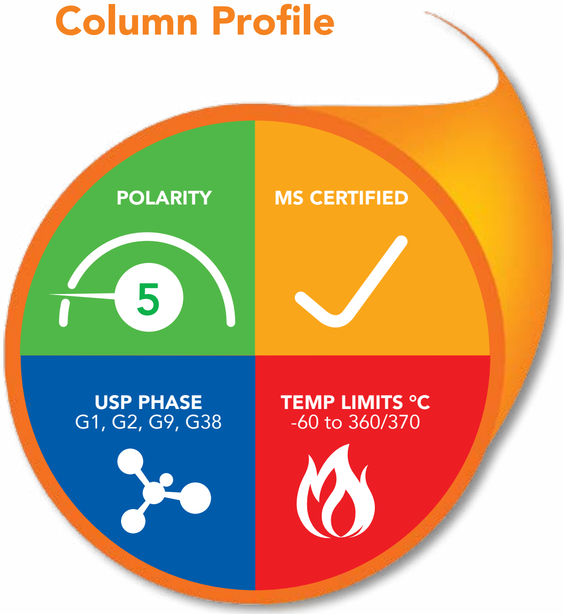
The Ultimate MS Certified "1" Phase

- Very low bleed phase especially suited to high sensitivity GC/MS
- Extremely inert for active compounds such as pesticides, or acids and bases
- Improved signal-to-noise ratio for better sensitivity and mass spectral integrity
- Identical selectivity to ZB-1

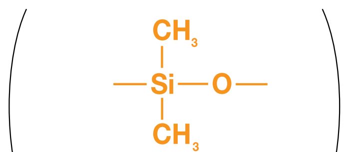
Upgrade to Zebron™ from any 100 % dimethylpolysiloxane phase:

Agilent®	Alltech	Restek®	SGE	Supelco
DB®-1	AT-1	Rtx®-1	BP1	SPB-1
DB-1ms	AT-1ms	Rtx-1ms	SolGel-1ms™	SE-30
DB-1ms Ultra Inert	EC-1	Rxi®-1ms		MET-1
HP-1				MDN-1
HP®-1ms				Equity®-1
HP-1ms Ultra Inert				
VF-1ms				
CP-Sil 5 CB				
Ultra 1				

Column Profile



Phase Chemistry

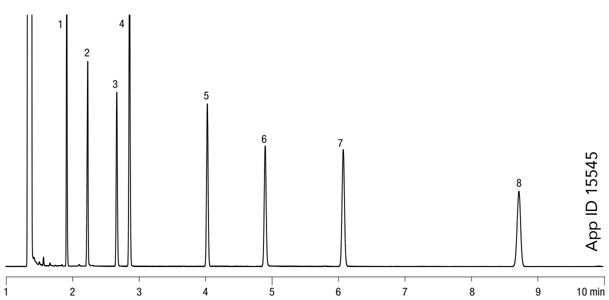


100 % Dimethylpolysiloxane

Lower Overall Column Activity

Activity is a key measure of column quality. Zebron ZB-1ms columns are aggressively tested to ensure full deactivation. Below is an example of the stringent QC test mix we use, notice the low tailing for even the most active compounds like 2-Ethylhexanoic Acid!

Test Conditions for Zebron ZB-1ms



Column: Zebron ZB-1ms
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G011-11
Injection: Split @ 250 °C, 1.0 µL
Carrier Gas: Hydrogen @ 1.18 mL/min (constant flow)
Oven Program: 140 °C (Isothermal)
Detector: FID @ 325 °C
Sample:
1. Decane
2. 2-Ethylhexanoic Acid
3. 4-Chlorophenol
4. Naphthalene
5. Tridecane
6. 1-Undecanol
7. Dicyclohexylamine
8. Pentadecane

Recommended Applications

- Acids
- Amines
- Essential Oils
- Flavors
- Fragrances & Fragrance Allergens
- Oxygenates
- PCBs
- Pesticides
- Solvent Impurities
- Sulfur Compounds (Light)

ZB-SemiVolatiles

Get Best-In-Class Performance

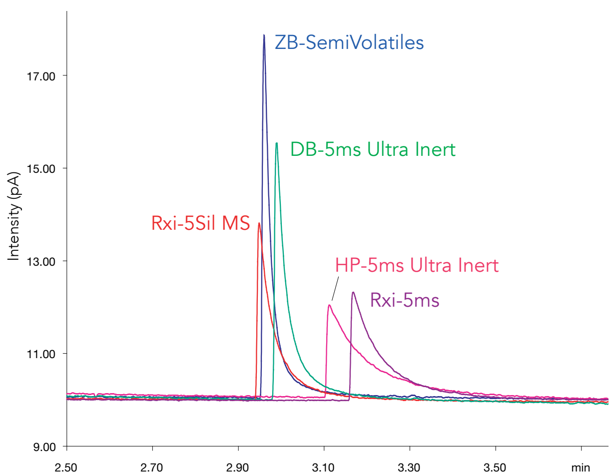
- Specifically designed to overcome obstacles for sensitive methods
- Enviro-Inert™ Technology provides improve inertness without compromising selectivity
- Our column of choice for acids, bases, amines, and other active compounds

Upgrade to Zebron™ from any 5 % phenyl or 5 % phenyl-arylene / 95 % dimethylpolysiloxane phase:

Agilent®		Restek®	Supelco
DB®-5ms	HP®-5ms	Rxi®-5Sil MS	SLB®-5ms
DB-5ms Ultra Inert	HP-5ms Ultra Inert	Rxi-5ms	
DB-5.625	VF-5ms	Rxi-1ms	
	CP-Sil 8 CB MS		

Improve Inertness for Active Compounds

Pyridine is a very active amine and a good indicator for both column lifetime and sensitivity. Columns with higher initial peak responses can be expected to maintain performance over time. Higher responses also allow you to run at lower levels of detection, improving sensitivity.

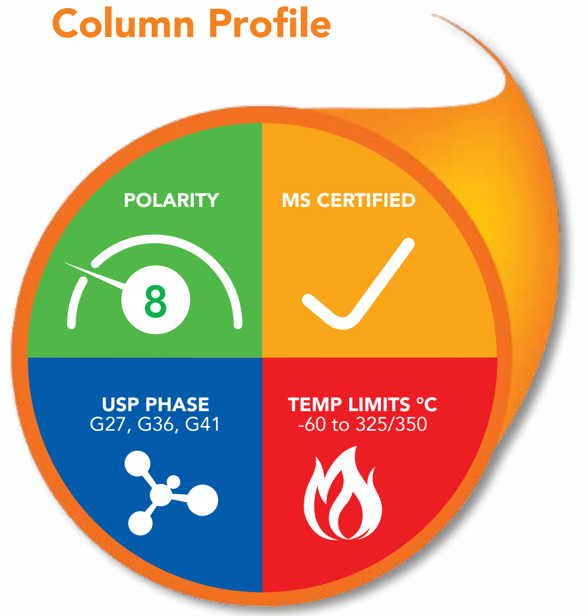


Customer Approved!

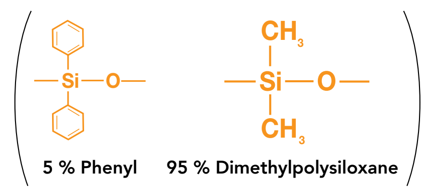
"I have found the Phenomenex ZB-SemiVolatiles columns to be superior in quality and durability than any other columns we have previously used. The columns not only last longer, but the reproducibility of column is extraordinary. The column holds calibrations particularly well, even after multiple injections of samples with far less than desirable matrices. All of this equates to less downtime and maintenance and more productivity for TestAmerica."

Ryan McKernan, GC/MS Semi-Volatile Analyst
TestAmerica Laboratories, Inc. Buffalo

Column Profile



Phase Chemistry



Recommended Applications

- Semivolatiles (SVOCs)
- Polycyclic Aromatic Hydrocarbons (PAHs)
- Polybrominated Diphenyl Ethers (PBDEs)
- Active Compounds, Acids, Bases

Comparative separations may not be representative of all applications.

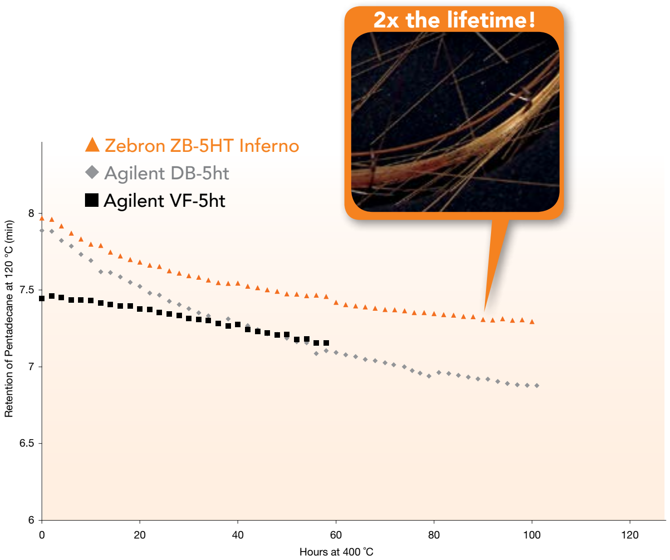
ZB-5HT Inferno™

Robust Performance Up To 430 °C

- First non-metal columns stable to 430 °C
- Robust column well suited for analysts struggling with high boilers, contaminants, or carryovers
- Longer lifetime with rugged high temperature, polyimide coated, fused silica tubing
- Low activity, provides good peak shape for acidic and basic samples
- Individually tested for low bleed, MS certified

Upgrade to Zebron™ from any 5 % phenyl / 95 % dimethylpolysiloxane phase:

Agilent®	Alltech	Restek®	SGE	Supelco
DB®-5ht	AT-5	Stx®-5HT	BP5	HT-5
DB-5	EC-5	XTI®-5HT	BPX5	
HP-5		Rtx®-5		
VF-5ht		Rxi®-5HT		



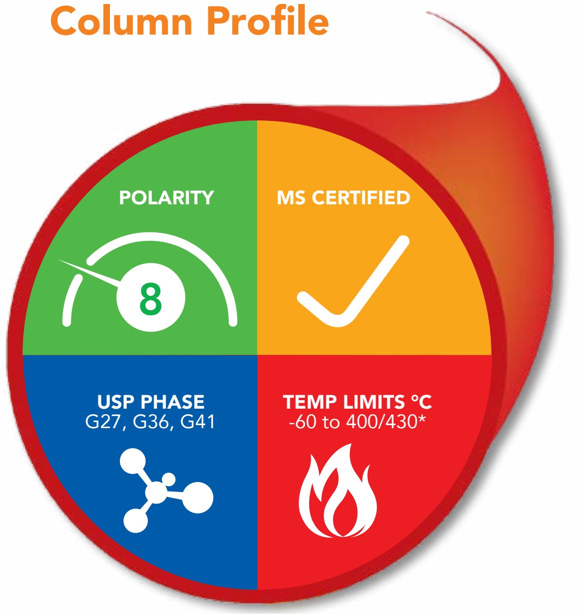
Conditions for all columns:
Dimensions: 30 meter x 0.25 mm x 0.10 µm
Injection: 1.0 µL of test mix AG0-7578
Carrier Gas: Helium @ 1.9 mL/min (constant flow)
Oven Program: 120 °C (Isothermal)
Detector: Flame Ionization Detector (FID) @ 400 °C
Sample: Pentadecane

How does the lifetime test work?

For the test, all columns were held at 400 °C for 2 hours and then the oven was lowered to 120 °C for Pentadecane analysis. Pentadecane retention between Zebron ZB-5HT Inferno and other traditional 5 % Phenyl 95 % dimethylpolysiloxane column was compared. The VF-5ht column died around 40 hours at 400 °C whereas ZB-5HT Inferno maintained great retention of pentadecane over 100 hours — over 2X the lifetime!

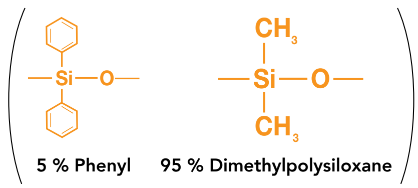
Comparative separations may not be representative of all applications.

Column Profile



* 0.53 mm ID columns are rated to 400 °C.

Phase Chemistry



Recommended Applications

- Dirty or Highly Contaminated Samples
- High Boiling Compounds
- High Molecular Weight Waxes
- Hydrocarbon Separations
- Polymers/Plastics
- Sterols
- Triglycerides

ZB-MultiResidue™-1 & -2

Optimized Results for Pesticides

- Proprietary phases specially designed for the separation of all types of pesticides, herbicides, and insecticides
- Reliable performance for multi-residue screens by GC/MS
- Low activity, decreased breakdown of sensitive pesticides such as DDT
- Provides robust column performance for high temperature bake outs

Upgrade to Zebron™ from these similar* phases:

Agilent®	Restek®
DB®-CLP1	Rtx®-CLPesticides
DB-CLP2	Rtx-CLPesticides2
	Stx®-CLPesticides
	Stx-CLPesticides2

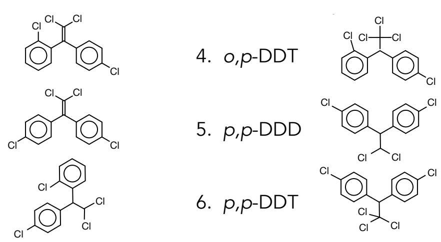
*not exact equivalent, selectivity might be different

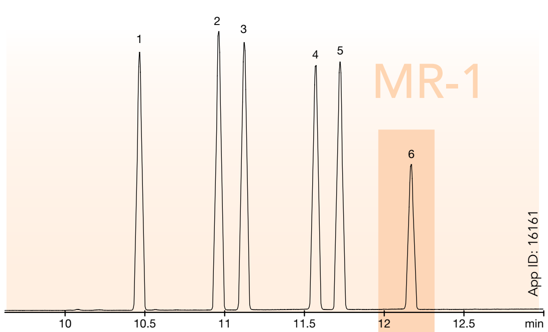
Perform Challenging Separations

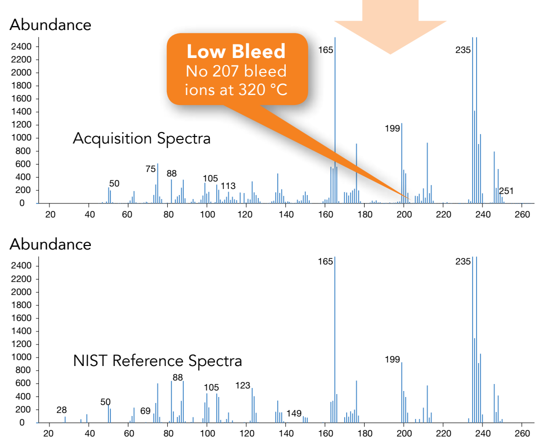
DDT, DDD, and DDE Isomer Separation at 25 ng On-Column

Sample:

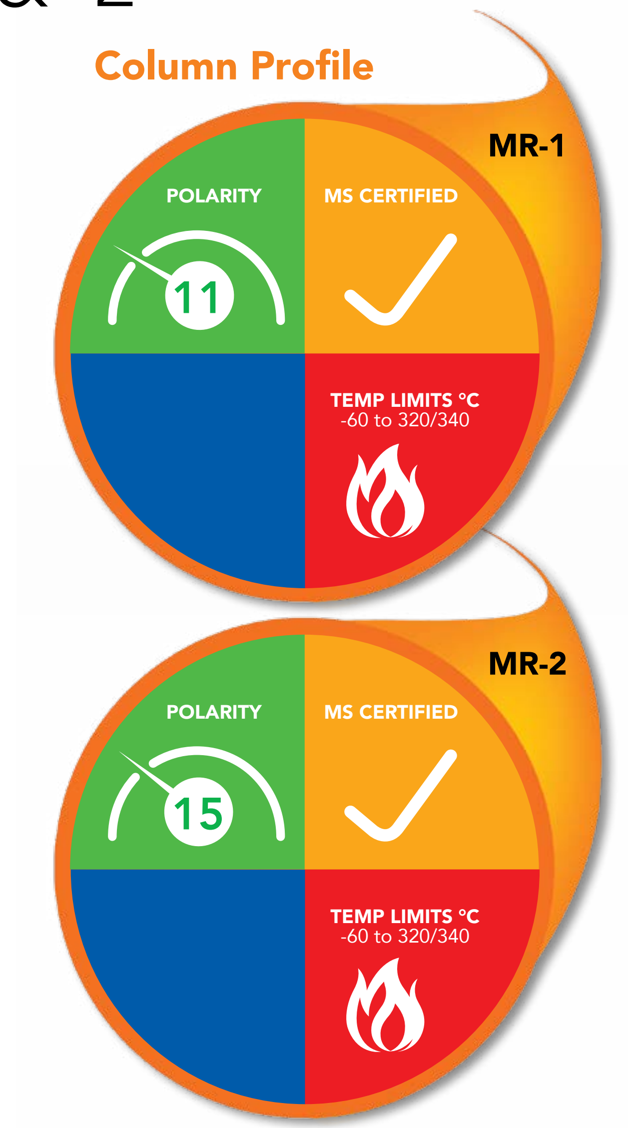
1. *o,p*-DDE
2. *p,p*-DDE
3. *o,p*-DDD
4. *o,p*-DDT
5. *p,p*-DDD
6. *p,p*-DDT



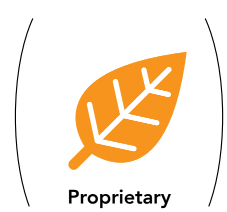




Column Profile



Phase Chemistry



Recommended Applications

- Haloacetic Acids (HAAs)
- Herbicides / Insecticides
- Multi-Pesticide Screening
- Nitrogen Containing Pesticides
- Organochlorine Pesticides
- Organophosphorous Pesticides
- PCBs / Aroclors

ZB-WAX*PLUS*™

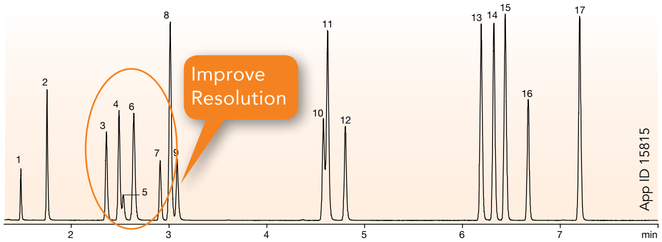
100 % Aqueous Stability for Polar Compounds

- Excellent for water samples
- Extremely inert for acidic compounds
- Enhanced selectivity for low boiling solvents
- High retention of alcohols and chlorinated solvents

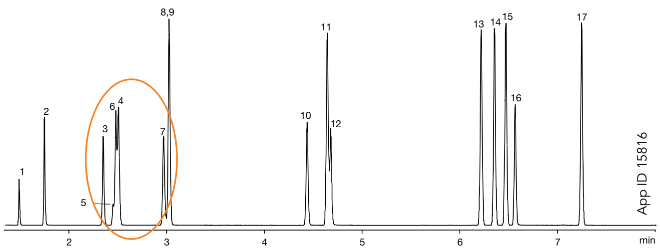
Upgrade to Zebron™ from any polyethylene glycol phase:

Agilent®	Alltech	Restek®	SGE	Supelco
DB®-WAX	AT-Wax	Stabilwax®	BP20	SUPELCOWAX® 10
CAM	AT-AquaWax			
HP-20M				
Carbowax 20M				
CP-Wax 52 CB				

Zebron ZB-WAX*PLUS*



Restek Stabilwax

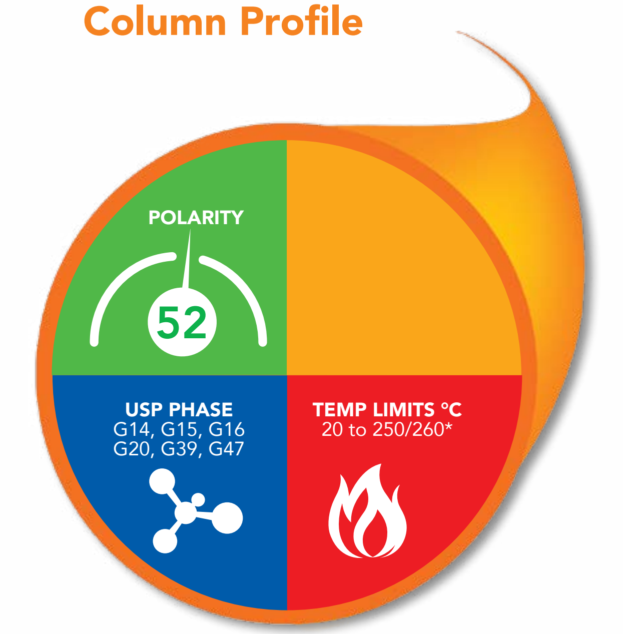


Conditions same for both columns:

Dimensions: 30 meter x 0.25 mm x 0.25 µm
Injection: Split 100:1 @ 250 °C, 1 µL
Carrier Gas: Hydrogen @ 1.0 mL/min (constant flow)
Oven Program: 5 °C for 2.5 min to 85 °C at 10 °C/min and hold until last peak elutes
Detector: FID @ 225 °C
Sample:
1. Methyl Formate
2. Acetone
3. Ethyl Acetate
4. Methyl Ethyl Ketone
5. Methanol
6. 2-Methyl-2-propanol
7. Methylene Chloride
8. Benzene
9. Ethanol
10. 2-Butanol
11. Toluene
12. n-Propanol
13. Ethyl Benzene
14. p-Xylene
15. m-Xylene
16. 1-Butanol
17. o-Xylene

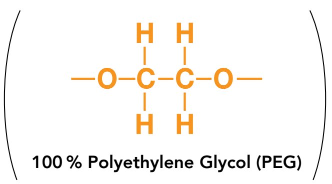
Comparative separations may not be representative of all applications.

Column Profile



* Thicker films (≥ 1.0 µm) are rated to 230/240 °C.

Phase Chemistry



Recommended Applications

- Alcohols & Alcoholic Beverages
- Aldehydes
- Aromatics
- Essential Oils
- Fatty Acid Methyl Esters (FAMEs)
- Flavors & Fragrances
- Glycols
- OVIs
- Solvents / Residual Solvents

Food Quality & Flavors

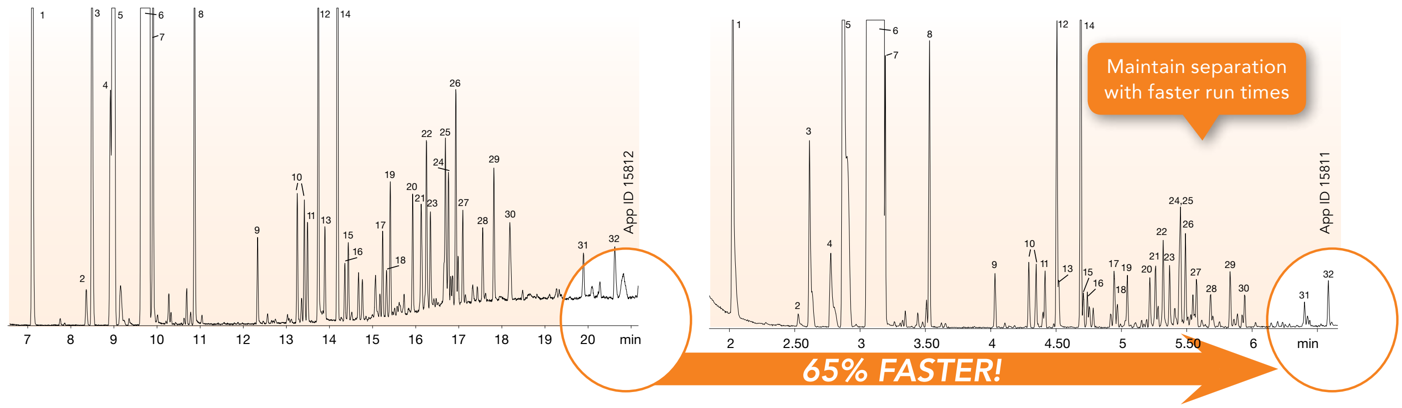
Featured Applications

Flavors

Essential Oils

Essential oils are fragrant plant essences primarily composed of terpenes, their derivatives, and other aromatic compounds. Variation in plant location and growing conditions produces natural differences in essential oil components, and due to their high price, premium oils are subject to adulteration with cheaper terpenes or poorer quality oils. Characterization of essential oils is therefore necessary, but testing is complex due to the number of compounds and their trace level presence. Runs under seven minutes can be achieved using efficient column dimensions, as demonstrated below.

Cold-Pressed Orange Oil by GC/MS



Column: ZB-WAXPLUS
Dimensions: 60 meter x 0.25 mm x 0.25 µm
Part No.: 7KG-G013-11
Injection: Split 40:1 @ 220 °C, 0.1 µL
Carrier Gas: Helium @ 1.2 mL/min (constant flow)
Oven Program: 40 °C for 0.2 min to 210 °C @ 10 °C/min for 10 min
Detector: MSD; 45-450 amu

Column: ZB-WAXPLUS
Dimensions: 10 meter x 0.10 mm x 0.10 µm
Part No.: 7CB-G013-02
Injection: Split 20:1 @ 220 °C, 0.2 µL
Carrier Gas: Helium @ 0.3 mL/min (constant flow)
Oven Program: 35 °C for 1 min to 250 °C @ 30 °C/min for 5 min
Detector: MSD; 45-450 amu

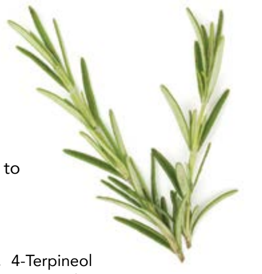
Sample:

1. α-Pinene
2. β-Pinene
3. Sabinene
4. 3-Carene
5. β-Myrcene
6. Limonene
7. β-Phellandrene
8. Octanal
9. Nonanal
10. Limonene Oxides
11. Citronellal
12. Decanal
13. α-Cubebene
14. Linalool
15. β-Cubebene
16. Octanol
17. Germacrene
18. Caryophyllene
19. trans-p-Mentha-2,8-dienol
20. cis-p-Mentha-2,8-dienol
21. Gerenial
22. α-Terpineol
23. Dodecanal
24. Valencene
25. Citral
26. Carvone
27. Cadinene
28. Perillaldehyde
29. trans-Carveol
30. cis-Carveol
31. Perillol
32. Octanoic acid

Flavors

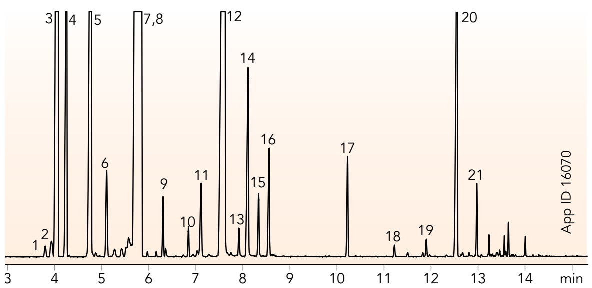
Essential Oils

Rosemary Oil by GC/MS



Column: Zebron™ ZB-1ms
Dimensions: 10 meter x 0.10 mm x 0.10 µm
Part No.: 7CB-G011-02
Injection: Split 120:1 @ 160 °C, 0.2 µL
Carrier Gas: Helium @ 0.4 mL/min (constant flow)
Oven Program: 45 °C for 2 min to 130 °C @ 8 °C/min to 200 °C @ 30 °C/min for 2 min
Detector: MSD; 18-400 amu
Sample: Sample was 10 % in dichloromethane

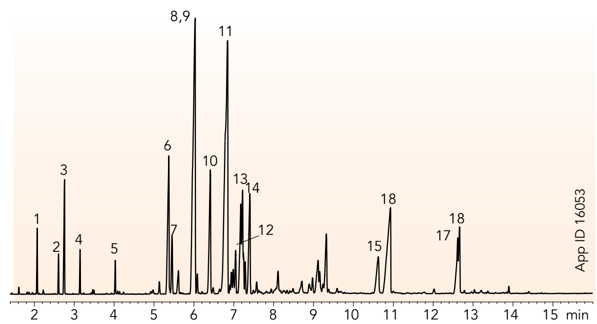
1. Tricyclene
2. α-Thujene
3. α-Pinene
4. Camphene
5. β-Pinene
6. β-Myrcene
7. Eucalyptol
8. Limonene
9. γ-Terpinene
10. Terpinolene
11. Linalool
12. Camphor
13. Isoborneol
14. Borneol
15. 4-Terpineol
16. Terpineol
17. Bornyl Acetate
18. Eugenol
19. Copaene
20. Caryophyllene
21. α-Caryophyllene



Ylang Ylang Oil by GC/MS

Column: Zebron ZB-1ms
Dimensions: 10 meter x 0.10 mm x 0.10 µm
Part No.: 7CB-G011-02
Injection: Split 120:1 @ 160 °C, 0.2 µL
Carrier Gas: Helium @ 0.5 mL/min (constant flow)
Oven Program: 60 °C to 120 °C @ 15 °C/min to 160 °C @ 5 °C/min to 220 °C @ 20 °C/min
Detector: MSD; 18-400 amu
Sample: Oil was 10 % in dichloromethane

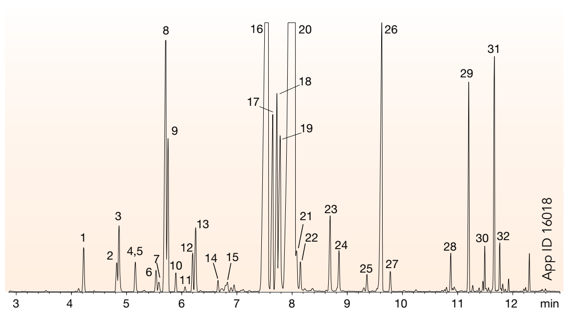
1. p-Methyl anisole
2. Methyl benzoate
3. Linalool
4. Benzyl acetate
5. Geraniol
6. Geranyl acetate
7. Copaene
8. β-Caryophyllene
9. Cinnamyl acetate
10. Humulene
11. Germacrene
12. α-Amorphene
13. Farnasene
14. δ-Cadinene
15. Farnesol
16. Benzyl benzoate
17. Benzyl salicylate
18. Farnesyl acetate



Peppermint Oil by GC/MS

Column: Zebron ZB-1ms
Dimensions: 10 meter x 0.10 mm x 0.10 µm
Part No.: 7CB-G011-02
Injection: Split 120:1 @ 160 °C, 0.2 µL
Carrier Gas: Helium @ 0.3 mL/min (constant flow)
Oven Program: 45 °C for 2 min to 130 °C @ 10 °C/min to 280 °C @ 30 °C/min for 3 min
Detector: MSD
Sample: Analytes are 10 % in dichloromethane

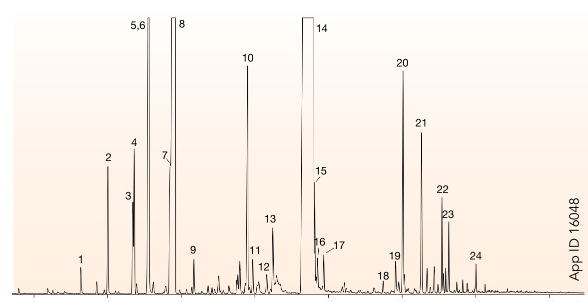
1. α-Pinene
2. Sabinene
3. β-Pinene
4. β-Myrcene
5. 3-Octanol
6. α-Terpenene
7. Cymene
8. Eucalyptol
9. δ-Limonene
10. cis-Ocimene
11. trans-Ocimene
12. γ-Terpinene
13. cis-Sabinene hydrate
14. β-Terpineol
15. Linalool
16. Menthone
17. Isomenthone
18. Menthonefuran
19. Neomenthol
20. Menthol
21. Neoisomenthol
22. α-Terpineol
23. Pulegone
24. Piperitone
25. Neomenthyl acetate
26. Menthyl acetate
27. Isomenthyl acetate
28. Bourbonene
29. Caryophyllene
30. Farnesene
31. Germacrene
32. Elemene



Spearmint Oil by GC/MS

Column: Zebron ZB-1ms
Dimensions: 10 meter x 0.10 mm x 0.10 µm
Part No.: 7CB-G011-02
Injection: Split 120:1 @ 160 °C, 0.2 µL
Carrier Gas: Helium @ 0.4 mL/min (constant flow)
Oven Program: 45 °C for 2 min to 130 °C @ 8 °C/min to 200 °C @ 30 °C/min for 2 min
Detector: MSD; 18-400 amu
Sample: Analytes are 10 % in dichloromethane

1. 2,5-Diethyltetrahydrofuran
2. α-Pinene
3. abinene
4. β-Pinene
5. β-Myrcene
6. 3-Octanol
7. Eucalyptol
8. Limonene
9. cis-Sabinene hydrate
10. Menthone
11. Isomenthone
12. 4-Terpineol
13. Dihydrocarveol
14. Carvone
15. Piperitenone
16. trans-Carvone oxide
17. cis-Carvone oxide
18. Carvyl acetate
19. cis-Jasmone
20. β-Bourbonene
21. Caryophyllene
22. β-Farnesene
23. Germacrene D
24. Caryophyllene oxide

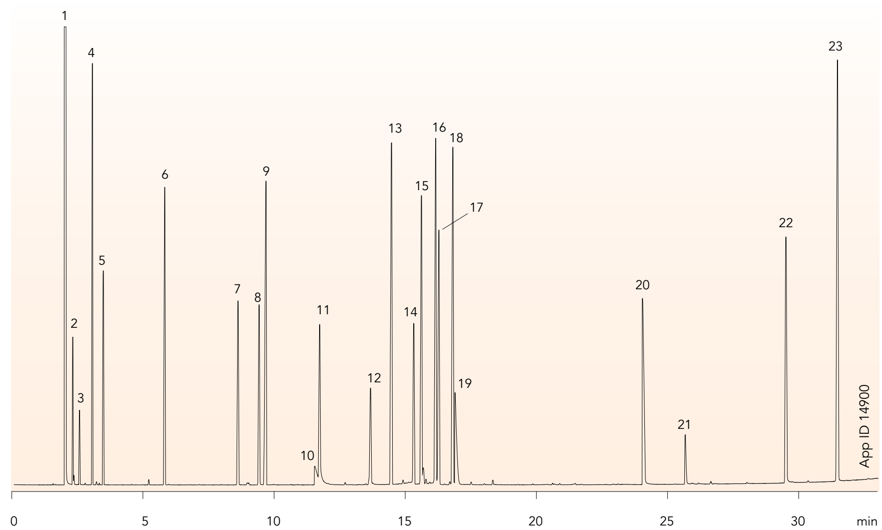


Flavors

Flavor Screening

Aromas can be exceedingly complex, with several hundred compounds playing a role. Polyethylene glycol (PEG) phases are routinely used for flavor analysis; common phases (ZB-WAX and ZB-FFAP) used for flavor screening are demonstrated below.

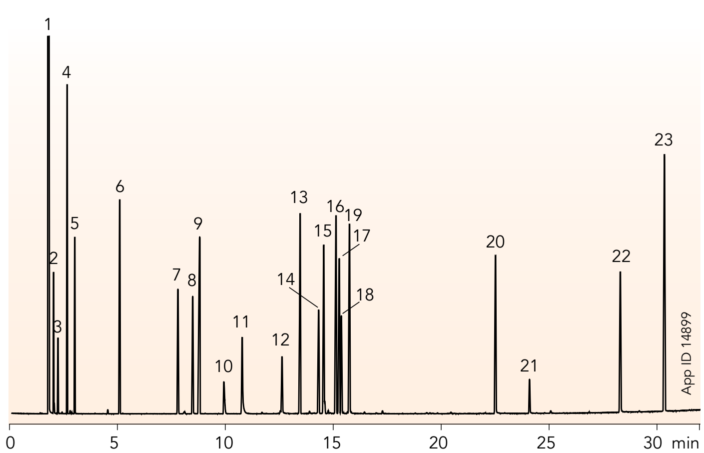
Flavors Analysis by GC/MS



Test mix courtesy of Frutarom (UK) Ltd., Flavour Chemistry Laboratory.

Column: Zebron ZB-WAX
Dimensions: 30 meter x 0.32 mm x 0.25 µm
Part No.: 7HG-G007-11
Injection: Split 100:1 @ 250 °C, 1.0 µL
Carrier Gas: Helium @ 1 mL/min (constant flow)
Oven Program: 50 °C to 250 °C @ 6 °C/min for 3 min
Detector: MSD @ 275 °C
Sample:
1. Acetone
2. Ethyl acetate
3. Ethanol
4. Decane
5. Ethyl butyrate
6. Limonene
7. 2,3-Dimethylpyrazine
8. (Z)-3-Hexenol
9. Tetradecane
10. Acetic acid
11. Decanal
12. Propylene glycol
13. Ethyl decanoate
14. Neral
15. α-Terpineol
16. Neryl Acetate
17. Geranial
18. Decanol
19. Valeric acid
20. Nonanoic acid
21. Decanoic acid
22. Vanillin
23. Anthracene

Flavors Analysis by GC/MS



Column: Zebron ZB-FFAP
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G009-11
Injection: Split 100:1 @ 250 °C, 1 µL
Carrier Gas: Helium @ 1 mL/min (constant flow)
Oven Program: 50 °C to 250 °C @ 6 °C/min for 3 min
Detector: MSD @ 270 °C
Sample:
1. Acetone
2. Ethyl Acetate
3. Ethanol
4. Decane
5. Ethyl Butyrate
6. Limonene
7. 2,3-Dimethylpyrazine
8. (z)-3-Hexenol
9. Tetradecane
10. Acetic Acid
11. Decanal
12. Propylene Glycol
13. Ethyl Decanoate
14. Neral
15. α-Terpineol
16. Neryl Acetate
17. Geranial
18. Valeric Acid
19. Decanol
20. Nonanoic Acid
21. Decanoic Acid
22. Vanillin
23. Anthracene

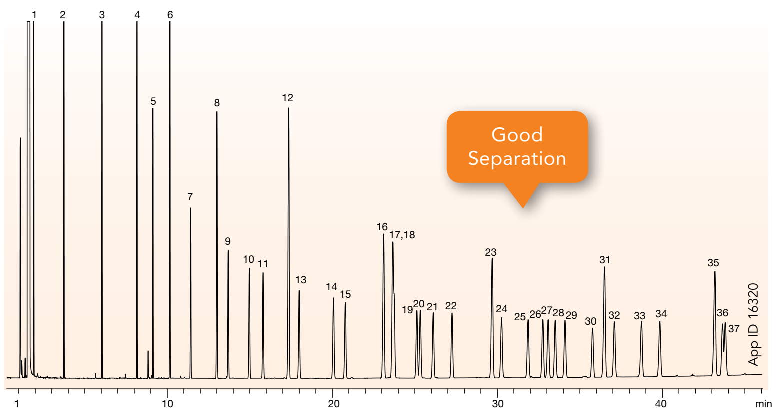
Fats & Oils

Fatty Acids & FAMEs

Fat and oil testing is important for both characterization as well as determination of total fat content. Both fatty acid methyl esters (FAMEs) and free fatty acids (FFAs) are commonly analyzed using polyethylene glycol (PEG) phases. The examples below display good resolution for both derivatized and underivatized fatty acids.

Food Industry FAMEs by GC/FID

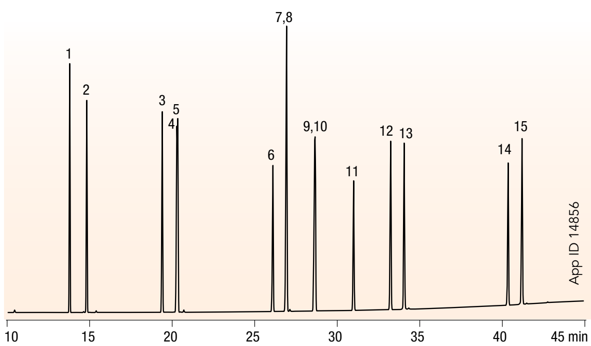
Column: Zebron ZB-WAX
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G007-11
Injection: Split 5:1 @ 220 °C, 1 µL
Carrier Gas: Helium @ 3 mL/min (constant flow)
Oven Program: 60 ˚C for 2 min to 150 ˚C @ 13 ˚C/min to 240 ˚C @ 2 ˚C/min
Detector: FID @ 250 °C
Sample: 133-266 ppm in methylene chloride



1. Methyl Butyrate (C4:0)
2. Methyl Hexanoate (C6:0)
3. Methyl Octanoate (C8:0))
4. Methyl Decanoate (C10:0)
5. Methyl Undecanoate (C:11)
6. Methyl Laurate (C12:0)
7. Methyl Tridecanoate (C13:0)
8. Methyl Myristate (C14:0)
9. Myristoleic acid methyl ester (C14:1)
10. Methyl Pentadecanoate (C15:0)
11. cis-10-Pentadecenoic acid methyl ester (C15:1)
12. Methyl Palmitate (C16:0)
13. Palmitoleic acid methyl ester (C16:1)
14. Methyl Heptadecanoate (C17:0)
15. cis-10-Heptadecenoic acid methyl ester (C17:1)
16. Methyl Stearate (C18:0)
17. Oleic acid methyl ester (C18:1n9c)
18. Elaidic acid methyl ester (C18:1n9t)
19. Linoleic acid methyl ester (C18:2n6c)
20. Linolelaidic acid methyl ester (C18:2n6t)
21. γ-Linolenic acid methyl ester (C18:3n6)
22. Linolenic acid methyl ester (C18:3n3)
23. Methyl Arachidate (C20:0)
24. cis-11-Eicosenoic acid methyl ester (C20:1)
25. cis-11-14-Eicosadienoic acid methyl ester (C20:2)
26. cis-8,11,14-Eicosatrienoic acid methyl ester (C20:3n6)
27. Methyl Heneicosanoate (C21:0)
28. Arachidonic acid methyl ester (C20:4n6)
29. cis-11,14,17-Eicosatrienoic acid methyl ester (C20:3n3)
30. cis-5,8,11,14,17-Eicosapentaenoic acid methyl ester (C20:5n3)
31. Methyl Behenate (C22:0)
32. Erucic acid methyl ester (C22:1)
33. cis-13,16-Docosadienoic acid methyl ester (C22:2)
34. Methyl Tricosanoate (C23:0)
35. Methyl Lignocerate (C24:0)
36. cis-4,7,10,13,16,19-Docosahexaenoic acid methyl ester (C22:6n3)
37. Nervonic acid methyl ester (C24:1)

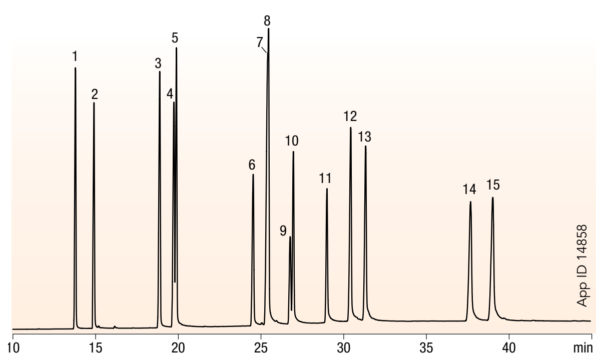
Unsaturated Fatty Acids Methyl Esters (FAMEs) by GC/FID

Column: Zebron ZB-FFAP
Dimensions: 60 meter x 0.25 mm x 0.25 µm
Part No.: 7KG-G009-11
Injection: Split 40:1 @ 220 °C, 0.2 µL
Carrier Gas: Helium @ 2.4 mL/min (constant flow)
Oven Program: 200 °C to 260 °C @ 2 °C/min for 30 min
Detector: FID @ 250 °C
Sample:
1. Methyl Myristate
2. Methyl Myristoleate
3. Methyl Palmitate
4. Methyl Palmitelaidate
5. Methyl Palmitoleate
6. Methyl Stearate
7. Methyl Oleate
8. Methyl Elaidate
9. Methyl Linoleate
10. Methyl Linelaidate
11. Methyl Linolenate
12. Methyl Arachidate
13. Methyl Gondonate
14. Methyl Behenate
15. Methyl Erucate



Unsaturated Free Fatty Acids by GC/FID

Column: Zebron ZB-FFAP
Dimensions: 60 meter x 0.25 mm x 0.25 µm
Part No.: 7KG-G009-11
Injection: Split 40:1 @ 220 °C, 0.2 µL
Carrier Gas: Helium @ 2.4 mL/min (constant flow)
Oven Program: 200 °C to 260 °C @ 2 °C/min for 30 min
Detector: FID @ 250 °C
Sample:
1. Myristic Acid
2. Myristoleic Acid
3. Palmitic Acid
4. Palmitelaidic Acid
5. Palmitoleic Acid
6. Stearic Acid
7. Elaidic Acid
8. Oleic Acid
9. Linolelaidic Acid
10. Linoleic Acid
11. Linolenic Acid
12. Arachidic Acid
13. Gondonic Acid (C15)
14. Behenic Acid (C17)
15. Erucic Acid (C19)



Fats & Oils

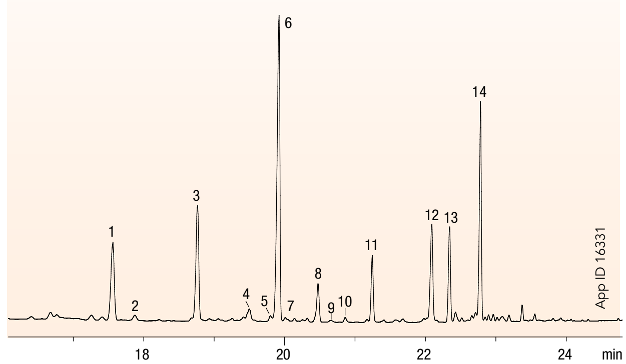
Sterols

Sterols are naturally occurring steroid alcohols in plants, animals, and fungi. Phytosterols and cholesterol are commonly tested; sterol content for example is analyzed to determine olive oil quality and authenticity. Dietary tocopherols are sometimes tested with sterols due to their related health effects. Methods for analysis of sterols from common food matrices and in combination with tocopherols are demonstrated below.

Vitamin E and Sterols by GC/FID

Column: Zebron™ ZB-5
Dimensions: 30 meter x 0.25 mm x 0.10 µm
Part No.: 7HG-G002-02
Injection: Splitless @ 220 °C, 1 µL
Carrier Gas: Helium @ 1.8 mL/min (constant flow)
Oven Program: 110 °C for 0.2 min to 140 °C @ 30 °C/min to 230 °C @ 10 °C/min for 6 min to 340 °C @ 10 °C/min for 15.8 min
Detector: FID @ 340 °C
Sample: Analytes derivatized via BSTFA:TMCS; 99:1 in pyridine

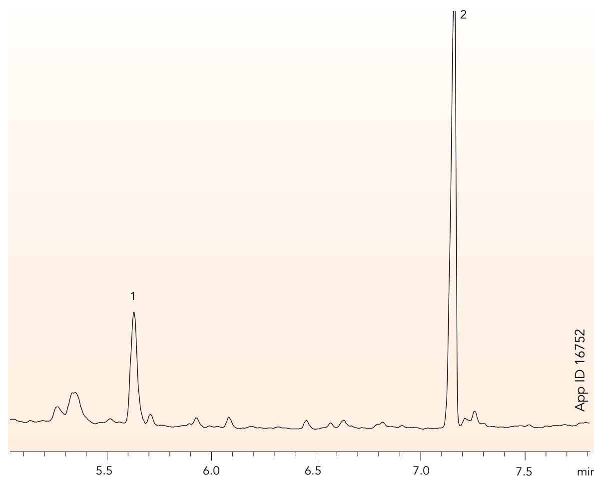
1. Squalene
2. Lignoceric acid
3. δ-Tocopherol
4. δ-Tocomonoenol
5. Campesta-3,5-diene
6. γ-Tocopherol
7. Stigmasta-3,5,22-triene
8. γ-Tocomonoenol
9. Stigmasta-3,5-diene
10. Cholesterol
11. α-Tocopherol
12. Campesterol
13. Stigmasterol
14. β-Sitosterol



Lard Sterols by GC/FID

Column: Zebron ZB-5HT Inferno™
Dimensions: 30 meter x 0.25 mm x 0.10 µm
Part No.: 7HG-G015-02
Injection: Splitless @ 350 °C, 0.5 µL
Carrier Gas: Helium @ 2 mL/min (constant flow)
Oven Program: 220 °C to 350 °C @ 15 °C/min
Detector: FID @ 350 °C
Sample: Prepared by saponification, solid phase extraction (SPE), and derivatization via BSTFA:TMCS; 99:1 in pyridine

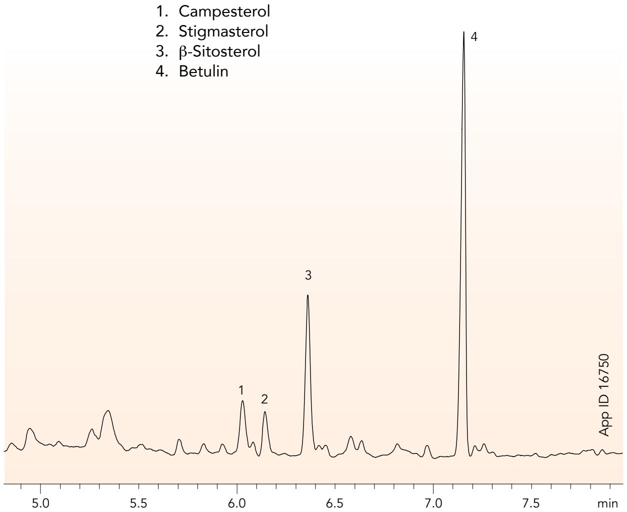
1. Cholesterol
2. Betulin



Margarine Sterols by GC/FID

Column: Zebron ZB-5HT Inferno
Dimensions: 30 meter x 0.25 mm x 0.10 µm
Part No.: 7HG-G015-02
Injection: Splitless @ 350 °C, 0.5 µL
Carrier Gas: Helium @ 2 mL/min (constant flow)
Oven Program: 220 °C to 350 °C @ 15 °C/min
Detector: FID @ 350 °C
Sample: Prepared by saponification, solid phase extraction (SPE), and derivatization via BSTFA: TMCS; 99:1 in pyridine

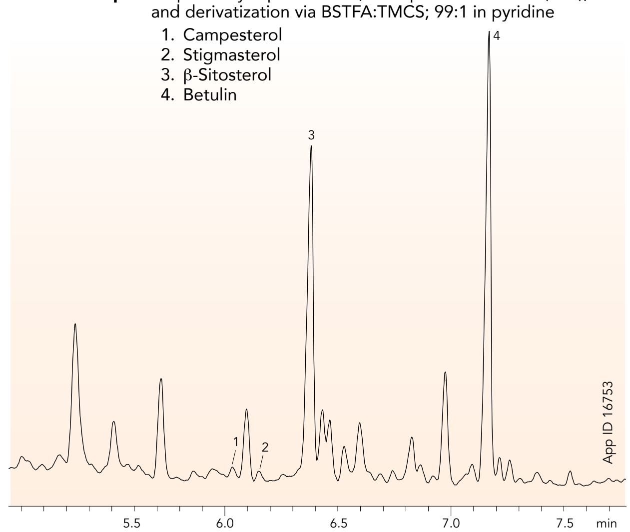
1. Campesterol
2. Stigmasterol
3. β-Sitosterol
4. Betulin



Olive Oil Sterols by GC/FID

Column: Zebron ZB-5HT Inferno
Dimensions: 30 meter x 0.25 mm x 0.10 µm
Part No.: 7HG-G015-02
Injection: Splitless @ 350 °C, 0.5 µL
Carrier Gas: Helium @ 2 mL/min (constant flow)
Oven Program: 220 °C to 350 °C @ 15 °C/min
Detector: FID @ 350 °C
Sample: Prepared by saponification, solid phase extraction (SPE), and derivatization via BSTFA:TMCS; 99:1 in pyridine

1. Campesterol
2. Stigmasterol
3. β-Sitosterol
4. Betulin



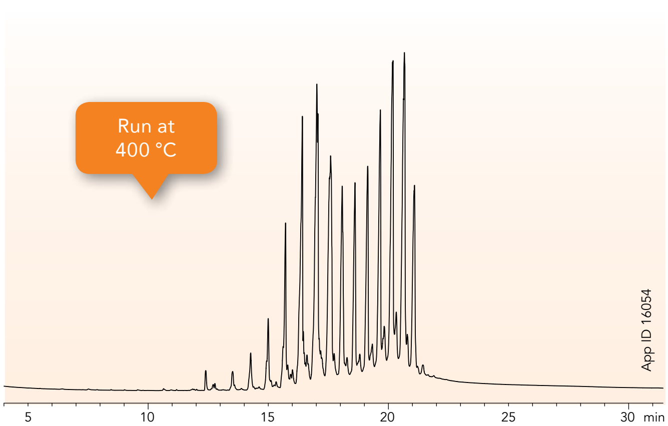
Fats & Oils

Triglycerides

Triglycerides are naturally-occurring esters of fatty acids and glycerol. Because these compounds have relatively high molecular weights and polarities that increase with the degree of unsaturation, high oven temperatures are necessary for sufficient separations. Choosing a GC column designed to withstand such temperatures (such as those with improved polyimide coatings that resist brittleness at 400 °C or higher) can provide the necessary robustness to achieve good separation. The separations below are performed using a ZB-5HT Inferno™ GC column, which is specifically designed to stand up to high oven temperatures, are also shown.

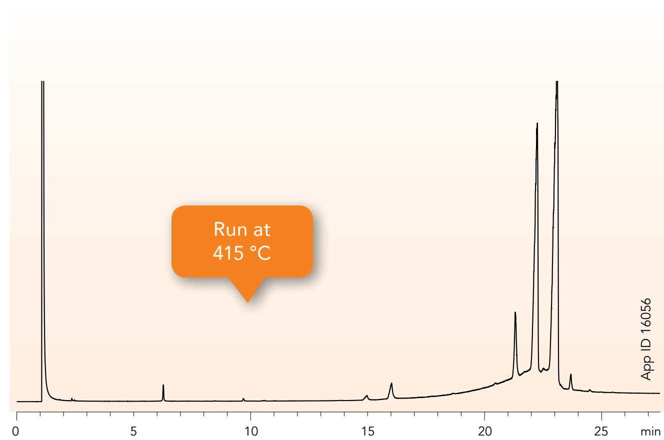
Butter Triglycerides by GC/FID

Column: Zebron™ ZB-5HT Inferno™
Dimensions: 15 meter x 0.32 mm x 0.10 µm
Part No.: 7EM-G015-02
Injection: On-Column @ 103 °C, 2 µL
Carrier Gas: Helium @ 1.8 mL/min (constant flow)
Oven Program: 100 °C to 400 °C @ 14 °C/min for 10 min
Detector: FID @ 400 °C
Sample: Butter



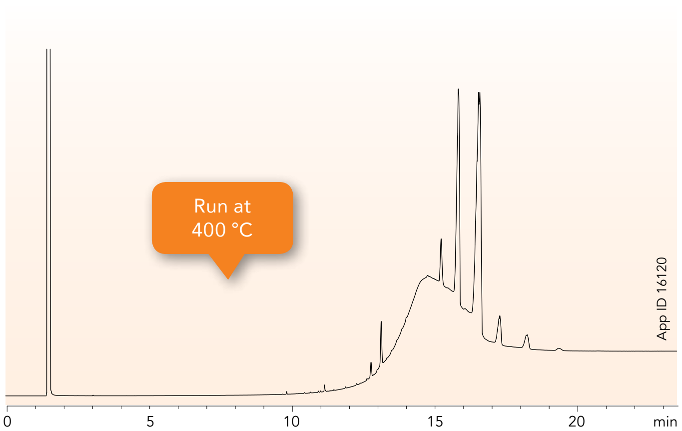
Olive Oil Triglycerides by GC/FID

Column: Zebron ZB-5HT Inferno
Dimensions: 30 meter x 0.25 mm x 0.10 µm
Part No.: 7HG-G015-02
Injection: On-Column @ 223 °C, 0.1 µL
Carrier Gas: Helium @ 1 mL/min (constant flow)
Oven Program: 220 °C for 1 min to 400 °C @ 8 °C/min for 4 min
Detector: FID @ 415 °C
Sample: Olive Oil



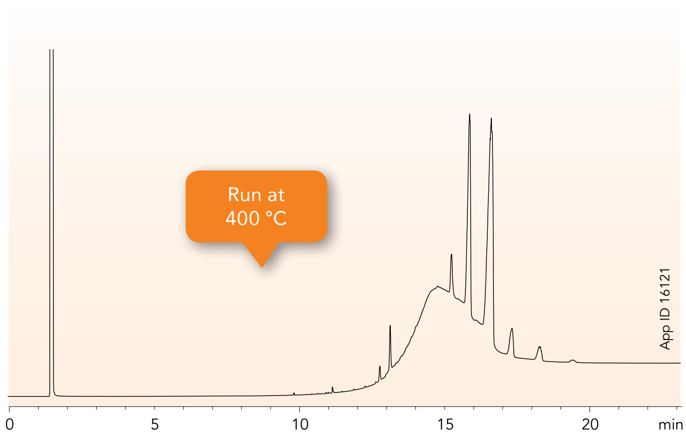
Peanut Oil Triglycerides by GC/FID

Column: Zebron ZB-5HT Inferno
Dimensions: 30 meter x 0.25 mm x 0.10 µm
Part No.: 7HG-G015-02
Injection: On-Column @ 63 °C, 0.1 µL
Carrier Gas: Helium @ 1.5 mL/min (constant flow)
Oven Program: 60 °C to 400 °C @ 25 °C/min for 10 min
Detector: FID @ 400 °C
Sample: Peanut Oil



Canola Oil Triglycerides by GC/FID

Column: Zebron ZB-5HT Inferno
Dimensions: 30 meter x 0.25 mm x 0.10 µm
Part No.: 7HG-G015-02
Injection: On-Column @ 63 °C, 0.1 µL
Carrier Gas: Helium @ 1.5 mL/min (constant flow)
Oven Program: 60 °C to 400 °C @ 25 °C/min for 10 min
Detector: FID @ 400 °C
Sample: Canola Oil



Application Spotlight

Analysis of Alcoholic Beverages (Distilled Spirits and Wines) Using a 100 % Aqueous Stable GC Column

Introduction

During wine and distilled spirit fermentation, compounds called congeners are formed. These congeners can contribute to a spirit's flavor, but can be harmful if consumed in excess. Some spirits, such as vodka, undergo extra processing steps to eliminate these compounds. Beyond health concerns, an overabundance of a specific congener can signify a problem with production or improper storage conditions. Distilleries also commonly perform congener profile analyses to mitigate adulteration claims and test for authenticity.

Because the congener profile of a distilled spirit is significant for both quality control and health safety, accurate analysis of these compounds is very important. Testing methods used to analyze these compounds must therefore be both qualitative, quantitative, and reproducible. GC/FID analysis of common congeners (such as those in **Table 1**) is known for its reproducibility and accuracy and is heralded as the industry standard. Polyethylene glycol (PEG) columns have historically provided acceptable selectivity but been unstable with aqueous samples, resulting in poor reproducibility and decreased lifetime. Traditional analysis is challenging because finished products are composed of 40 and 80 percent water, and congeners are present only in low parts per million (ppm).

Headspace sampling can eliminate some matrix effects and enhance the performance of the more volatile congeners, but will suppress the response of less volatile analytes which may be responsible for unique flavors. Direct injection is therefore still required to verify specific samples. This work explores the separation of distillation congeners on a Zebron ZB-WAX*PLUS*™, a water-stable PEG phase.

Methods and Materials

Analyses were performed using an Agilent® 6890 (Agilent Technologies, Palo Alto, CA, USA). Liquid injections used an Agilent liquid autosampler. Headspace samples used an HT-200 Automatic Headspace Sampler (Overbrook Scientific, Boston, MA, USA). All standards are > 95 % purity, and wine and distilled samples were purchased from local grocery stores. Instrument conditions for each method are included with the chromatogram.

Results and Discussion

Some of the primary congeners are very volatile and may be easily determined using headspace injection. A headspace injection of main congeners and flavor compounds is presented in **Figure 1**. This helps to keep most of the water and contaminants out of the system, which can contribute to decreased chromatographic performance and result in premature column deterioration. The earlier eluting peaks give excellent responses and can easily be quantified. Baseline resolution was achieved for acetaldehyde, ethyl acetate, and methanol (important components in monitoring the distillation process).

In some analyses, it is important to focus on the later eluting compounds because these have a large impact on the complicated flavors of fermented beverages. These congeners form as a result of the conditions of storage and aging and must be monitored to ensure product consistency. These later eluting compounds have lower volatility, and are better analyzed via liquid injections. A liquid injection of the same flavor standard is injected in **Figure 2**. Notice that the later eluting compounds have higher responses given the same concentration. This allows for a more accurate analysis of the flavor compounds which may be unique to a particular brand. For this reason, liquid injections are the preferred method for determining flavors.

On other PEG-based WAX columns, water can affect system performance and reproducibility. Zebron ZB-WAX*PLUS* columns are specially bonded to stand up to repeated aqueous injections. This can be seen in **Figure 3**, where multiple injections were made of a Scotch whiskey consisting of ~60 % water. There is no change in peak shape or retention times over time, and repeated injections have < 5 % RSD.

In addition to providing aqueous stability, ZB-WAX*PLUS* also provides very low activity for acidic compounds. This allowed for the fatty acids (eluting past 12 min) to be analyzed within the same run. The lack of acetic acid in the sample suggests that the product was well stored prior to opening and that the cork seal from the bottle was not compromised.

Additional beverages that have not been distilled can also be analyzed using the ZB-WAX*PLUS*. A chromatogram for an Italian wine is shown in **Figure 4**. In this instance, sample preparation consisted of only filtering before injecting. This chromatogram shows baseline separation of early eluting congeners, which can be used to monitor the fermentation process.

Conclusion

Method reproducibility and accuracy for distilled spirit analysis is very important for both quality control and health safety. Therefore, using an aqueous stable GC column is the best approach for congener analysis as it allows direct injection. Fermented beverages including distilled spirit congeners have historically been difficult to analyze by direct injection, but can be analyzed successfully using the Zebron ZB-WAX*PLUS* GC column. By using a Zebron ZB-WAX*PLUS* GC column for distilled spirit analysis, accuracy and reproducibility can be achieved without sacrificing resolution.

Table 1: Common Distilled Spirit Congeners

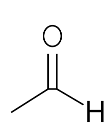
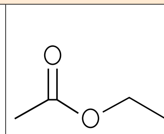
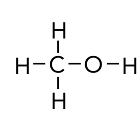
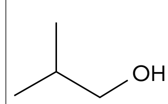
			
Acetaldehyde	**Ethyl acetate**	**Methanol**	**Isobutanol**

Figure 1: Distilled Alcohol Standard by Headspace GC/FID

Column: Zebron ZB-WAX*PLUS*™
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HM-G013-11
Injection: Split 25:1 @ 210 °C, 1 µL
Carrier Gas: Hydrogen @ 1 mL/min (constant flow)
Oven Program: 35 °C for 7 min to 60 °C @ 5 °C/min for 2 min to 210 °C @ 10 °C/min
Detector: FID @ 230 °C
Note: Static headspace injection (80 °C for 20 min)
Sample:

1. Acetaldehyde
2. Isobutanal
3. Ethyl formate
4. Acrolein
5. Ethyl acetate
6. Acetal
7. Methanol
8. Ethanol
9. Isobutyl acetate
10. 2-Butanol
11. Ethyl butyrate
12. 1-Propanol
13. Isobutanol
14. Allyl alcohol
15. Isoamyl acetate
16. Butanol
17. 4-Methyl-2-pentanol
18. Methyl-2-butanol
19. Methyl-3-butanol
21. Ethyl heptanoate
22. Ethyl lactate
23. Hexanol
24. cis-3-Hexenol
25. Ethyl caprylate
26. Furfural
27. Benzaldehyde
26. Furfural
27. Benzaldehyde
28. Ethyl caprate
29. Diethyl succinate
30. Ethyl laurate
31. Phenyl-2-ethanol

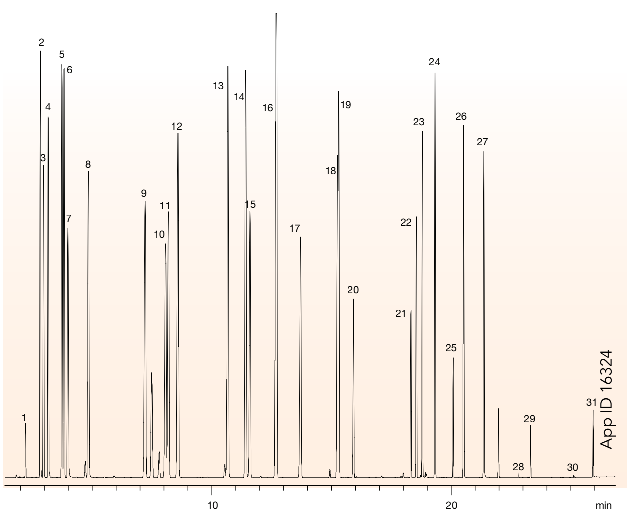


Figure 2: Distilled Alcohol Standard by Liquid Injection

Column: Zebron ZB-WAX*PLUS*
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G013-11
Injection: Split 25:1 @ 210 °C, 1 µL
Carrier Gas: Hydrogen @ 1 mL/min (constant flow)
Oven Program: 35 °C for 6 min to 60 °C @ 5 °C/min for 2 min to 210 °C @ 10 °C/min
Detector: FID @ 230 °C
Note: 200 ppm standard in methylene chloride
Sample:

1. Acetaldehyde
2. Isobutanal
3. Ethyl formate
4. Acrolein
5. Ethyl acetate
6. Acetal
7. Methanol
8. Methylene chloride
9. Ethanol
10. Isobutyl acetate
11. 2-Butanol
12. Ethyl butyrate
13. 1-Propanol
14. Isobutanol
15. Allyl alcohol
16. Isoamyl acetate
17. 1-Butanol
18. 4-Methyl-2-pentanol
19. Methyl-2-butanol
20. Methyl-3-butanol
21. Ethyl caproate
22. Ethyl heptanoate
23. Ethyl lactate
24. Hexanol
25. cis-3-Hexenol
26. Ethyl caprylate
27. Furfural
28. Benzaldehyde
29. Linalool
30. Linalyl acetate
31. Ethyl caprate
32. Diethyl succinate
33. Ethyl laurate
34. 2-Phenyl ethanol

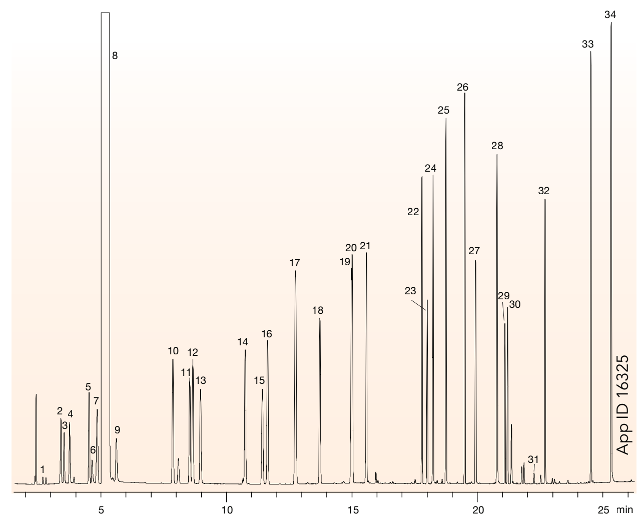


Figure 3: Replicate Liquid Injections of Undiluted Scotch Whiskey

Column: Zebron ZB-WAX*PLUS*
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G013-11
Injection: Split 30:1 @ 140 °C, 0.2 µL
Carrier Gas: Helium @ 1.4 mL/min (constant flow)
Oven Program: 35 °C for 5 min to 85 °C @ 10 °C/min to 200 °C @ 25 °C/min for 1 min
Detector: FID @ 200 °C
Sample:

1. Acetaldehyde
2. Ethyl acetate
3. Methanol
4. Ethanol
5. Propanol
6. Isobutanol
7. 2-Methylbutanol
8. 3-Methylbutanol

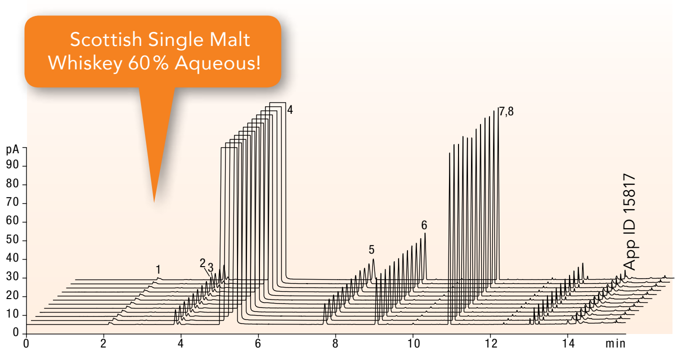
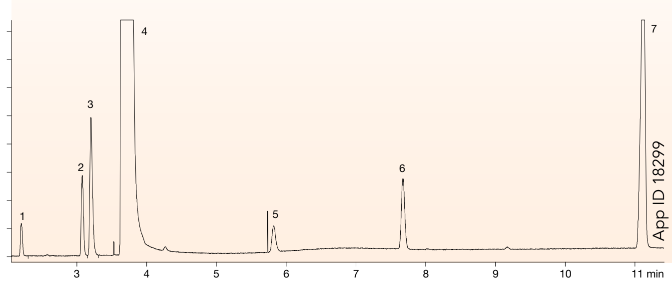


Figure 4: Filtered Liquid Injection of Italian Wine

Column: Zebron ZB-WAX*PLUS*
Dimensions: 30 meter x 0.32 mm x 0.25 µm
Part No.: 7HM-G013-11
Injection: Split 10:1 @ 150 °C, 0.2 µL
Carrier Gas: Helium @ 2.3 mL/min (constant flow)
Oven Program: 40 °C for 5 min to 150 °C @ 5 °C/min for 5 min to 220 °C @ 20 °C/min for 2 min
Detector: FID @ 280 °C
Accessories: Phenex™-RC Syringe Filter (AF0-2203-52)
Note: Wine has been filtered through 0.2 µm regenerated cellulose filter and directly injected.
Sample:

1. Acetaldehyde
2. Ethyl acetate
3. Methanol
4. Ethanol
5. Propanol
6. Isobutanol
7. 3-Methyl-1-butanol



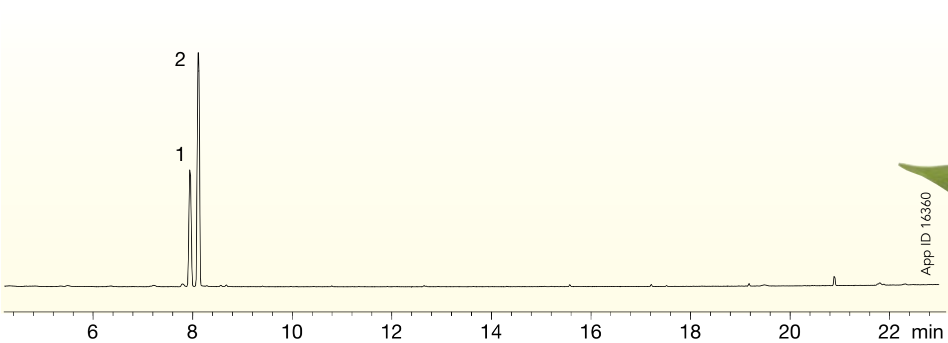
Food Safety
Featured Applications

Additives & Preservatives

Many additives and preservatives are commonly tested using GC. Borneol, a naturally occurring terpene derived from plant essential oils, is used as a flavor or fragrance additive in small amounts. Though not approved by the U.S. Food and Drug Administration, borneol is not prohibited as an ingredient in herbal/dietary supplements, and additionally used in traditional Chinese medicines for its antibiotic, sedative, and pain-relieving properties. However, risk of allergenic side effects to the respiratory and skin systems has increased its testing.

Borneol by GC/MS

Column: Zebron™ ZB-MultiResidue™-1
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G016-11
Injection: Splitless @ 270 °C (hold 0.66 sec), 1.0 µL
Carrier Gas: Helium @ 0.3 mL/min (constant flow)
Oven Program: 60 °C for 2 min to 270 °C @ 10 °C/min
Detector: MSD @ 230 °C, 45-450 amu
Sample: 1. Isoborneol
2. Borneol



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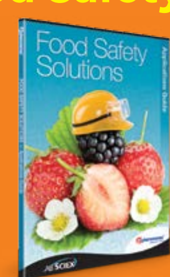


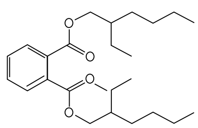
www.phenomenex.com/Food

Food Contact Materials

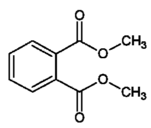
Phthalates

Phthalates are internationally regulated carcinogenic, teratogenic, and mutagenic plasticizers that can migrate from packaging into food and beverage products. GC/MS has been widely used for phthalate residue testing, but late elution of the heavier compounds can lead to low detection limits. Many of the compounds additionally require chromatographic separation due to virtually identical fragmentation patterns. The below method features a fast, 11 minute run with good thermal stability.

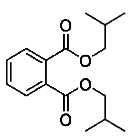
Bis(2-ethylhexyl) phthalate



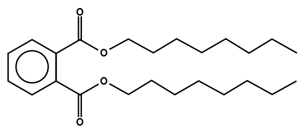
Dimethyl phthalate



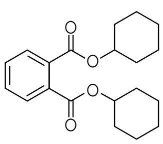
Diisobutyl phthalate



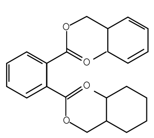
Di-n-octyl phthalate



Dicyclohexyl phthalate



Benzyl butyl phthalate

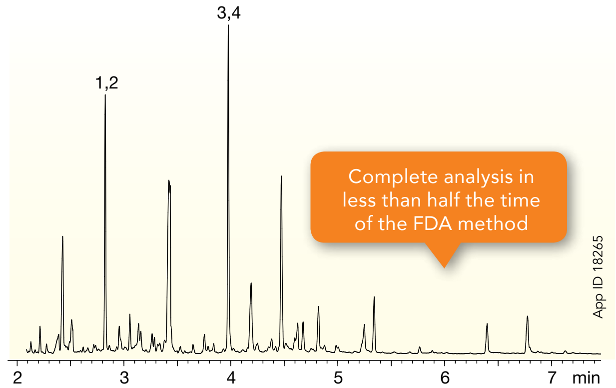


Melamine and Cyanuric Acid

Though low in toxicity individually, melamine and cyanuric acid crystallize in a 1:1 ratio in concentrations exceeding 2 µg/mL to form melamine cyanurate, a very poorly water-soluble complex. Consumption of melamine cyanurate can result in adverse health problems, including kidney failure and death. The GC/MS method below yields fully resolved peaks in less than 9 minutes and allows for the high-temperature removal of residual on-column contaminants for longer column lifetime.

Column: Zebron™ ZB-XLB-HT Inferno™
Dimensions: 15 meter x 0.25 mm x 0.25 µm
Part No.: 7EG-G024-11
Injection: On-Column @ 103 °C, 1 µL
Carrier Gas: Helium @ 1.4 mL/min (constant flow)
Oven Program: 100 °C for 0.5 min to 320 °C @ 25 °C/min
Detector: MSD @ 325 °C
Sample: Analytes are 200 ng / 100 µL in BSTFA / Pyridine (1:1)

1. Cyanuric Acid 13C3 (IS)
2. Cyanuric Acid
3. Melamine 13C3 15N3 (IS)
4. Melamine

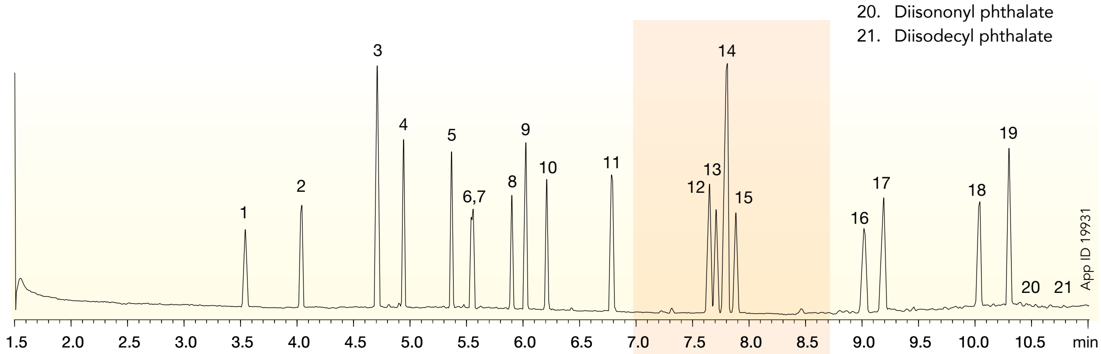


Phthalates by GC/MS

Column: Zebron ZB-50
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G004-11
Injection: Split 10:1@ 260 °C, 1 µL
Carrier Gas: Helium @ 1 mL/min (constant flow)
Oven Program: 135 °C to 275 °C @ 25 °C/min for 3.5 min to 340 °C @ 35 °C/min for 1 min
Detector: MSD @ 320 °C , 45-500 amu

Sample

1. Dimethyl phthalate
2. Diethyl phthalate
3. Diallyl phthalate
4. Di-n-propyl phthalate
5. Di-n-butyl phthalate
6. Diisobutyl phthalate
7. Di-n-hexyl phthalate
8. Bis(2-methoxyethyl) phthalate
9. Di-n-pentyl phthalate
10. Bis(2-ethoxyethyl) phthalate
11. Di-(4-methyl-2-pentyl) phthalate
12. Bis(2-ethylhexyl) phthalate
13. Benzyl butyl phthalate
14. Di-n-heptyl phthalate
15. Bis(2-n-butoxyethyl) phthalate
16. Dicyclohexyl phthalate
17. Di-n-octyl phthalate
18. Diphenylhexyl phthalate
19. Di-n-nonyl phthalate
20. Diisononyl phthalate
21. Diisodecyl phthalate



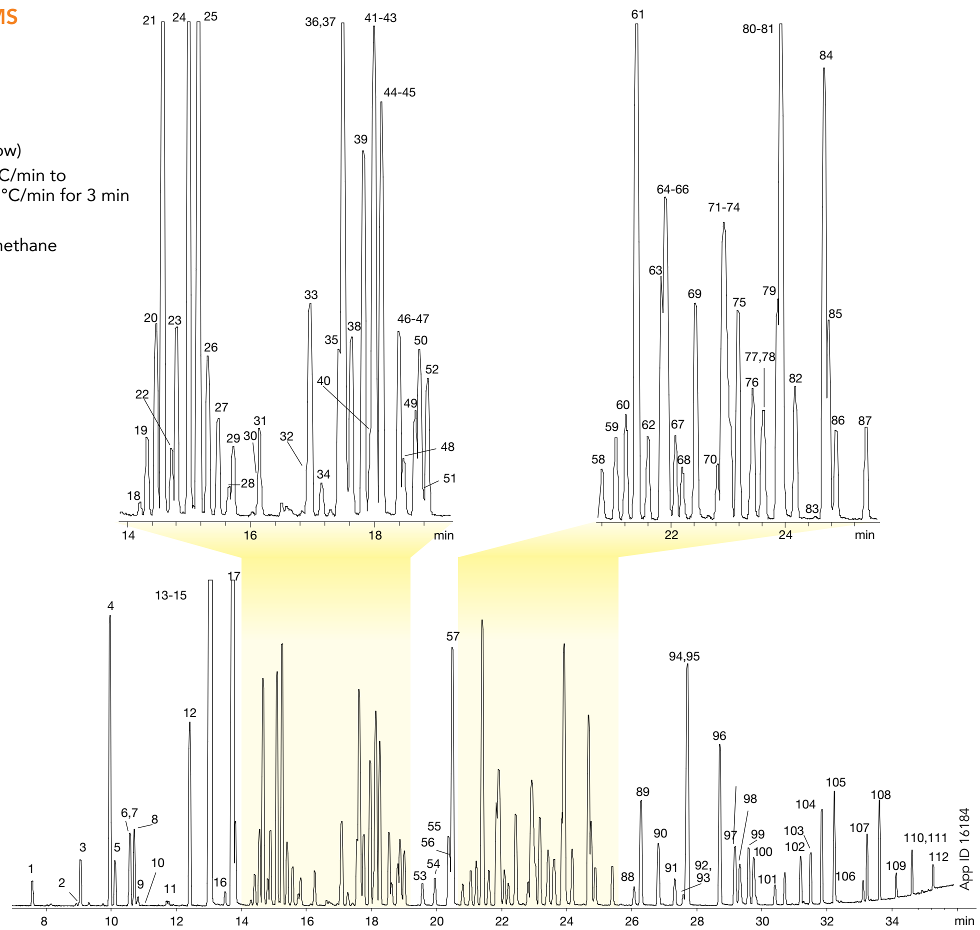
Pesticides & Antimicrobials

Multi-Residue Screening

Though globally regulated due to their detrimental health effects, restrictions on pesticide use differ from one country to the next. Since many different types of pesticides can be used on the same food product, multi-residue screening approaches by GC/MS are used to analyze large lists of 100 compounds or more in a single run. As demonstrated below, optimized selectivity offers increased resolution of critical compounds vs. standard "5ms" phases.

Multi-Residue Pesticide Screen by GC/MS

Column: Zebron™ ZB-MultiResidue™-1
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G016-11
Injection: Splitless @ 260 °C, 1 µL
Carrier Gas: Helium @ 0.9 mL/min (constant flow)
Oven Program: 80 °C for 0.5 min to 150 °C @ 10 °C/min to 240 °C @ 4 °C/min to 320 °C @ 15 °C/min for 3 min
Detector: MSD @ 320 °C; 45-400 amu
Note: Analytes were 1 ppm in dichloromethane



Sample:

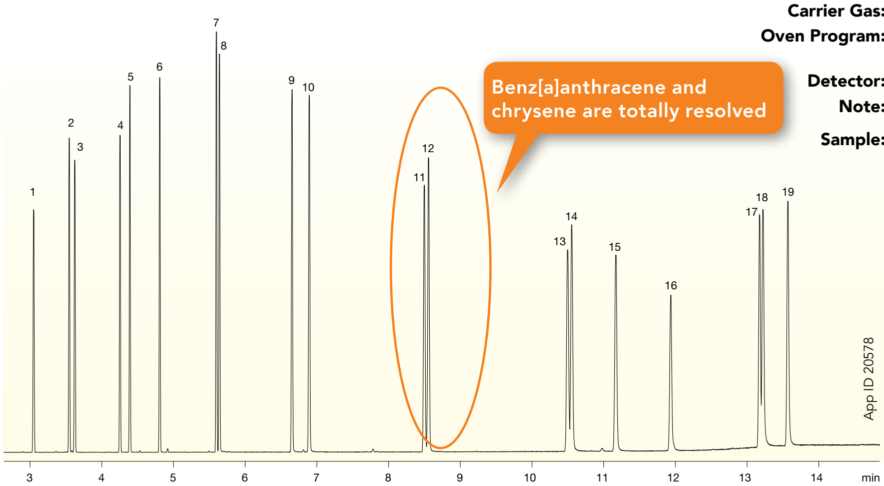
1. Dichlorvos
2. DEET
3. EPTC
4. 3,5-Dichlorobenzoic acid (methyl ester)
5. Butylate
6. 4-Nitrophenol (methyl ester)
7. Vernolate
8. Mevinphos
9. Mevinphos isomer
10. Pebulate
11. Trichlorfon
12. Dicamba (methyl ester)
13. Molinate
14. Tebuthiuron
15. MCPP (methyl ester)
16. Tetraethyl pyrophosphate (methyl ester)
17. MCPA (methyl ester)
18. Demeton Isomer
19. Thionazin (zinophos)
20. Dichloroprop (methyl ester)
21. Propachlor
22. Cycloate
23. Ethoprop
24. Trifluralin
25. Benefin
26. 2,4-D (methyl ester)
27. Sulfotep
28. Naled
29. Chlorpropham
30. Dicrotophos
31. Phorate
32. Monocrotophos
33. Pentachlorophenol (methyl ester)
34. Demeton
35. Atraton
36. Profluralin
37. Prometon
38. Silvex (methyl ester)
39. Terbufos
40. Dimethoate
41. Simazine
42. Propazine
43. Atrazine
44. Diazinon
45. Dioxathion
46. Terbuthylazine
47. Fonofos
48. Pronamide (propyzamide)
49. Chloramben (methyl ester)
50. 2,4,5-T Methyl ester
51. Phosphamidon isomer
52. Disulfoton
53. Secbumeton
54. Terbacil
55. Dinoseb (methyl ester)
56. Dichlofenthion
57. 2,4-DB (methyl ester)
58. Phosphamidon
59. Chlorpyrifos methyl
60. Alachlor
61. Bentazon (methyl ester)
62. Ronnel
63. Prometryn
64. Methyl parathion
65. Ametryn
66. Simetryn
67. Aspon
68. Metribuzin
69. Terbutryn
70. Malathion
71. Fenitrothion
72. Picloram (methyl ester)
73. Metolachlor
74. Chlorpyrifos
75. DCPA
76. Bromacil
77. Fenthion
78. Trichloronate
79. Triadimeton
80. Isopropalin
81. Parathion
82. MGK-624
83. Merphos
84. Pendimethalin (Penoxaline)
85. Diphenamide
86. MGK-264 isomer
87. Clofenvinfos
88. Crotoxyphos
89. Butachlor
90. Stirofos
91. Tokuthion
92. Napropamide
93. Fenamiphos
94. Merphos oxide
95. Oxadiazon
96. Oxyfluorfen
97. Carboxin
98. Tricyclazole
99. Acifluorfen
100. Ethion
101. Fensulfothion
102. Carbofenothion
103. Famfur
104. Norflurazon
105. Hexazinone
106. EPN
107. Phosmet
108. Leptophos
109. Azinphos-methyl
110. Fenarimol
111. Azinphos-ethyl
112. Coumaphos

Environmental Contaminants

Polycyclic Aromatic Hydrocarbons (PAHs)

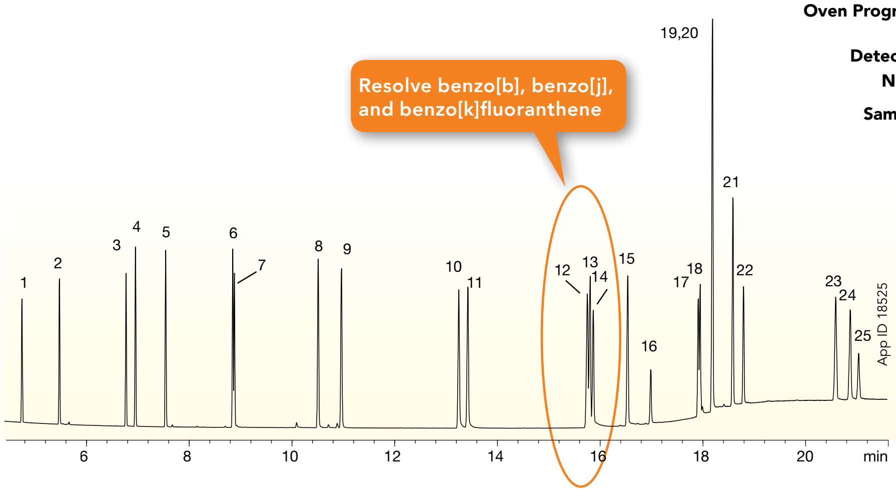
PAHs are a class of persistent organic pollutants (POPs) that are implicated as carcinogens. Due to their potential health risk at low levels, PAH testing is often performed from a variety of food matrices. PAH isomers are additionally well-known for their challenging isomers and interferences. Separations of common troublesome analytes are demonstrated below.

PAHs by GC/MS



Column: Zebron™ ZB-SemiVolatiles
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G027-11
Injection: Split 10:1 µL @ 280 °C, 1 µL
Carrier Gas: Helium @ 1.4 mL/min (constant flow)
Oven Program: 100 °C for 0.5 min to 260 °C @ 30 °C/min to 295 °C @ 6 °C/min to 325 °C @ 25 °C/min for 2 min
Detector: MSD @ 340 °C; 45-450 amu
Note: Analytes are 25 ppm in Dichloromethane
Sample:
1. Naphthalene
2. 2-Methylnaphthalene
3. 1-Methylnaphthalene
4. Acenaphthylene
5. Acenaphthene
6. Fluorene
7. Phenanthrene
8. Anthracene
9. Fluoranthene
10. Pyrene
11. Benz[a]anthracene
12. Chrysene
13. Benzo[b]fluoranthene
14. Benzo[k]fluoranthene
15. Benzo[a]pyrene
16. 3-Methylcholanthrene
17. Indeno[1,2,3-cd]pyrene
18. Dibenz[a,h]anthracene
19. Benzo[g,h,i]perylene

PAHs by GC/MS



Column: Zebron ZB-35
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G003-11
Injection: On-Column @ 83 °C, 1 µL
Carrier Gas: Helium @ 1.2 mL/min (constant flow)
Oven Program: 80 °C for 0.66 min to 250 °C @ 20 °C/min to 360 °C @ 8 °C/min for 6 min
Detector: MSD @ 360 °C; 45-450 amu
Note: Analytes are 10 ppm in Dichloromethane
Sample:
1. Naphthalene
2. 2-Methylnaphthalene
3. Acenaphthylene
4. Acenaphthene
5. Fluorene
6. Phenanthrene
7. Anthracene
8. Fluoranthene
9. Pyrene
10. Benz[a]anthracene
11. Chrysene
12. Benzo[b]fluoranthene
13. Benzo[k]fluoranthene
14. Benzo[j]fluoranthene
15. Benzo[a]pyrene
16. 3-Methylcholanthrene
17. Dibenz[a,h]acridine
18. Dibenz[a,j]acridine
19. Indeno[1,2,3-cd]pyrene
20. Dibenz[a,h]anthracene
21. Benzo[g,h,i]perylene
22. 7H-Dibenzo[c,g]carbazole
23. Dibenzo[a,e]pyrene
24. Dibenzo[a,i]pyrene
25. Dibenzo[a,h]pyrene

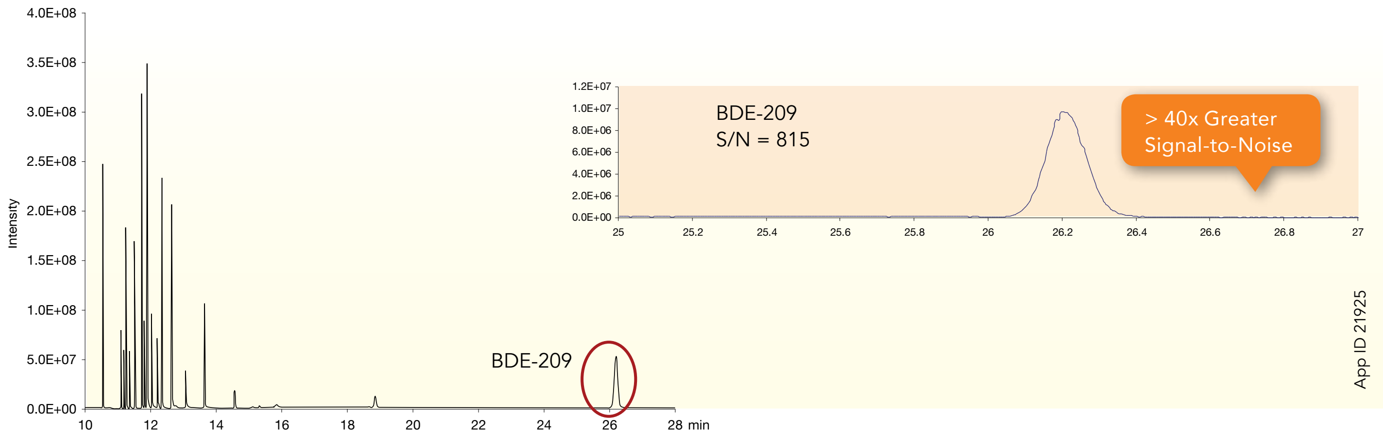
Environmental Contaminants

Polybrominated Diphenyl Ethers (PBDEs)

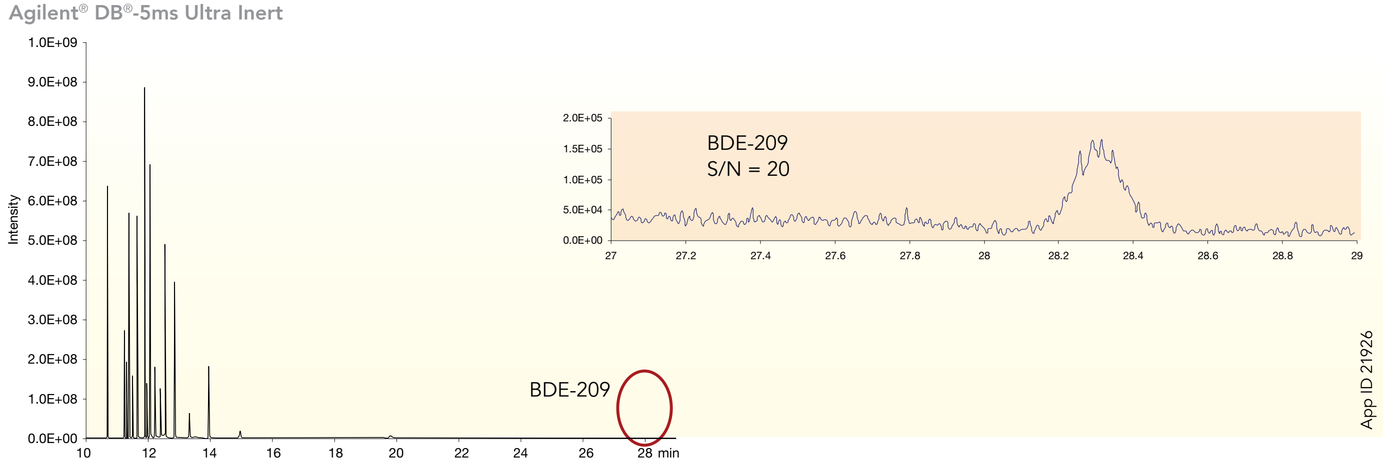
PBDEs consist of 209 individual congeners which vary in toxicity. Though congener testing is performed by high resolution GC (HRGC/HRMS), achieving accurate, well-resolved separations is historically problematic due to the sheer number of compounds and analyte stability. The fast method below includes the quantitation of BDE-209 in a single analytical run, with improved column stability compared to traditionally used phases.

PBDEs by GC/HRMS

Zebron™ ZB-SemiVolatiles



Agilent® DB®-5ms Ultra Inert



Conditions for both columns:

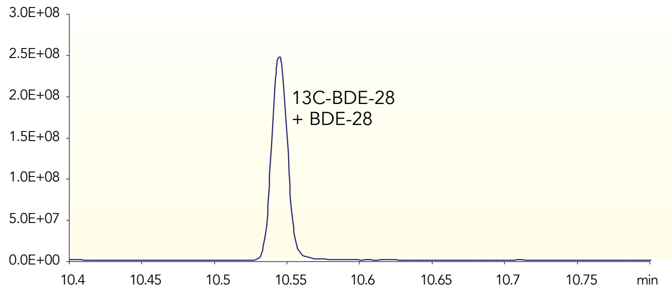
Column: A: Zebron ZB-SemiVolatiles
B: Agilent DB-5ms Ultra Inert
Dimensions: 20 meter x 0.18 mm x 0.18 µm
Injection: Splitless @ 85 °C, 5 µL
Carrier Gas: Helium @ 0.85 mL/min (constant flow)
Oven Program: 70 °C for 1.25 min to 240 °C @ 20 °C/min to 320 °C @ 50 °C/min for 18 min
Detector: High Res Mass Spec (HRMS) @ 325 °C
Note: Used a PTV in Solvent Vent Mode with temperature program to 320 °C in 2 min

Comparative separations may not be representative of all applications.

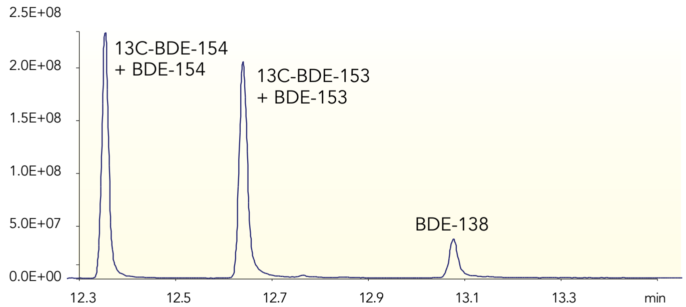
Environmental Contaminants

PBDEs by GC/HRMS

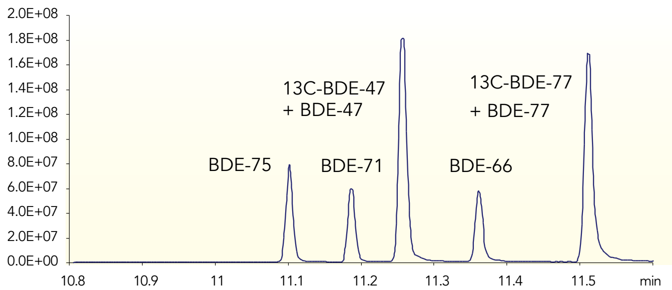
Tribromophenyl ethers



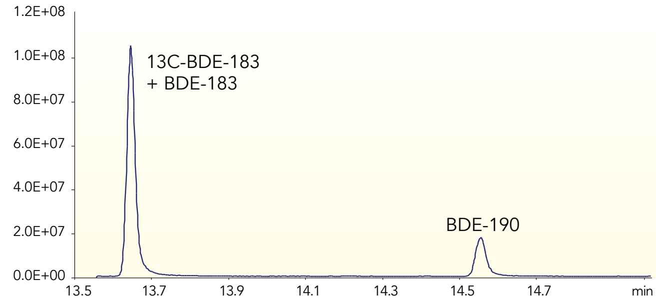
Hexabromophenyl ethers



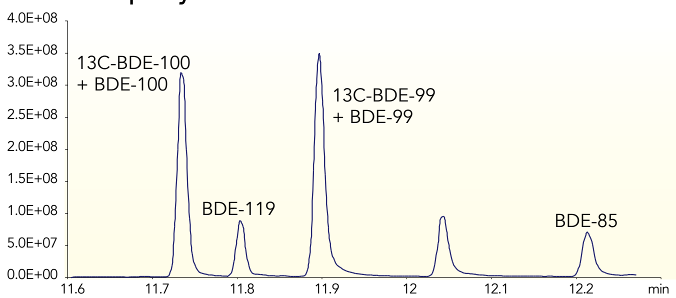
Tetrabromophenyl ethers



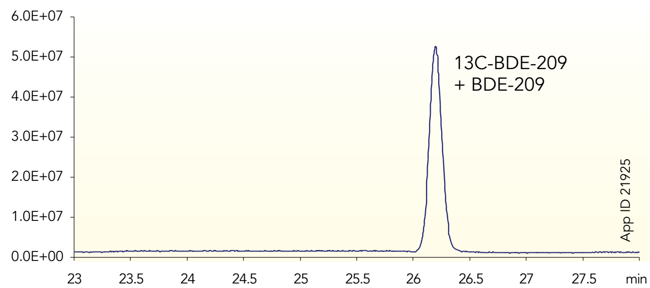
Heptabromophenyl ethers



Pentabromophenyl ethers



Decabromophenyl ether



“We have had great difficulties with the stability of BDE-209 with our previous GC columns, and we were forced to use a very short column (6 m) for this specific compound instead of a regular 20-30 m column. To be able to run all PBDEs in one run we decided to test Zebron™ ZB-SemiVolatiles.

With a narrow bore 20 m x 0.18 mm ID x 0.18 µm film ZB-SemiVolatiles column we are now able to successfully analyze our suite of PBDEs from BDE-28 to BDE-209 in a single run. Peak height of BDE-209 with this column is 10-30 times higher than with a brand new column of similar (5 % phenyl) chemistry and dimensions from another well-known manufacturer. Use of ZB-SemiVolatiles roughly halves the time required for analysis as there is no longer a need for a second injection with a shorter column.

ZB-SemiVolatiles represents a major improvement in the GC analysis of highly brominated flame retardants.”

Panu Rantakokko
National Institute for Health and Welfare, Finland

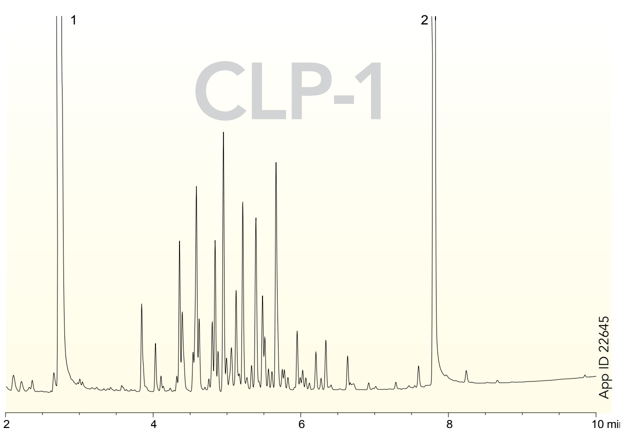
Environmental Contaminants

Polychlorinated Biphenyls (PCBs)

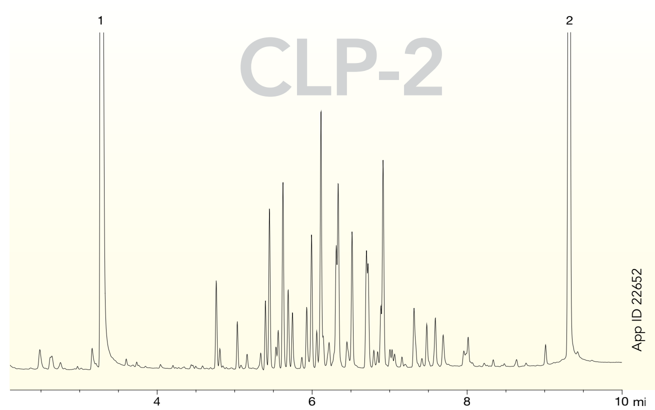
PCBs are commonly tested in addition to chlorinated pesticides, and are analyzed either as individual congeners, or as Aroclor mixtures. A number of methods for PCB analysis exist, including EN 1528, AOAC 970.52, ISO 10382, and EPA 1668 and 8082, among others. Some congeners have toxicity characteristics similar to 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD). These "dioxin-like" PCBs have been assigned Toxic Equivalency Factors (TEF) relative to the 2,3,7,8-TCDD isomer. Aroclor mixtures are first qualitatively identified by their unique fingerprint in comparison to a standard. Any contaminants present in the run may interfere with Aroclor fingerprints, making data analysis difficult.

PCBs by Dual-Column GC/ECD

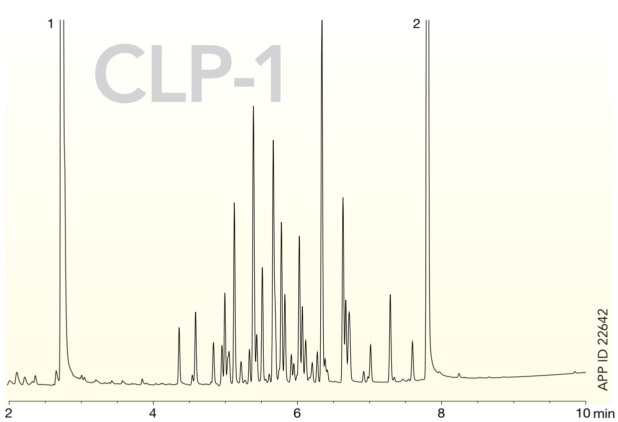
Aroclor 1254



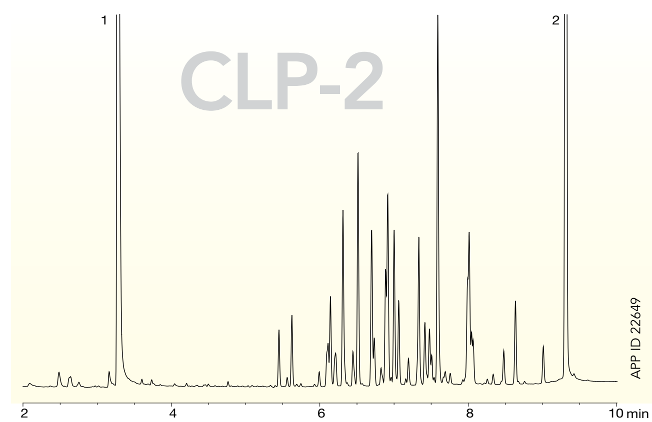
Aroclor 1254



Aroclor 1260



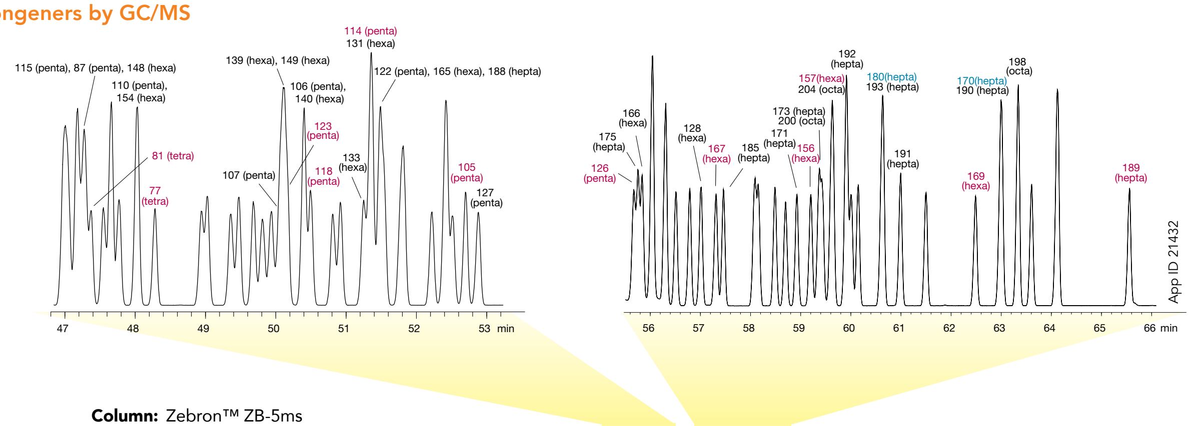
Aroclor 1260



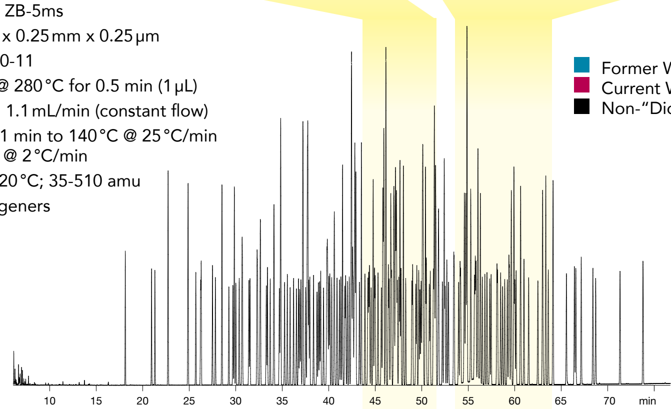
Conditions for all columns:

Columns: As listed
Dimensions: 30 meter x 0.25 mm x 0.25 µm (ZB-CLPesticides-1)
30 meter x 0.32 mm x 0.32 µm (ZB-CLPesticides-2)
Part Number: 7HM-G028-51 (ZB-CLPesticides-1)
7HM-G029-11 (ZB-CLPesticides-2)
Injection: Pulsed Splitless @ 30 psi (hold 20 sec) @ 250 °C, 1 µL
Carrier Gas: Helium @ 60 cm/sec (constant flow)
Oven Program: 120 °C to 200 °C @ 45 °C/min to 330 °C @ 15 °C/min for 2 min
Detector: ECD @ 330 °C
Y-Connector: AG0-4717 (Borosilicate Glass)
Guard Column: 7AM-G000-00-GZ0 (5 m Z-Guard™)
Liner: AG0-8499 (Single Taper with Wool at Bottom)
Septum: AG0-4696 (PhenoRed™-400)
Inlet Seal: AG0-8620 (Gold-Plated Easy Seals™)
Sample: Aroclor is 1000 ng/mL and SS and IS are 100 ng/mL in hexane.
1. Tetrachloro-meta-xylene (TCMX) (surrogate standard)
2. Decachlorobiphenyl (internal standard)

PCB Congeners by GC/MS

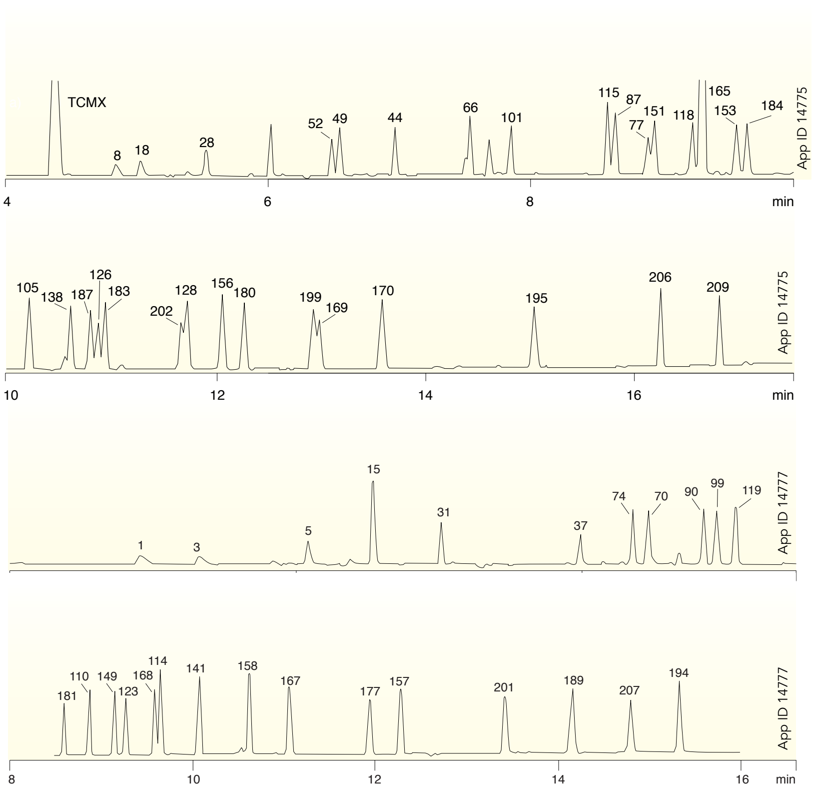


Column: Zebron™ ZB-5ms
Dimensions: 60 meter x 0.25 mm x 0.25 µm
Part No.: 7KG-G010-11
Injection: Splitless @ 280 °C for 0.5 min (1 µL)
Carrier Gas: Helium @ 1.1 mL/min (constant flow)
Oven Program: 60 °C for 1 min to 140 °C @ 25 °C/min to 290 °C @ 2 °C/min
Detector: MSD @ 320 °C; 35-510 amu
Sample: PCB Congeners



- Former WHO Toxic Congener Under 1994 Report
- Current WHO Toxic Congener Under 1997 Report
- Non-"Dioxin-Like" PCB Congener

PCB Congeners by GC/ECD



Conditions for both columns:
Column: As listed
Dimensions: 30 meter x 0.32 mm x 0.25 µm
Part No.: 7HM-G004-11 (ZB-50)
Injection: Splitless @ 225 °C, 1.0 µL
Carrier Gas: Helium @ 2.5 mL/min (constant flow)
Oven Program: 130 °C to 230 °C @ 20 °C/min to 270 °C @ 4 °C/min to 300 °C @ 20 °C/min (hold 1min)
Detector: ECD @ 325 °C
Sample: PCB Congeners

Application Spotlight

Analysis of Dioxins, Dibenzofurans, and Polychlorinated Biphenyls from Animal Feed and Tissue Using High Resolution Gas Chromatography (HRGCMS)

Introduction

Dioxins (PCDDs) are a particularly toxic class of priority organic pollutants (POPs) that are extremely persistent in the environment. They enter our food chain primarily through combustion of organic materials in waste incineration. Several other classes of POPs including dibenzofurans (PCDFs) and polychlorinated biphenyls (PCBs) display dioxin-like biological activity and are often monitored in conjunction with dioxins to give an overall toxic equivalent (TEQ) for a sample.

The European Union (EU) has published regulatory limits for the presence of these dioxin-like compounds in food and feed products. Recent contamination of 2,256 tons of fat incorporated in feed products in Germany has once again drawn attention to the need for routine monitoring. Dioxins are highly fat soluble and tend to bioaccumulate as they work their way up the food chain, posing a health risk to anyone consuming products from animals that were fed contaminated feed.

Robust analytical testing procedures are needed to determine potential threats in both feed and animal fat. In this article we discuss a rapid extraction and analysis procedure that allows all dioxin-like compounds to be determined in a single test. This methodology was used to analyze non-organic chicken feed as well as Mississippi River fish. The same methodology has also been applied to fish from the Great Lakes and shrimp collected internationally, though the data is not presented here.

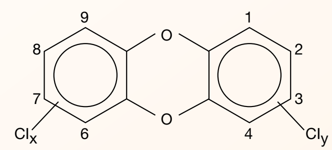
Methods and Materials

A 10 gram sample aliquot was spiked with labeled internal standards and extracted for 16 hours in toluene. The extracts were cleaned up and analyzed for PCDDs, PCDFs and PCBs using high resolution mass spectrometry (HRMS). PCDD and PCDF analysis was performed using a Zebron™ ZB-5ms 60 meter x 0.25 mm x 0.25 µm (p/n: 7KG-G010-11) GC column in accordance with US EPA Method 1613B. PCB analysis was performed using a Zebron ZB-1 60 meter x 0.25 mm x 0.25 µm (p/n: 7KG-G001-11) GC column in accordance with US EPA Method 1668A.

Results and Discussion

The general dioxin structure has two benzene rings connected by two oxygen atoms and substituted at various positions with one to eight chlorine atoms (**Figure 1**). The position of the chlorine atoms around the two benzene rings determines the toxicity of the isomer with the tetra and penta chlorinated dioxin (TCDD/PeCDD) substituted at the 2, 3, 7, and 8 positions being the most toxic. Penta through Octa substituted isomers with chlorines at the 2,3,7,8 position are assigned toxicity equivalent factor (TEF) relative to the 2,3,7,8-TCDD isomer (**Table 1**). Similar TEF values have also been determined for other dioxin-like compounds. The relative toxicity or toxic equivalent (TEQ) of a sample can then be determined by summing the concentration of each congener multiplied by its TEF value (**Equation 1**).

Figure 1: General Structure for a Dioxin Isomer

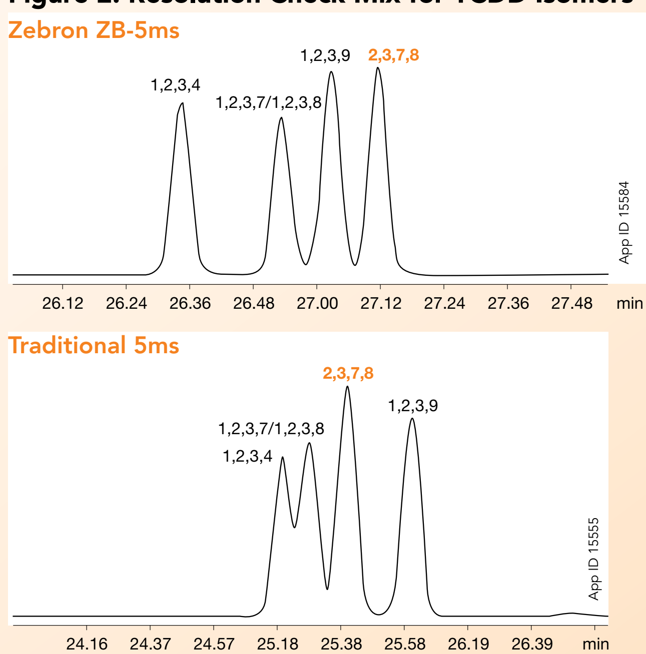


Equation 1:

TEQ = ∑ Ci x I-TEFi, where Ci is the concentration of a 2,3,7,8 substituted dioxin isomer

The analysis of these dioxin-like compounds was done using a gas chromatograph (GC) connected to an HRMS. This allowed for extremely high resolution between dioxin isomers, which allowed for accurate quantitation. However, even the advanced HRMS is unable to differentiate between isomers having the same degree of chlorination. This requires a high efficiency GC column capable of resolving compounds with the same chemical properties, but slightly different geometry. The Zebron ZB-5ms column uses an arylene matrix bonding technology that significantly improves resolution between dioxin isomers **(Figure 2).**

Figure 2: Resolution Check Mix for TCDD Isomers



Using this optimized procedure, we compared the TEQ for fish samples from the Southern Mississippi river. There was very little, if any, difference between farm-raised and wild-caught fish **(Table 2).** This may be due to downstream effects, the possibility of point sources near to where the fish were collected, and even the age of the fish collected. For the Mississippi River, tissue levels were on average, lower than expected. This may be due to several factors including the larger volume of water in this river and the rapid currents compared to those of smaller and shallower rivers[1].

We then applied this methodology to a non-organic chicken feed used by various farms near Vista Analytical Laboratory (**Table 3**). We only observed three of the World Health Organization (WHO) dioxin-like compounds: 1,2,3,4,6,7,8-HpCDD, OCDD, and PCB 118. The calculated TEQ value for the sample was 0.00461, which was well below the EU maximum. These levels are a consistent continuation of the downward trend of PCDD/F and PCB levels in food products initially reported by several researchers in the mid-1990s.[2,3,4]

Table 1: Toxic Equivalency Factors for Dioxin-Like Compounds

Congener	WHO-TEF 2005	Congener	WHO-TEF 2005
2,3,7,8-TCDD	1	**PCB-77**	0.0001
1,2,3,7,8-PeCDD	1	**PCB-81**	0.0003
1,2,3,4,7,8-HxCDD	0.1	**PCB-105**	0.00003
1,2,3,6,7,8-HxCDD	0.1	**PCB-114**	0.00003
1,2,3,7,8,9-HxCDD	0.1	**PCB-118**	0.00003
1,2,3,4,6,7,8-HpCDD	0.01	**PCB-123**	0.00003
OCDD	0.0003	**PCB-126**	0.1
2,3,7,8-TCDF	0.1	**PCB-156**	0.00003
1,2,3,7,8-PeCDF	0.03	**PCB-157**	0.00003
2,3,4,7,8-PeCDF	0.3	**PCB-167**	0.00003
1,2,3,4,7,8-HxCDF	0.1	**PCB-169**	0.03
1,2,3,6,7,8-HxCDF	0.1	**PCB-189**	0.00003
2,3,4,6,7,8-HxCDF	0.1		
1,2,3,7,8,9-HxCDF	0.1		
1,2,3,4,6,7,8-HpCDF	0.01		
1,2,3,4,7,8,9-HpCDF	0.01		
OCDF	0.0003		

Table 2: TEQ Values for Fish Caught in the Mississippi River

	Samples (N)	Mean TEQ (pg/g)	Range
Wild-Caught	33	1.50	0.13 - 4.96
Farm-Raised	31	0.98	0.15 - 2.56

Conclusion

Thanks to aggressive regulatory action, the level of dioxin-like compounds in the environment is on the decline. Most high level samples are the result of remediation efforts or accidents, such as was observed in Germany. When such testing is required, testing laboratories must use the most advanced techniques available in order to quickly and accurately determine the potential risk to the general population.

Table 3: Dioxin-Like Compounds Found in Non-Organic Chicken Feed

Dioxin-Like Compound	Concentration (pg/g)
2,3,7,8-TCDD	ND
1,2,3,7,8-PeCDD	ND
1,2,3,4,7,8-HxCDD	ND
1,2,3,6,7,8-HxCDD	ND
1,2,3,7,8,9-HxCDD	ND
1,2,3,4,6,7,8-HpCDD	0.421
OCDD	1.05
2,3,7,8-TCDF	ND
1,2,3,7,8-PeCDF	ND
2,3,4,7,8-PeCDF	ND
1,2,3,4,7,8-HxCDF	ND
1,2,3,6,7,8-HxCDF	ND
2,3,4,6,7,8-HxCDF	ND
1,2,3,7,8,9-HxCDF	ND
1,2,3,4,6,7,8-HpCDF	ND
1,2,3,4,7,8,9-HpCDF	ND
OCDF	ND
PCB-77	ND
PCB-81	ND
PCB-105	ND
PCB-114	ND
PCB-118	2.90
PCB-123	ND
PCB-126	ND
PCB-156	ND
PCB-157	ND
PCB-167	ND
PCB-169	ND
PCB-189	ND
TEQ	0.00461

References

1. Ferriby, LL, Luksemburg, W, Paustenbach, D, Birnbaum, LS, and Harris, MA. Comparing PCDDs, PCDFs, and dioxin-like PCBs in farmed-raised and wildcaught catfish from Southern Mississippi, Organohalogen Compounds Vol 68 (2006)
2. Furst P, Wilmers K. Organohalogen Comp 1995; 26:101-104.
3. Liem AKD, de Jong APJM, Theelen RMC, van Zorge JH. Occurrence of dioxins and related compounds in Dutch foodstuffs – Part I: Sampling strategy and analytical results. 11th International Symposium on Chlorinated Dioxins and Related Compounds, Research Triangle Park, NC, Abstract P-156: 1991, p. 365
4. Mayer R. Organohalogen Comp 1995; 26:109-111.

Ordering Information

Use the selection tables to locate the Zebron™ columns for your application. Below is a list of the most popular dimensions. Contact your GC Specialist or visit **www.phenomenex.com/GC** for more.

Phase	ID (mm)	df (µm)	10 meter	15 meter	30 meter	60 meter
ZB-SemiVolatiles 5% phenyl-arylene phase specially deactivated for supreme inertness to acids, neutrals, and amines	0.25	0.25			7HG-G027-11	7KG-G027-11
	0.25	0.50			7HG-G027-17	
ZB-1 Low polarity phase for boiling point separations, essential oils, and flavor volatiles	0.25	0.10		7EG-G001-02	7HG-G001-02	7KG-G001-02
	0.25	0.25		7EG-G001-11	7HG-G001-11	7KG-G001-11
	0.25	0.50			7HG-G001-17	
	0.25	1.00		7EG-G001-22	7HG-G001-22	7KG-G001-22
	0.32	0.10				7KM-G001-02
	0.32	0.25		7EM-G001-11	7HM-G001-11	7KM-G001-11
	0.32	0.50			7HM-G001-17	
	0.32	1.00		7EM-G001-22	7HM-G001-22	7KM-G001-22
	0.32	3.00			7HM-G001-36	7KM-G001-36
	0.32	5.00			7HM-G001-39	
	0.53	0.15		7EK-G001-05		
	0.53	0.50		7EK-G001-17	7HK-G001-17	
	0.53	1.50		7EK-G001-28	7HK-G001-28	7KK-G001-28
	0.53	2.65	7CK-G001-35			
	0.53	3.00		7EK-G001-36	7HK-G001-36	
	0.53	5.00		7EK-G001-39	7HK-G001-39	
ZB-1ms Very low bleed for GC/MS analyses; recommended for fragrance allergens and sulfur in beer	0.10	0.10	7CB-G011-02			
	0.18	0.18	7CD-G011-08			
	0.25	0.10			7HG-G011-02	
	0.25	0.25		7EG-G011-11	7HG-G011-11	7KG-G011-11
	0.25	0.50			7HG-G011-17	
	0.25	1.00			7HG-G011-22	7KG-G011-22
	0.32	0.25		7EM-G011-11	7HM-G011-11	
	0.32	1.00			7HM-G011-22	7KM-G011-22
	0.53	1.00			7HK-G011-22	
ZB-5 Rugged, low bleed phase for a variety of applications; recommended for additives and preservatives (tocopherols in food)	0.25	0.10		7EG-G002-02	7HG-G002-02	7KG-G002-02
	0.25	0.25		7EG-G002-11	7HG-G002-11	7KG-G002-11
	0.25	0.50		7EG-G002-17	7HG-G002-17	7KG-G002-17
	0.25	1.00		7EG-G002-22	7HG-G002-22	7KG-G002-22
	0.32	0.10		7EM-G002-02	7HM-G002-02	
	0.32	0.25		7EM-G002-11	7HM-G002-11	7KM-G002-11
	0.32	0.50			7HM-G002-17	
	0.32	1.00		7EM-G002-22	7HM-G002-22	7KM-G002-22
	0.53	0.50		7EK-G002-17	7HK-G002-17	
	0.53	1.50		7EK-G002-28		
	0.53	1.50			7HK-G002-28	7KK-G002-28
	0.53	3.00		7EK-G002-36	7HK-G002-36	
	0.53	5.00			7HK-G002-39	
ZB-5ms Stable, low bleed 5% phenyl-arylene phase for GC and GC/MS; recommended for a variety of food safety applications	0.10	0.10	7CB-G010-02			
	0.18	0.18	7CD-G010-08			
	0.25	0.25		7EG-G010-11	7HG-G010-11	7KG-G010-11
	0.25	0.50			7HG-G010-17	
	0.25	1.00			7HG-G010-22	
	0.32	0.25			7HM-G010-11	7KM-G010-11
	0.32	0.50			7HM-G010-17	
	0.32	1.00			7HM-G010-22	
ZB-5HT Inferno™ Robust fused silica phase stable to 430 °C; excellent for testing of triglycerides and sterols	0.25	0.10		7EG-G015-02	7HG-G015-02	
	0.25	0.25		7EG-G015-11	7HG-G015-11	
	0.32	0.10		7EM-G015-02	7HM-G015-02	
	0.32	0.25		7EM-G015-11	7HM-G015-11	
	0.53	0.15		7EK-G015-05	7HK-G015-05	

guarantee

If Zebron columns do not provide you with equivalent or better separations as compared to any other GC column of the same phase and comparable dimensions, send in your comparative data within 45 days and keep the column for FREE!

Ordering Information

Phase	ID (mm)	df (µm)	10 meter	15 meter	30 meter	60 meter
ZB-35 Rugged, inert, intermediate polarity phase for PAHs; separate benzo[b], [j], and [k]fluoranthene isomers	0.25	0.25		7EG-G003-11	7HG-G003-11	7KG-G003-11
	0.25	0.50		7EG-G003-17	7HG-G003-17	
	0.32	0.25			7HM-G003-11	7KM-G003-11
	0.53	0.50			7HK-G003-17	
	0.53	1.00		7EK-G003-22	7HK-G003-22	
ZB-50 Inert high polarity column with temperature limits up to 340 °C; recommended for triazine pesticides, phenolic antioxidants, and amino acids	0.25	0.15		7EG-G004-05		
	0.25	0.25		7EG-G004-11	7HG-G004-11	7KG-G004-11
	0.25	0.50			7HG-G004-17	7KG-G004-17
	0.32	0.25		7EM-G004-11	7HM-G004-11	
	0.32	0.50		7EM-G004-17	7HM-G004-17	
	0.53	1.00		7EK-G004-22	7HK-G004-22	
ZB-624 Specifically designed for VOC separations, including food packaging volatiles and flavor/fragrance volatiles	0.25	1.40			7HG-G005-27	7KG-G005-27
	0.32	1.80			7HM-G005-31	7KM-G005-31
	0.53	3.00			7HK-G005-36	7KK-G005-36
ZB-WAX Bonded, solvent rinseable phase excellent for separating polar complex mixtures and essential oils	0.10	0.10	7CB-G007-02			
	0.25	0.15			7HG-G007-05	7KG-G007-05
	0.25	0.25		7EG-G007-11	7HG-G007-11	7KG-G007-11
	0.25	0.50			7HG-G007-17	7KG-G007-17
	0.25	1.00			7HG-G007-22	
	0.32	0.15			7HM-G007-05	
	0.32	0.25		7EM-G007-11	7HM-G007-11	7KM-G007-11
	0.32	0.50		7EM-G007-17	7HM-G007-17	7KM-G007-17
	0.53	0.50			7HK-G007-17	
	0.53	1.00		7EK-G007-22	7HK-G007-22	7KK-G007-22
ZB-WAX*PLUS*™ 100 % aqueous stable with high retention of alcohols and chlorinated solvents; recommended for FAMEs, alcoholic beverages, flavors, and food safety applications	0.10	0.10	7CB-G013-02			
	0.25	0.25		7EG-G013-11	7HG-G013-11	7KG-G013-11
	0.32	0.25			7HM-G013-11	7KM-G013-11
	0.32	0.50			7HM-G013-17	7KM-G013-17
	0.53	1.00		7EK-G013-22	7HK-G013-22	7KK-G013-22
ZB-FFAP High polarity nitroterephthalic acid modified PEG phase with good peak shape for underivatized acids; especially suited for organic acids, free fatty acids, and alcohols	0.25	0.25		7EG-G009-11	7HG-G009-11	7KG-G009-11
	0.32	0.25		7EM-G009-11	7HM-G009-11	
	0.32	0.50		7EM-G009-17	7HM-G009-17	
	0.32	1.00			7HM-G009-22	
	0.53	1.00		7EK-G009-22	7HK-G009-22	
ZB-MultiResidue™-1 Specially designed for pesticide, herbicide, and insecticide separations; suited for analysis of TMS sugars and borneol additives	0.25	0.25			7HG-G016-11	
	0.32	0.50			7HM-G016-17	
	0.53	0.50			7HK-G016-17	
ZB-MultiResidue-2 Specially designed for pesticide, herbicide, and insecticide separations with orthogonal selectivity to ZB-MultiResidue-1	0.25	0.20			7HG-G017-10	
	0.32	0.25			7HM-G017-11	
	0.53	0.50			7HK-G017-17	
ZB-XLB Alternative selectivity to 5-type phases; suited for testing of essential oils	0.18	0.18	7CD-G019-08			
	0.25	0.25		7EG-G019-11	7HG-G019-11	7KG-G019-11
	0.25	0.50			7HG-G019-17	
	0.32	0.25			7HM-G019-11	
	0.32	0.50			7HM-G019-17	
ZB-XLB-HT Inferno™ Enhanced durability with 400 °C temperature stability; recommended for food safety testing of melamine, cyanuric acid, and PCBs	0.25	0.10		7EG-G024-02	7HG-G024-02	
	0.25	0.25		7EG-G024-11	7HG-G024-11	7KG-G024-11
	0.32	0.10		7EM-G024-02		
	0.32	0.25			7HM-G024-11	
ZB-Bioethanol Designed for fast, accurate ethanol analysis	0.25	1.00		7EG-G020-22	7HG-G020-22	

guarantee

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Sensitive, Reproducible Results for Food Testing

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www.phenomenex.com/FoodGC

Australia
t: +61 (0)2-9428-6444
f: +61 (0)2-9428-6445
auinfo@phenomenex.com

Austria
t: +43 (0)1-319-1301
f: +43 (0)1-319-1300
anfrage@phenomenex.com

Belgium
t: +32 (0)2 503 4015 (French)
t: +32 (0)2 511 8666 (Dutch)
f: +31 (0)30-2383749
beinfo@phenomenex.com

Canada
t: +1 (800) 543-3681
f: +1 (310) 328-7768
info@phenomenex.com

China
t: +86 (0)20 2282-6668
f: +86 (0)20 2809-8130
chinainfo@phenomenex.com

Denmark
t: +45 4824 8048
f: +45 4810 6265
nordicinfo@phenomenex.com

Finland
t: +358 (0)9 4789 0063
f: +45 4810 6265
nordicinfo@phenomenex.com

France
t: +33 (0)1 30 09 21 10
f: +33 (0)1 30 09 21 11
franceinfo@phenomenex.com

Germany
t: +49 (0)6021-58830-0
f: +49 (0)6021-58830-11
anfrage@phenomenex.com

India
t: +91 (0)40-3012 2400
f: +91 (0)40-3012 2411
indiainfo@phenomenex.com

Ireland
t: +353 (0)1 247 5405
f: +44 1625-501796
eireinfo@phenomenex.com

Italy
t: +39 051 6327511
f: +39 051 6327555
italiainfo@phenomenex.com

Luxembourg
t: +31 (0)30-2418700
f: +31 (0)30-2383749
nlinfo@phenomenex.com

Mexico
t: 001-800-844-5226
f: 001-310-328-7768
tecnicomx@phenomenex.com

The Netherlands
t: +31 (0)30-2418700
f: +31 (0)30-2383749
nlinfo@phenomenex.com

New Zealand
t: +64 (0)9-4780951
f: +64 (0)9-4780952
nzinfo@phenomenex.com

Norway
t: +47 810 02 005
f: +45 4810 6265
nordicinfo@phenomenex.com

Puerto Rico
t: +1 (800) 541-HPLC
f: +1 (310) 328-7768
info@phenomenex.com

Spain
t: +34 91-413-8613
f: +34 91-413-2290
espinfo@phenomenex.com

Sweden
t: +46 (0)8 611 6950
f: +45 4810 6265
nordicinfo@phenomenex.com

United Kingdom
t: +44 (0)1625-501367
f: +44 (0)1625-501796
ukinfo@phenomenex.com

USA
t: +1 (310) 212-0555
f: +1 (310) 328-7768
info@phenomenex.com

All other countries
Corporate Office USA
t: +1 (310) 212-0555
f: +1 (310) 328-7768
info@phenomenex.com

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