



Petrochemical Gas Chromatographs

● Analyzer Solutions



Scion Instruments has the experience and know-how to provide pre-configured gas chromatographs that are ready at power up to handle your key applications. Our long experience in designing, configuring and manufacturing complete systems, with all their analytical benefits, ensures you get the solution that's right for you. With a host of standard solutions configured to meet the performance specifications outlined in international methods, and the capability to produce unique, tailor-made solutions, we have the answer that you seek.

Scion Analyzer Solutions

Benefits



Scion configures and tests GC hardware and software according to widely used industry standard methods (e.g. ASTM, UOP, EN, ISO, GPA,...), to save its clients time and to ensure confidence in results. Solutions are configured to meet the performance specifications outlined in the set method itself.

Included with all Scion Analyzer solutions:

- All Hardware
- Software (incl. special "plug-ins" where appropriate)
- Pre-Installed methods
- Test Chromatograms
- Installation/ Validation Data
- Trouble Shooting Guide
- User documentation customized for the specific method

Scion Simulated Distillation Analyzers

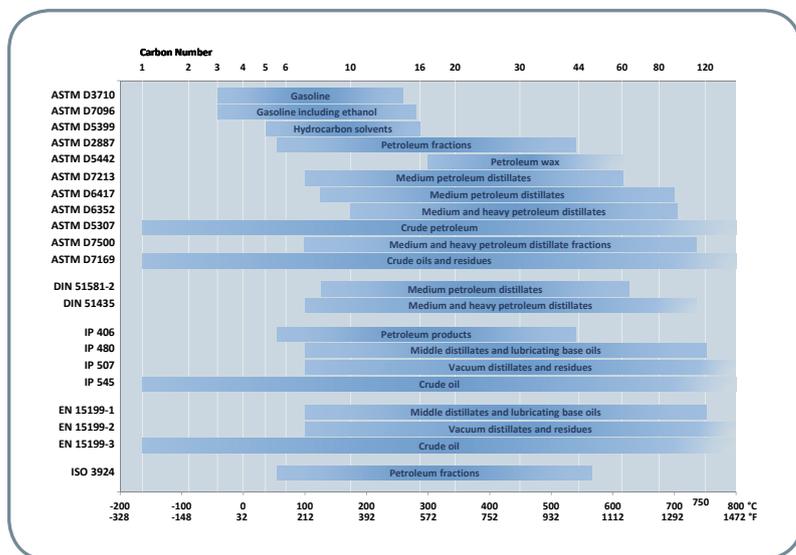


Table 1: Overview Simdist methods.

A gas chromatographic (GC) technique, Simulated Distillation (SimDist) reproduces the physical distillation of petroleum materials and products by determining boiling point distribution. Scion Instrument's range of Simulated Distillation Analyzers are designed to meet all industry standard methods, Bruker's analyzer software includes both ASTM D86 and ASTM D1160 correlations. Scion's highly automated GC, CompassCDS Chromatography Data Handling Software, and integrated SimDist software are also designed to meet worldwide industry standard test methods.



436-GC with Sampler

Key Benefits Include:

- Accurate boiling point distribution up to 750°C
- Integrated standard test methods, applications fully comply with ASTM, IP, DIN and ISO standard test methods
- Complete, single vendor solution
- Complete control from initial setup to final report
- ASTM D86 and ASTM D1160 correlation

Built-in Reports:

Scion's SimDist software provides a wide variety of report options to meet specific requirements including;

- Chromatogram with merged corrected blank analysis and IBP/FBP marks versus retention time
- Boiling point versus percentage of sample
- Table and plot with retention time versus boiling point
- D86 and D1160 correlations
- DIN Noak and motor oil volatility reports
- Table with cut points and fractions plus residue analysis with recovery calculation up to C120

Hydrocarbon Analysis by Group (PIONA+™)



Figure 1: Traps are easily accessible and do not require any tools to install or replace.

Characterization of Engine Fuels by Hydrocarbon Group Type

Scion's PIONA+™ Analyzer is a highly flexible GC analysis platform to obtain comprehensive characterization and quantitative information, including hydrocarbon group types, oxygenates and carbon number distribution for spark ignition engine fuels.

Key Analyzer Capabilities:

- Unparalleled operational flexibility
- Compliant with established standards
- A complete and fully integrated solution
- A powerful analyzer, easy to use, generating outstanding analysis results day after day

The PIONA+ Analyzer performs a complete analysis (as described in ASTM D6839 and similar methods) and provides unprecedented analytical flexibility and simplified operation through the use of a novel approach to column/trap heating and exchange (Figure 1).

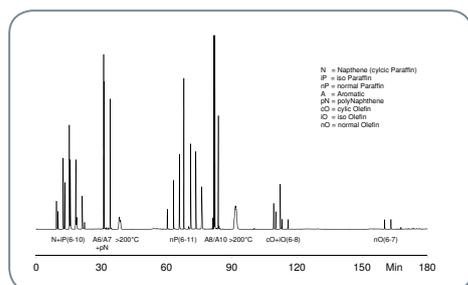


Figure 2: Chromatogram of a test mix in conventional PIONA mode. (Analysis Time - 180mins)

Reduced Analysis Time and Increased Sample Throughput Efficiency through the Use of "Concurrent" Heating

A unique aspect of the design of the Scion PIONA+ system is the ability to independently heat the individual traps.

This has 2 major operational benefits:

- Enhanced elution integrity for wide range sample
- Significantly Reduced Analysis Time

The independent and concurrent heating design permits greater trap control and benefits in improved elution integrity of the component groups e.g naphthene, iso-paraffins and n-paraffins even for a wide range sample (C4 - C11). In addition, only a single Molsieve column temperature cycle is employed thus reducing the analysis time by almost half allowing a "fast" PIONA mode of operation (see figures 2 and 3).

By employing this technique, sample throughput can be nearly doubled compared to systems that do not offer this unique capability.

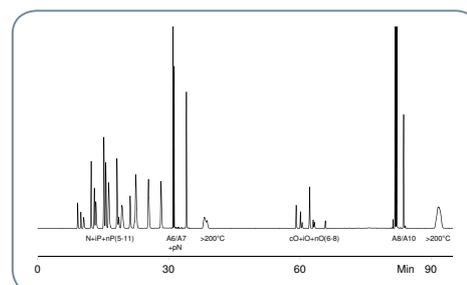


Figure 3: Chromatogram of a test mix using concurrent heating in "fast" PIONA mode. (Analysis Time = 95mins)

Determining Total Olefin Content Is Now Practical

The stability, sample loading and lifetime for all of the critical chromatographic components have been improved and optimized in the Scion PIONA+ Analyzer. Of special and particular note is the improvement in increasing the sample loading capacity of the "olefin" trap. As a result, it is now possible to analyze streams with olefin content as high as 35-40% or more. This makes it practical to employ a single analytical method to obtain total olefin content (Table 2).

Table 2

Carbon	Saturates			Unsaturates			Aromatics	Oxygenates	Total
	Cyclic	Iso	Normal	Cyclic	Iso	Normal			
3	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
4	0.00	0.00	0.06	0.00	0.03	0.54	0.00	0.00	0.63
5	0.31	11.37	2.98	0.87	9.92	7.58	0.00	0.00	33.03
6	3.19	9.98	1.40	2.40	8.59	4.40	1.72	0.00	31.68
7	4.31	6.77	0.00	2.14	4.76	1.91	7.47	0.00	27.36
8	1.42	3.12	0.00	0.39	2.06	0.00	0.07	0.00	7.06
9	0.01	0.00	0.00	0.04	0.02	0.00	0.03	0.00	0.10
10	0.01	0.00	0.00	0.09	0.00	0.03	0.01	0.00	0.14
11	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Total	9.25	31.24	4.44	5.93	25.38	14.46	9.30	0.00	100.00

Example weight % report for a Naphtha sample with high (46%) olefinic content; (highlighted in blue).

The analyzer design allows the operator to conduct analyses in any one of a number of different operational modes including PNA, PONA, PIONA, O-PONA and O-PIONA in standard and concurrent heating configuration. The system is compliant with established standard methods (see adjacent array).

	PNA	PONA	PIONA	O-PONA	O-PIONA
EN 14517					✓
EN-ISO-22854					✓
ASTM D6839				✓	
DIN 51448-2			✓		
ASTM D1319 (FIA)		✓			
DIN 51448-1	✓				
ASTM D5443	✓				
UOP 870	✓				
IP 382	✓				



Compliant with the method



More information generated than required for the method

Detailed Hydrocarbon Analyzer

The DHA Analyzer is a complete high resolution GC solution for the analysis of hydrocarbons in petroleum streams. It is capable of performing all of the standard methods including ASTM D6729, D6730, D6733, D5134, D6623, IP 344/ DHA "Front End" and "Fast DHA".

Although each DHA analyzer is configured, tested and certified at the factory for a standard method specified by the customer, the DHA software permits the operator to utilize any of the other popular standard methods as well. And, because of the outstanding performance and flexibility of the Scion GC and CompassCDS software design, Scion is able to quickly modify the existing methods or add new ones if required as a result of the on-going dynamic industry standard processes.

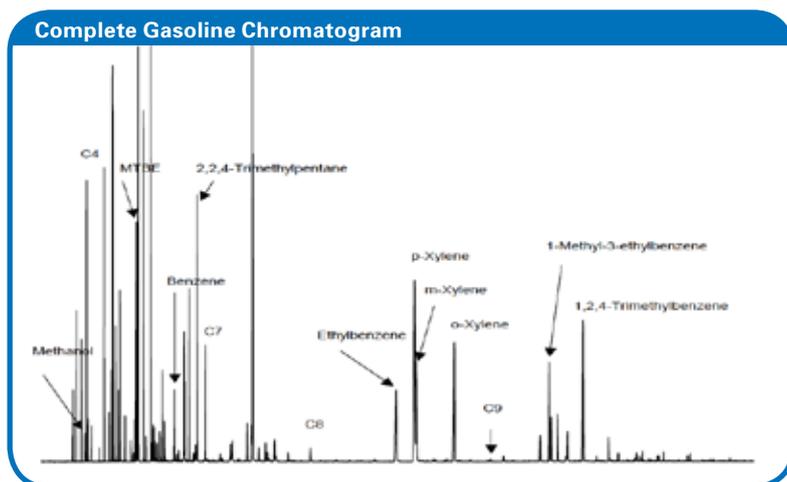


Figure 4: The analysis of permanent gases and hydrogen using the Rapid RGA.

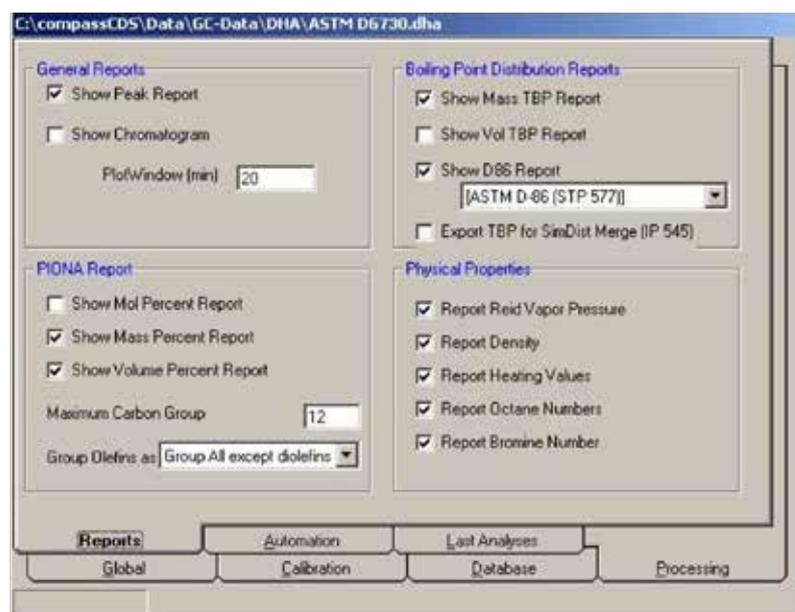


Figure 5: Report selection output.

Scion Refinery Gas Analyzers

Peak Identification

1. Hydrogen
2. Carbon Dioxide
3. Hydrogen Sulfide
4. Oxygen
5. Nitrogen
6. Carbon Monoxide
7. Methane
8. Ethane
9. Ethylene
10. Propane
11. Cyclo Propane
12. Propylene
13. i-Butane
14. n-Butane
15. Propadiene
16. Acetylene
17. t-2-Butene
18. i-Butene
19. c-2-Butene
20. i-Pentane
21. n-Pentane
22. 1, 3-Butadiene
23. Propyne
24. Butyne
25. C6+
26. Helium

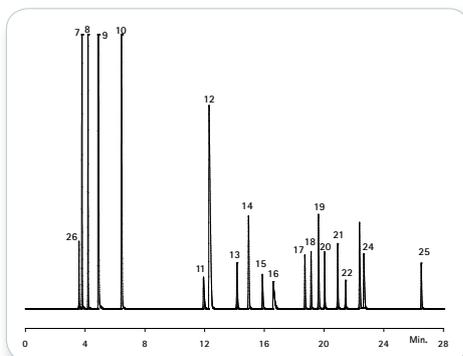


Figure 6: The separation of light hydrocarbons using the Standard RGA.

The source and composition of refinery gases varies considerably. Measuring gas composition precisely and accurately is a significant challenge in today's refinery operations. Scion's Refinery Gas Analyzers (RGA) are designed to deliver superior, reliable results for a wide range of sources and analysis throughput requirements.

Key Benefits:

- Pre-configured and tuned
- Standard methods including UOP 539, DIN-51666 and ASTM D2163
- Integrated micro-gasifier ensures complete vaporization of LPGs and high pressure samples to prevent sample discrimination (option)
- Multi-channel approach

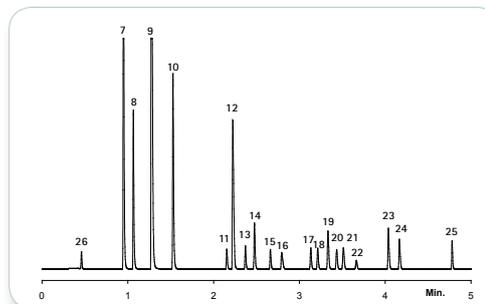


Figure 7: The analysis of light hydrocarbons using the Rapid RGA, with complete separation in less than five minutes.

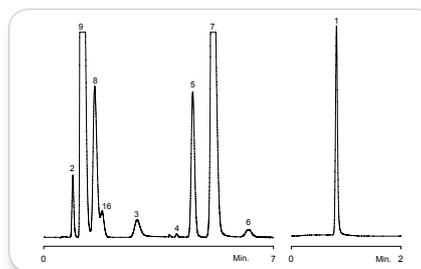


Figure 8: The analysis of permanent gases and hydrogen using the Rapid RGA.

Scion Offers Two RGA Systems to Meet the Widest Range of Analysis Requirements:

Standard RGA:

A three channel multi-valve design using both capillary and packed columns.

- Channel 1 - Analysis of permanent gases
- Channel 2 - Light hydrocarbons
- Channel 3 - Hydrogen.

Total analysis time for all components in 25 minutes.

Rapid RGA:

The Standard RGA packed columns in the hydrogen and permanent gas channels are replaced by micro packed columns and installed in a separate column oven. Key benefits of this design are:

- Flexibility
- Reduced Analysis Time - 5mins (with H₂S - 7mins)
- Increased Sample Throughput

Characteristics	Standard RGA	Rapid RGA
No. of Channels/Detectors Used	3	3
No. of Column Ovens	1	2
Analysis Time	25 min	5 min (7 min with H ₂ S)
Repeatability	<1%	<1%
Linear Bench Space Required	66 cm/26 in.	66 cm/26 in.
Minimum Component Detection Level	0.01% all components except H ₂ S = 0.05%	0.01% all components except H ₂ S = 0.05%
Suitability		
Typical Refinery Gas	Excellent	Excellent
Impurities in Bulk Ethylene	Excellent	Excellent
Impurities in Bulk Propylene	Excellent	Good
Impurities in Bulk C4	Good	Good

Table 3: Standard RGA vs Rapid RGA.

Low Level Oxygenates Analyzer

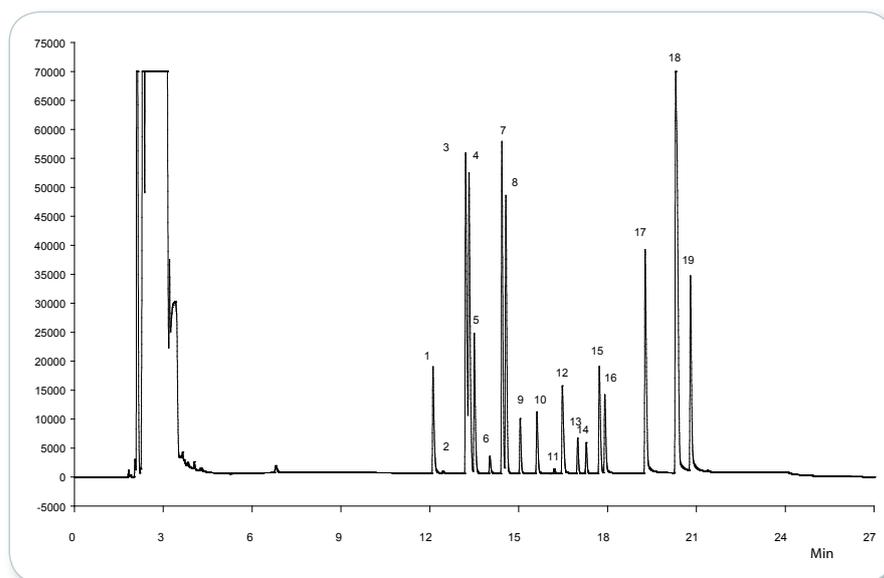


Figure 9: Typical chromatogram showing a wide range analysis of a liquid sample stream.

The determination of sub to high ppm levels of ethers, alcohols, aldehydes and ketones in different hydrocarbon matrices is a recurring challenge in the petroleum refining and petrochemical industry. The Scion Low Level Oxygenates Analyzer is an easy to use solution to meet this challenge and is according ASTM D7423.

The Low Level Oxygenates Analyzer is designed and optimized to quantify ppm and sub ppm levels of ethers (e.g. DME, MTBE, ETBE, DIPE), alcohols (e.g. methanol, ethanol, propanol), ketones (e.g. acetone, MEK) and aldehydes in various hydrocarbon matrices. In general, all oxygenated components with a boiling point of up to 100°C can be analyzed and the sample can be a gas, LPG or liquid under ambient conditions with a final boiling point up to 250°C.

The system is comprised of a Scion GC configured with gas and liquid sampling valves, two high performance capillary analysis columns, digitally controlled pneumatics including a 'fluidic' switch and Flame Ionization Detector (FID). An optional 'pressure station' can be added to eliminate the possibility of losing sample due to evaporation when analyzing LPG. The GC is controlled via the CompassCDS Chromatography Data Handling Software, which acquires data, processes it and generates analyses reports.

Peak Identification

1. Diethylether
2. Acetaldehyde
3. Ethyl tert. Butyl ether
4. Methyl tert. Butyl ether
5. Diisopropylether
6. Propanal
7. tert amyl methyl ether
8. Propylether
9. Isobutyraldehyde
10. Butyraldehyde
11. Methanol
12. Acetone
13. Isovaleraldehyde
14. Valeraldehyde
15. 2-Butanone
16. Ethanol
17. 1-Propanol
18. tert Butyl alcohol
& Isobutanol
19. 1-Butanol

Scion 4815 GC Oxygenates Analyzer

The Scion 4815 GC Analyzer provides a highly cost effective solution for the analysis of oxygenates in gasoline, according to the widely used industry standard method ASTM D4815. The combination of Scion's reliable GC hardware, powerful software and industry leading pre- and post-sales support teams make this analyzer package the most comprehensive solution available today.

Oxygenated compounds can be present in various hydrocarbon matrices either because they were purposely added (e.g. into gasoline), because they are naturally present, or formed during catalytic processes such as polymer production. In gasoline, oxygenated compounds are added as 'anti-knock' agents to increase the octane number and decrease emissions by replacing organo-lead compounds.

The type and concentration of oxygenated compounds must be measured in reformulated gasolines as part of ongoing product quality assessment, and to confirm the oxygenated components have been added in the correct amounts according to regulatory requirements (e.g. California Air Resources Board).

ASTM D4815 is frequently chosen as the standard method for the determination of oxygenated compounds. Individual ethers and alcohols are quantified in gasoline including: MTBE, ETBE, TAME, DIPE, C1-C4 alcohols and tert-amylalcohol. Individual ether components are measured from 0.1 to 20.0 mass %. The individual alcohols are measured from 0.1 to 12.0 mass %.



456-GC with Sampler

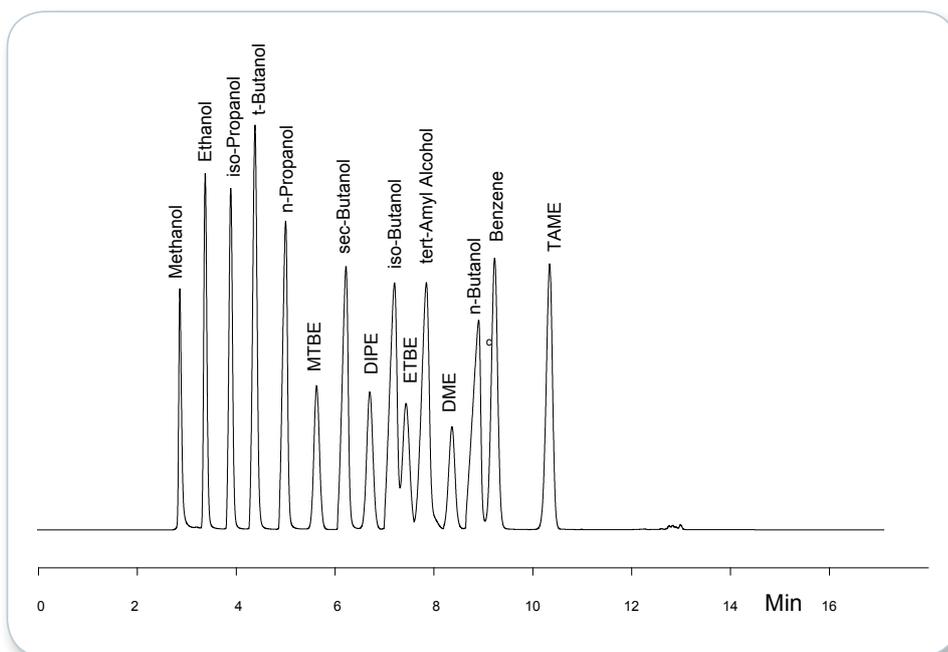


Figure 10 : Typical Chromatogram of the test sample.

Trace Impurity Analyzers

Sulfur Components in LPGs

Low level analysis of sulfur containing components such as H₂S, COS and mercaptanes is extremely challenging and a configured GC offers the solution.

Firstly, the system employs a micro-gasifier enabling the direct coupling of an LPG stream. Secondly, an inert steel sample path ensures a trouble free analysis of sulfur containing components at low concentrations. Finally, a two channel PFPD/ two column approach permits the analysis of all components of interest in one run whatever the LPG matrix. Two differing columns ensures quenching of PFPD signal by the matrix is overcome and full sulfur component analysis is achieved. Figures 11 and 12 show chromatograms obtained in a propane matrix and illustrates the novel benefits of the 2 channel approach.

Permanent Gases in LPGs

Impurities such as CO, CO₂, H₂, O₂ and N₂ need to be determined at low levels in LPGs. Complete separation of these components is done using a two channel single detector (PDHID) system. The GC employs a permanent gas channel for analyzing H₂, O₂, N₂, CH₄ and CO and a specific second channel for the analysis of CO₂. A gasifier is used as a sample introduction device thus giving the capability of handling LPG samples C2 through C4.

Detection limits are at the ppb level (Figure 13), depending on the component of interest.

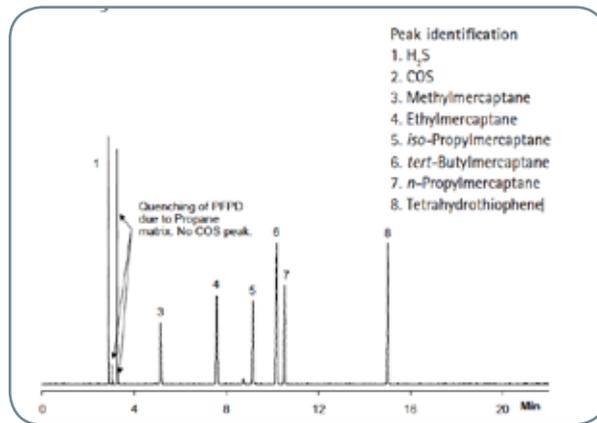


Figure 11: Sulfur components in propane, BR-1 column.

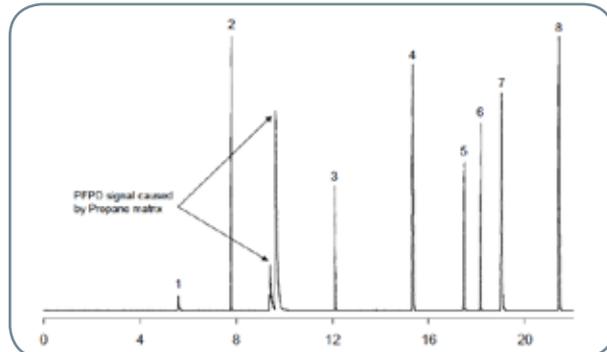


Figure 12: Sulfur components in propane, BR-Q PLOT.

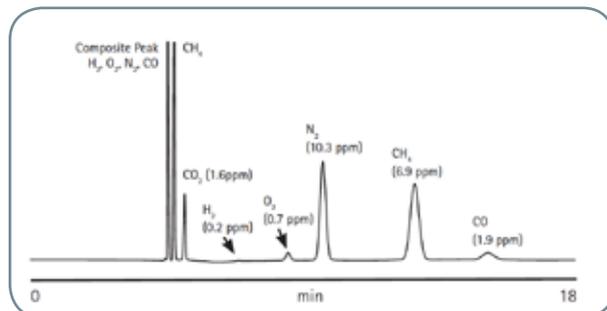


Figure 13: LPG sample.

Total Characterization of Ethylene Impurities

For a total characterization of impurities in ethylene and also propylene six GC channels are required. By coupling two Scion GCs with three channels each, a comprehensive solution is available for analyzing these components. The channels used in this analyzer are analytical tools principally developed for the determination of different gases in various hydrocarbon types of gaseous matrices.

GC-1

- H₂ Channel (TCD)
- O₂/N₂ Channel (TCD)
- CO, CO₂ Channel (Methanizer/FID)

GC-2

- Light Hydrocarbon Channel (FID)
- Oxygenates Channel (FID)
- Sulfur Channel (PFPD)

The results (see figures 14 to 19) demonstrate how this 6 channel system is perfectly suited for the total characterization of ethylene and its impurities.

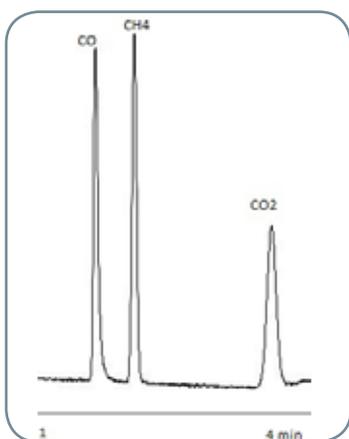


Figure 14: CO, CH₄ and CO₂ on GC-1.

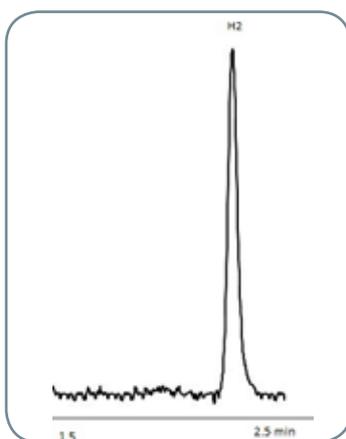


Figure 15: H₂ on GC-1.

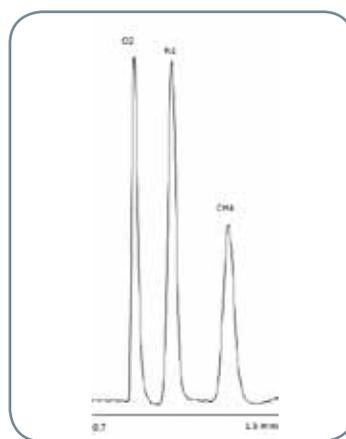


Figure 16: O₂ and N₂ on GC-1.

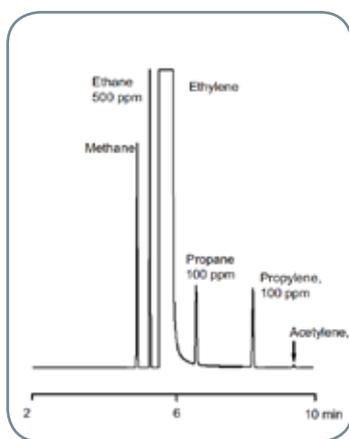


Figure 17: Light hydrocarbons on GC-2.

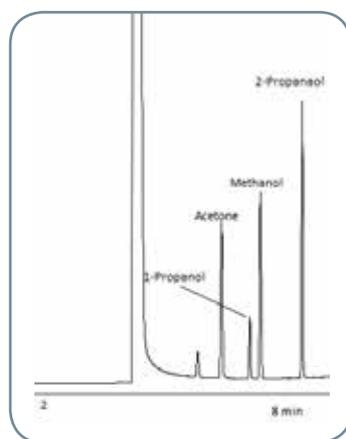


Figure 18: Oxygenates on GC-2.

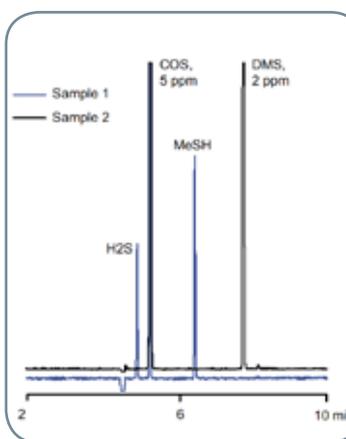


Figure 19: Sulfur components on GC-2.

Scion-Certified Consumables for Your SCION GC Series

Scion GC columns span a broad range of column diameters, stationary phases, and capillary column materials: Fused Silica (FS) and Inert Steel (IS). Ideal for either routine or research type analyses. Scion GC column offerings bridge across many important applications and include a number of offerings such as:

- Standard WCOT (Wall Coated Open Tubular)
- Solid Stationary Phase PLOT (Porous Layer Open Tubular)
- Inert Steel Micro-Packed and Packed



Super Clean™ Gas Filters

Scion Gas Purification Systems have the range to satisfy your needs from individual to combination filters, from Ultra purity combined with Ultra capacity, to all in one solution kits. Innovative features designed into the product yield extensive benefits to the user.

- Ultra-high capacity for long life, less change and improved productivity
- High-purity output ensures 99.9999% Pure Gas
- “Quick connect” fittings for easy, leak-tight filter changes
- Glass internals prevent diffusion; plastic externally for safety
- Easy-to-read indicators for planned maintenance and improved up-time



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Application Note #1820229

GC/MS Linear Calibration with Extended Dynamic Range (EDR) Technology for ASTM D5769

Introduction

ASTM D5769 [1] is a method used commonly in refineries to quantify volatile organic compounds in gasoline. This can be a problem for GC/MS systems, because the analysis involves a wide range of volatile components at very high concentrations. The detector can easily become saturated and non-linear curves result. Modifying MS tuning conditions, ion source hardware, or electron multiplier settings during the run have been proposed ways of dealing with this problem.

However, such modifications involve more complexity in method setup for routine testing labs.

The SCION™ GC-MS uses a patented detection technology known as Extended Dynamic Range or EDR. EDR senses the level of the ion signal every scan and then adjusts the detector voltage and the perceived gain in order to keep the signal output at the optimum level for detection. This results in meeting the method linear calibration criteria without operator intervention or changes to the method procedure.

Author 

Ed George,

Keywords	Instrumentation and Software
ASTM D5769	SCION Select SQ
Volatile Organic Chemicals (VOCs)	SCION 436-GC
Petrochemical	CP-8400 Autosampler
Gasoline	MSWS 8 Software
Extended Dynamic Range	
Refineries	

Experimental

The recommended instrument and operating parameters are listed in Table 1.

Table 1: Gas Chromatograph and Mass Spectrometer Conditions.

Gas Chromatograph	SCION 436		
Inlet	SSL (Split/Splitless)		
Mode	Split Injection, 0.1 μ L		
Injection Temp	250 $^{\circ}$ C		
Pressure Pulse	None		
Split Ratio	500:1		
Inlet Liner	4 mm ID Split liner w/Frit, p/n RT233305		
Oven			
Oven Ramp	$^{\circ}$C/min	Temp $^{\circ}$C	Hold (min)
Initial		60	0.5
Ramp 1	3.0	120	1.0
Ramp 2	10	250	1.0
Total Run Time:		35.5 min	
Column			
Scion BR-1ms, 60 m x 0.25 mm x 1.0 μ m, p/n BR86644			
Constant Flow 1.0 mL/min			
Mass Spectrometer	SCION Select SQ		
Solvent Delay	4 minutes		
Scan Range	45-300 da		
Dwell Time	250 ms		
Ion Source Temp	250 $^{\circ}$ C		
Transfer line Temp	280 $^{\circ}$ C		
Detector	EDR		

Calibration standards were obtained from AccuStandard Corporation, New Haven, CT, Gasoline Refinery Aromatics Standard, p/n D-5769-CAL/IS SET. The calibration levels were prepared based on the weight fraction found in refined gasoline as referenced in ASTM D5769.

A mass fragmentation pattern standard was also obtained to check for expected ion ratios for 1,3,5-Trimethylbenzene.

The advantage of EDR technology is that it makes automatic adjustments of the electron multiplier gain 'on the fly', in real time. If the signal is very large, the detector voltage goes down to avoid saturation. If the signal is small, the voltage goes up, so it efficiently detects low signal levels. Other MS detectors are set at a single voltage and must be manually adjusted to avoid saturation. If set too low, smaller signals are missed, and if set too high, the detector will saturate. In this case, methods must be set up with multiple segments making method development more complex.

Figure 1 is a representation of how EDR adjusts the detector gain as a peak elutes from the GC and ions arrive at the detector. Figure 2 shows the linear range of the detector for OFN, from a concentration on-column of 0.1 pg to 100 ng. Without EDR turned on, peaks will have the characteristic 'flat top' indicating saturation and result in poor quantitation. The mass spectrum also becomes distorted, resulting in poor qualitative identification in library searches.

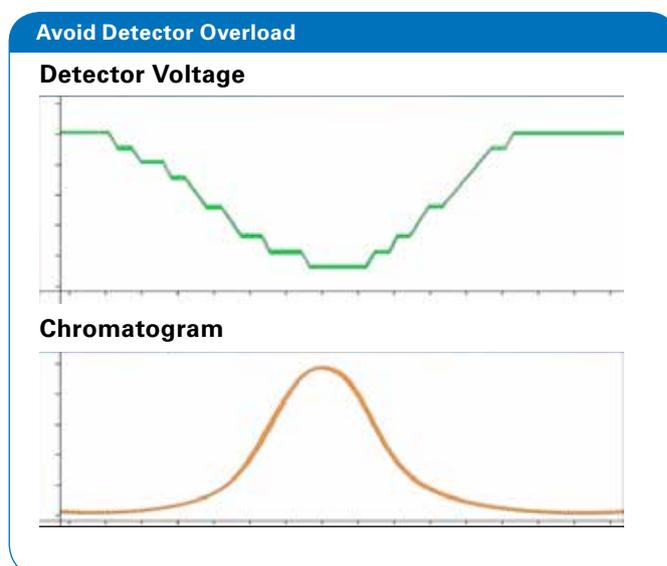


Figure 1: EDR automatically adjusts electron multiplier voltage in real time as peak elutes from the gas chromatograph, preventing detector saturation.

EDR: Extended Dynamic Range

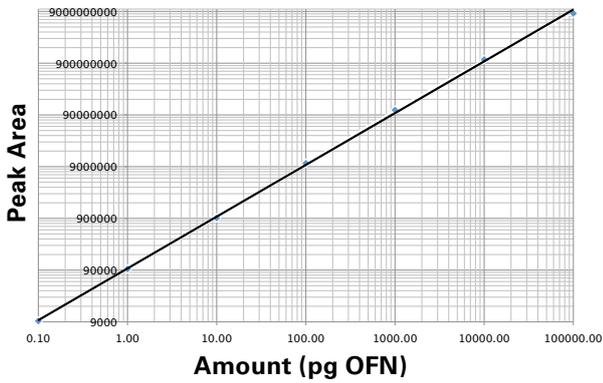


Figure 2: The linear range of the detector with EDR is demonstrated by injection of OFN from 0.1 pg to 100 ng on-column.

Results

The SCION GC-MS system was set up for ASTM D5769 and shown to easily meet the requirements for the mass fragmentation pattern standard and resolution. Built-in reporting tools help routine labs check this quickly as can be seen in Figures 3a and 3b.

Resolution Reports with Microsoft Access Quick-EP

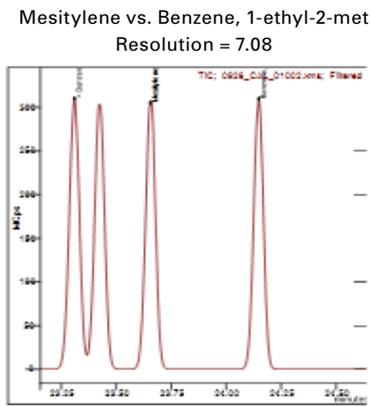
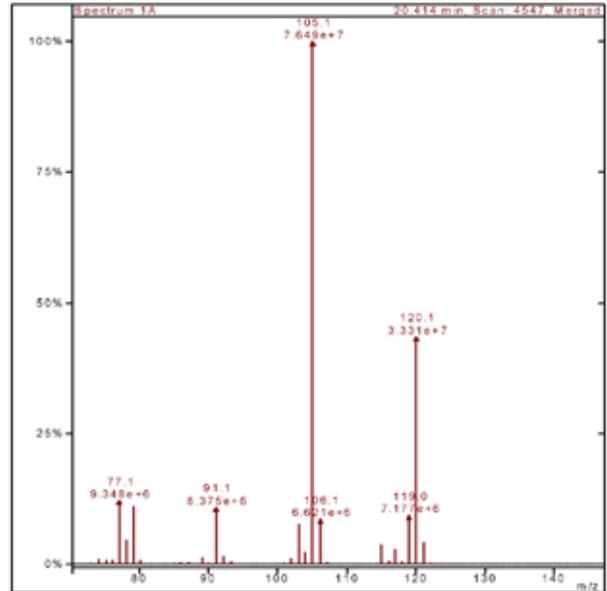


Figure 3a: Example report for 1,3,5-Trimethylbenzene (Mesitylene) and 1-Methyl-2-ethylbenzene.

Mass Fragmentation Pattern



Ion (m/z)	Acceptance Criteria	Observed Value
120	30-60	43
105	100	100
91	7-15	10.5

Figure 3b: Ion abundance requirement is easily met with the SCION Select SQ.

The calibration standards contain 23 volatile organics commonly found in gasoline. The ASTM method requires a 5 point calibration and if linear fits are used, the curves must have r^2 values greater than 0.99 and maximum of 5% RSD of response factors. All of the compounds were found to be linear over their respective ranges, with average % RSDs for all curves at 2.6% with average r^2 value of 0.9993.

Toluene has the highest concentration range and can easily saturate most MS detectors, even with split ratios greater than 1000:1. The SCION Select SQ maintains good linearity and mass spectral quality at the highest concentration standard as demonstrated in Figures 4a and 4b.

Toluene Calibration Curve

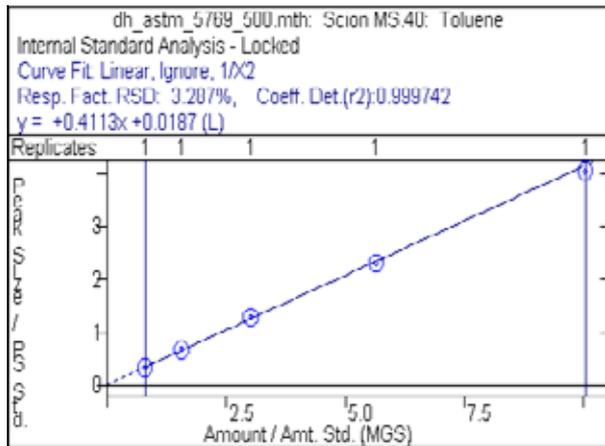


Figure 4a: Toluene not saturated at Level 01 (High Level) with linear curve on SCION SQ.

Toluene Mass Spectrum

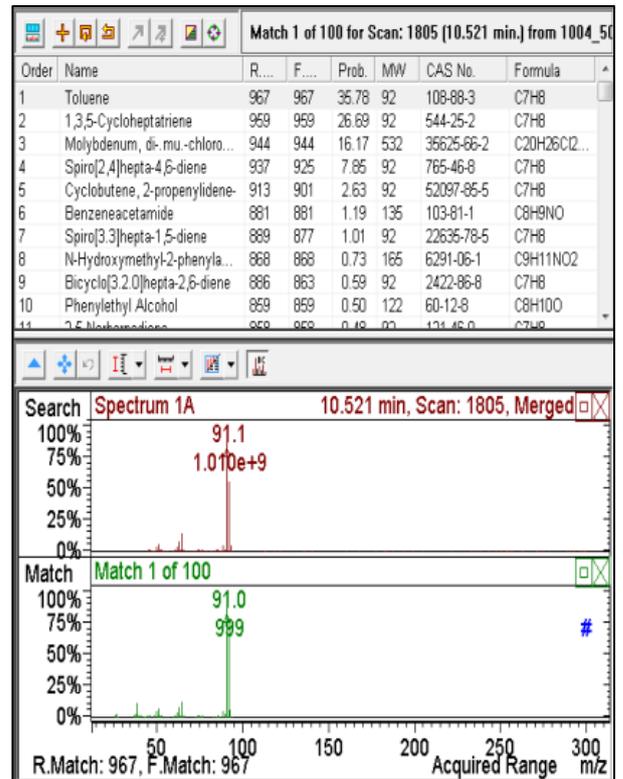


Figure 4b: Mass spectrum at apex of highest level standard shows no saturation and excellent spectral match vs. NIST library.

References

- [1] ASTM D 5769-04, Standard Test Method for Determination of Benzene, Toluene, and Total Aromatics in Finished Gasoline by Gas Chromatography/Mass Spectrometry

Conclusion

SCION GC-MS systems with EDR technology will extend the linear range of the mass spectrometer without requiring special modifications to the ion source hardware, MS tuning, or need for manual detector gain programming. EDR detects the ion signal and adjusts the gain in real time in order to avoid saturation. This makes method development easier for methods such as ASTM D5769 that require a wide dynamic range for volatile organics in gasoline.

For research use only. Not for use in diagnostic procedures.

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Application Note #1820230

Performance of Method 8270 Using Hydrogen Carrier Gas on SCION™ GC-MS

Introduction

USEPA Method 8270 [1] for semivolatiles is used by laboratories to measure a mixture of acids, bases, and neutrals in a wide variety of matrices. Sample extracts can range from clean water to sludge coming from Superfund clean-up sites. The complexity of these extracts demand a robust instrument that is easy to operate and maintain. Adding to method complexity is the uncertainty in both cost and supply of helium, forcing some laboratories to consider hydrogen as carrier gas. Hydrogen is not an inert gas; it is reactive and can be an explosion hazard if allowed to build up in either the GC oven or manifold of a mass spectrometer. The SCION Helium-free Package will ensure safe routine operation, with little or no performance change for EPA Method 8270. Scion's unique axial ion source design provides excellent robust operation and minimizes unwanted protonation and spectral distortions. In addition, the Gas Chromatograph with Scion Split/Splitless (SSL) injector and inert pathway prevent compound degradation and reactions with hydrogen.

Author 

Ed George,

Keywords	Instrumentation and Software
Environmental Testing	SCION Select SQ
GC/MS	SCION 436-GC
8270	PEAK Precision 500 Hydrogen Generator
SW-846	CP-8400 Autosampler
Semi-volatiles	MSWS 8 Software
Hydrogen Carrier Gas	

Table 1: Gas Chromatograph and Mass Spectrometer Conditions.

Gas Chromatograph	SCION 436		
Inlet	SSL (Split/Splitless)		
Mode	Pulsed Split Injection, 0.5 μ L		
Injection Temp	290 $^{\circ}$ C		
Pressure Pulse	40 psi for 0.3 min		
Split Flow	70 mL/min		
Gas Type	Hydrogen		
Gas Supply	PEAK Precision 500 TRACE Hydrogen Generator		
Inlet Liner	SGE 4 mm ID Liner w/ goose-neck, p/n 092017		
Oven			
Oven Ramp	$^{\circ}$C/min	Temp $^{\circ}$C	Hold (min)
Initial		45	3.0
Ramp 1	25	100	1.0
Ramp 2	10	310	1.0
Total Run Time:	28 min		
Column			
SGE BP-5 MS, 20 m x 0.18mm x 0.18 μ m, p/n 054301			
Constant Flow 1.0 mL/min Hydrogen Carrier			
Mass Spectrometer	SCION Select SQ with Helium-free Package		
Solvent Delay	2 minutes		
Scan Range	45-500 da		
Dwell Time	250 ms		
Ion Source Temp	330 $^{\circ}$ C		
Transfer line Temp	300 $^{\circ}$ C		

Experimental

The recommended instrument and operating parameters are listed in Table 1. When using hydrogen carrier for the first time, more hydrocarbon background is observed but will clean up over time. This initial background can be reduced significantly by increasing the ion source temperature to 350 $^{\circ}$ C, with hydrogen column flow at 4 mL/min and filament turned ON for 4 hours.

Calibration standards were obtained from Restek Corporation, Bellefonte PA, MegaMix p/n 31850, containing 76 target compounds. The calibration for most analytes ranged from 1 to 200 ppm. Internal and surrogate standards were added at a concentration of 40 ppm. Solvent: Dichloromethane.

A pulsed split injection was used to minimize contact and residence time of compounds inside the inlet. This is critical in hydrogen carrier gas, due to its low viscosity and tendency to react with dichloromethane and form HCl. Figure 1 illustrates the pulsed split injection technique with the Scion SSL.

The single goose-neck 4 mm open inlet liner is commonly used with Method 8270. It does not contain glass wool. Glass wool could contribute to compound degradation, especially when using reactive hydrogen as carrier gas.

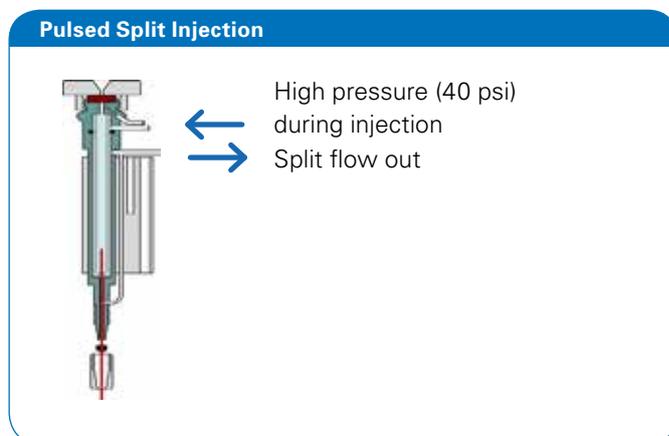


Figure 1: During the pulsed split injection, the inlet is set to 40 psi with split flow at 70:1. Under this condition, the column flow is near 5 mL/min, allowing rapid transport of analytes to column.

Results

In order for hydrogen carrier gas to be considered for use, Method 8270 specifications in terms of tuning, resolution, calibration, peak shape (Gaussian Factor), and system performance checks (SPCCs) must be met. In addition, the solvent specified in the method is dichloromethane (DCM), therefore essential to minimize degradation in the inlet. The GC/MS system must also produce mass spectra that match NIST library and demonstrate robust operation in heavy matrices.

The SCION with Helium-free Package can be auto-tuned normally and used with the tune-to-target feature for passing DFTPP tune. Figure 2 illustrates a DFTPP spectrum and report generated with the EnviroPro™ software.

The viscosity of hydrogen changes at a much slower rate vs. temperature than helium, and is an advantage for maintaining good solubility in the stationary phase during the GC oven ramp. Also, at a flow of 1.0 mL/min using the 20 meter 0.18 mm ID column, an optimal linear velocity for peak separation is maintained. This assures that the peak resolution criteria in the method is met, as illustrated in Figure 3 for Benzo(b) and Benzo(k)fluoranthene. The peak Gaussian Factor or peak Tailing Factor for Pentachlorophenol demonstrates the inert behavior of the system.

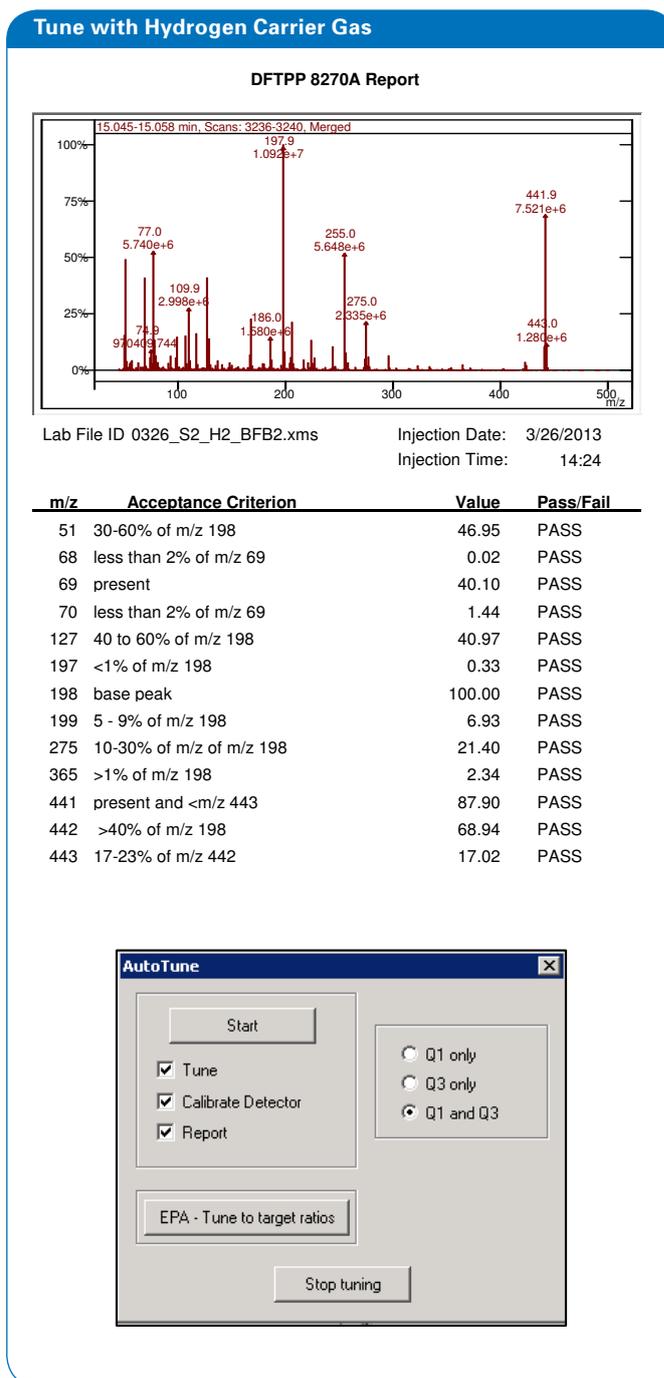


Figure 2: Auto-tune and tune-to-target work without modification using hydrogen carrier. DFTPP report easily generated from EnviroPro™ Software Package.

EnviroPro™ Software Reports

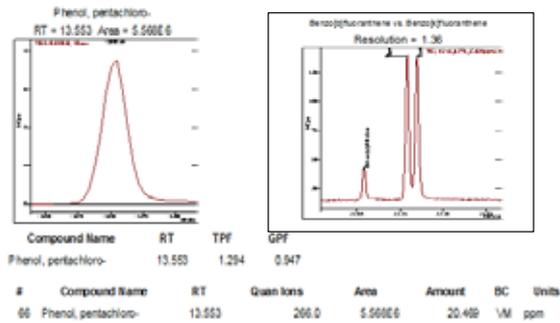


Figure 3: SPCC Peak Gaussian report for Pentachlorophenol (left) and Peak Resolution Test for Benzo(b) and (k) Fluoranthene generated using EnviroPro™ Software.

The SPCCs and initial calibration criteria are also met with hydrogen carrier. For the SPCCs the relative response factors (RRFs) are measured and compared to the criteria given in Table 2.

Table 2: Relative Response Factors with Hydrogen Carrier Gas.

Compound	Method 8270 Min. RRF	RRF on SCION Select SQ
N-nitroso-di-n-propylamine	0.05	0.715
Hexachlorocyclopentadiene	0.05	0.178
2,4-Dinitrophenol	0.05	0.070
4-Nitrophenol	0.05	0.134

An example calibration curve of Hexachlorocyclopentadiene is shown in Figure 4, with % RSD and corr. coefficient (r^2) for this compound of 3.9% and 0.9998, respectively.

Calibration with Hydrogen Carrier Gas

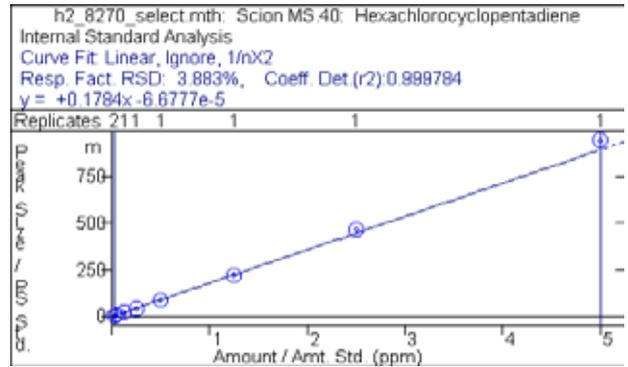


Figure 4: Calibration curve for Hexachlorocyclopentadiene with % RSD and corr. coefficient (r^2) of 3.9% and 0.9998, respectively.

The overall average % RSD of all 76 compounds was 8.5%. Calibration for the critical quality control (QC) check and SPCC compounds are listed in Table 3, along with some %RSDs shown for very active compounds in Figure 5.

Table 3: Quality control check compounds for Method 8270 pass using hydrogen as carrier gas.

QC Check Compounds	Corr. Coeff.	Avg. RRF	% RSD	CCC	SPCC
Phenol	0.9980	0.722	11.9	PASS	
Benzene, 1,4-dichloro-	0.9985	0.980	7.6	PASS	
1-Propanamine, N-nitroso-N-propyl-	0.9991	0.719	8.5		PASS
Phenol, 2-nitro-	0.9968	0.116	13.3	PASS	
Phenol, 2,4-dichloro-	0.9981	0.114	8.4	PASS	
1,3-Butadiene, 1,1,2,3,4,4-hexachloro	0.9991	0.315	7.0	PASS	
Phenol, 4-chloro-3-methyl-	0.9958	0.147	9.8	PASS	
Hexachlorocyclopentadiene	0.9998	0.178	3.9		PASS
Phenol, 2,4,6-trichloro-	0.9997	0.142	8.0	PASS	
Acenaphthene	0.9997	1.16	4.9	PASS	
Phenol, 2,4-dinitro-	0.9993	0.070	14.5		PASS
Phenol, 4-nitro-	0.9940	0.134	6.8		PASS
Phenol, pentachloro-	0.9994	0.082	14.2	PASS	
Fluoranthene	0.9998	1.42	6.3	PASS	
Di-n-octyl phthalate	0.9998	0.199	4.6	PASS	
Benzo[a]pyrene	0.9993	1.57	14.8	PASS	

The SPCC compounds must have average RRF > 0.05 for initial calibration **PASS**

The CCC compounds must have % RSD of RRFs for the initial calibration < 30% **PASS**

The overall average of all compound % RSDs must be < 15% **PASS**

Calibration Response Factors for Active Compounds

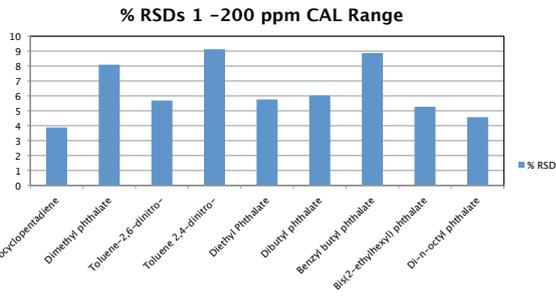


Figure 5: Active compounds show calibration response factors less than 10% indicating minimal reactivity with liner and ion source with hydrogen carrier and dichloromethane solvent.

Spectral quality may be problematic using hydrogen because unwanted protonation or other reactions in the ion source are possible. The Scion axial ion source design with Helium-free Package minimizes these reactions, as evidenced by good quality library matches to NIST. During this study, all 76 compounds were detected using automated AMDIS deconvolution/library search against NIST 11. Figure 6 illustrates example library match hits at 900+ for 1,2,4-Trichlorobenzene and Dibutyl phthalate.

Excellent Library Matches with Hydrogen Carrier Gas

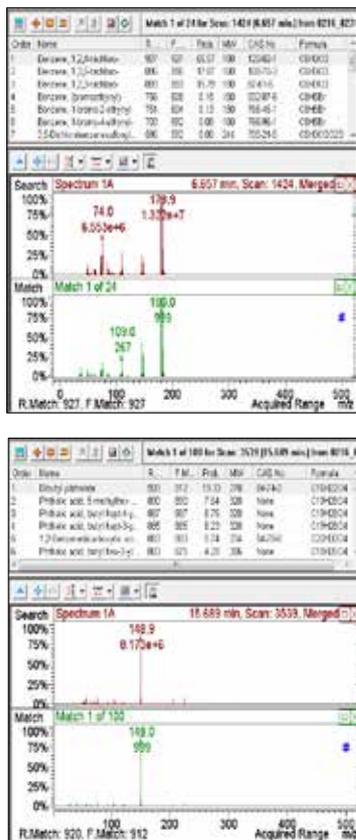


Figure 6: NIST library matches for 1,2,4-Trichlorobenzene and Dibutyl phthalate.

Method 8270 performance was tested using repeated injections of a contaminated sludge extract. A total of fifty injections were made, with an injection of the continuing calibration check (CCC) after every 10 extracts. The calculated concentration of the surrogates (target 40 ppm) and the % differences in response of the compounds were compared to the initial injection and then plotted. Figure 7 shows a TIC of the sludge extract. Figures 8 illustrates an example surrogate (2,4,6-Tribromophenol) and Figure 9 shows percent differences observed for the CCCs. As can be seen from the plots, excellent robust operation of the SCION Select SQ is maintained with hydrogen carrier.

Sludge Extract Injections

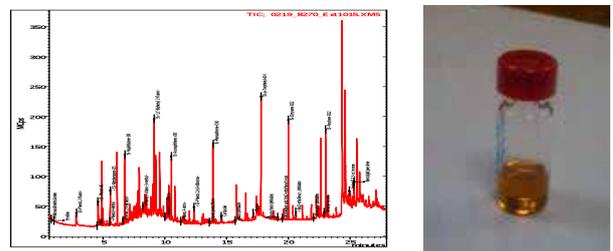


Figure 7: A Method 8270 sludge extract (right) was injected 50 times. A CCC was injected between every 10 extract injections for monitoring the % difference of surrogate and CCC compounds.

Surrogate Standard Recovery

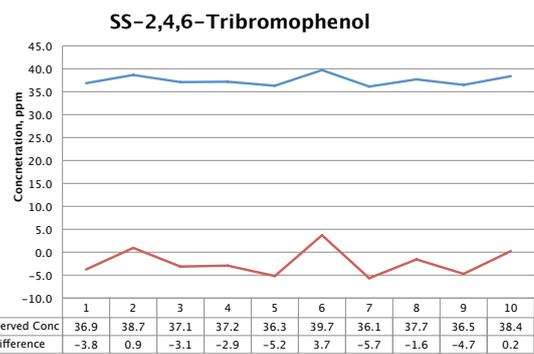


Figure 8: The target concentration = 40 ppm, with % difference measured relative to initial CCC before extract injections.

Continuing Calibration Check Stability

Average % Difference for CCCs

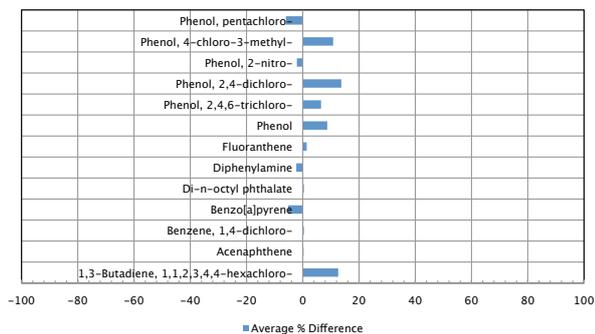


Figure 9: Target concentration = 20 ppm. The plot shows the average % difference for the base neutral and acid compounds in the CCC mixture over 50 matrix injections.

Conclusion

Method 8270 is a challenging method covering a wide variety of compound classes and matrix types. As the cost and scarcity of helium rises, laboratories have begun to convert methods to hydrogen carrier gas, due to the ability to safely produce it on demand with generators. Since hydrogen is a reactive gas and can develop explosive build-up in certain situations, a GC/MS solution must be safe and also produce data that pass both the qualitative and quantitative aspects of the method.

The SCION Select SQ has demonstrated excellent performance for Method 8270. The axial ion source, as well as the pulsed-split injection technique with the Scion SSL, was shown to produce excellent library-searchable mass spectra, passing quality control criteria, and robust operation in heavy matrices.

For research use only. Not for use in diagnostic procedures.

References

[1] USEPA Method 8270D, Revision 4, February 2007

Acknowledgements

PEAK Scientific for use of the Precision 500 TRACE Hydrogen Generator

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compassCDS

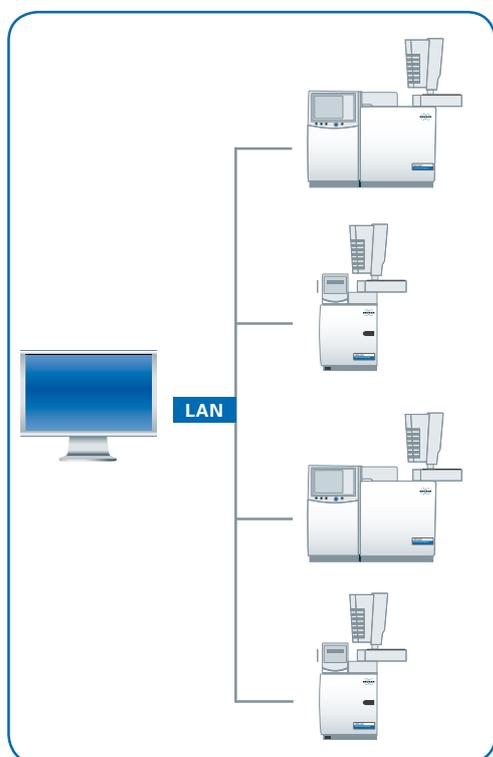
- GC Chromatography Data System

One Platform. One Solution. Designed to Meet Your Needs.

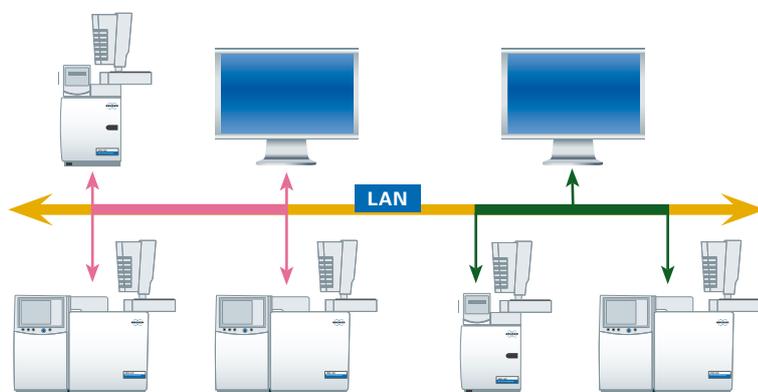
CompassCDS is a scalable, readily configured, easy to use software solution for all of your gas chromatography instrument control, data collection and analysis needs. CompassCDS includes a wide variety of standard data processing functionality, as well as numerous plug-ins, that enhance your capabilities to generate outstanding results and deliver clear, concise reports.

With its unique capacity for customization, CompassCDS provides a powerful tool for a variety of analytical operations. Available in either a stand alone or server based environment, CompassCDS can easily grow with your requirements.

CompassCDS provides you with the results you need in the format you want.

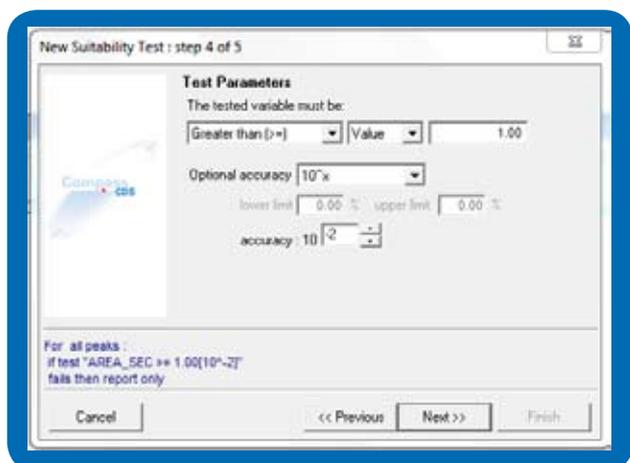


Direct connection of multiple GCs to one PC is the most straight forward and simple configuration especially when a standalone version is required or no network is available.



GCs can be placed anywhere on a server network. Via a PC connected to the same server complete control and data acquisition is possible. A second PC/GC configuration on the same network is also possible. The number of GCs connected to one PC is virtually unlimited.

● Chromatography Data Handling



Easy set up of a new suitability test parameter.

System Suitability Determination

System suitability software determines the quality of a chromatographic analysis, calculating parameters such as peak asymmetry and column efficiency. The software can compare the results generated from injected control samples to their expected concentrations. Limits put on these results can then be used to determine the best action to take to correct potential problems and ensure the integrity of data and results.

Column Tracker Function

Column Tracker, an integrated database, keeps track of the identity and usage for all system columns. Every time an injection is made, Column Tracker records the injection, no matter which instrument or which column is being used. This information is stored in the database, as well as with each data file, providing a completely documented usage and performance history for every GC column.

Multimode Print Manager

Print Manager allows users to conveniently export reports and chromatograms, in multiple formats to one or more network printers. CompassCDS can automatically convert chromatogram results to many different formats such as ASCII, PDF, Excel, or AIA, simplifying the ability to perform further calculations or modeling. Furthermore, it's easy to interface with other systems like LIMS.

Configuration Manager

The configuration manager is a user friendly tool that organizes access levels and data storage locations defined in user profiles. This will keep administrative functions away from the day to day use of the software by laboratory technicians.

Experimental Deviation Compensation

In some cases instrument and system effects can cause experimental deviations. CompassCDS SmartTimeUpdate™ function can be used to automatically correct retention times and timed events on the same run where the chromatographic deviations occurred, without changing instrument parameters. This function is applicable with packed columns, multi-column systems, and manual and electronic gas control, as well as for use with verified methods.



● Customized Plug-Ins For Particular Applications

The Plug-in Tool Kit

This series of software customization tools allows power users to develop customized calculation modules, which fully integrate with CompassCDS. Plug-ins, developed using standard computer languages, can be automatically called from CompassCDS, yet remain independent, eliminating the need for revalidating CompassCDS after installation. A large number of standard plug-ins are available that allow special reporting and other post analysis functions.

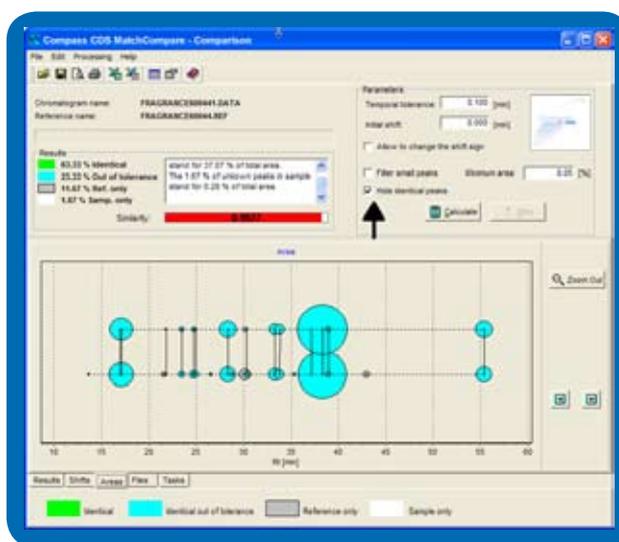
Some examples of application specific utilization of the CompassCDS Plug-ins include:

CompassCDS MatchCompare

CompassCDS MatchCompare software provides rapid visual and quantitative comparison of complex chromatograms. This unique plug-in is already widely used in the flavor and fragrance industry.

Simulated Distillation

The Simulated Distillation package provides automated boiling point distributions of a full range of petroleum products for applications that comply with ASTM, IP, DIN, and ISO standard test methods.



MatchCompare automatically match peaks between two complex mixtures.

Detailed Hydrocarbon Analysis

The Detailed Hydrocarbon Analysis package reports in an automated way the physical properties of gasoline and similar products based on its individual components for applications that comply with Bruker developed methods and ASTM and IP and standard test methods.



Simply Powerful

CompassCDS provides a number of key features that make it the platform of choice for GC system operation, data processing and report generation. These key features include:

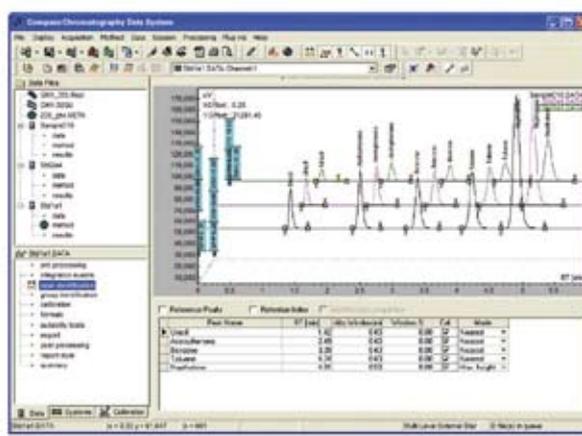
- Single Screen Interface
- Flexible Data Handling and Report Generation
- Comprehensive Security Options
- Efficient Summary Reports
- System Suitability Determination
- Column Tracking Function
- Multimode Print Manager
- Experimental Deviation Compensation
- Customized Software Plug-Ins for Specific Applications

Single Screen Operation

CompassCDS's single screen approach to building methods and sequences, as well as viewing and manipulating data, make it very easy to use and learn the software platform. With CompassCDS, all pre- and post run activities as well as instrument control and status monitoring are done from a single screen.

A single CompassCDS method contains several functions including: instrument control, data acquisition, display, processing, and reporting. Those functions are distributed into smaller, easier-to-use visual segments to avoid distracting users with too much on-screen information. Each method segment deals with a single aspect, such as integration, peak identification, calibration, or reporting.

Data Analysis with CompassCDS



Flexible Data Handling and Report Generation

CompassCDS data handling and reporting is based on system and user defined variables for both peak and global information. Variables such as peak retention time and the identity of the operator are provided by the system and stored as part of the chromatographic data file.

In addition to the variables generated by the system, users can create their own, representing more advanced calculations or specific user inputs and outputs. Customized variables make data entry and results interpretation easier. They can be created in any language and allow data sets to be optimized and reported in formats specific to your laboratory.

#	Name	Time [Min]	Quantity [g/l]	Height [µV]	Area [µV.Sec]	Area % [%]	Area Validation
1	n-Nonane	4.62	100.00	91.1	123.6	3.770	Passed
2	2-Octanone	5.66	12.00	85.4	82.6	2.520	Passed
3	1-Octanol	6.97	12.00	222.2	410.6	12.528	Passed
4	N-Decane	7.50	20.00	221.0	140.1	4.275	Passed
5	2,6-Dimethylpiperidine	8.46	12.00	499.5	1551.2	47.322	Passed
6	n-Dodecane	8.99	12.00	1191.8	414.9	12.656	Passed
7	2,4-Dimethylpiperidine	9.31	12.00	89.6	461.8	14.088	Passed
8	n-Hexabenzene	10.01	200.00	44.6	93.1	2.841	Passed
Total			392.00	0.0	3277.9	100.000	

Comprehensive Security Options

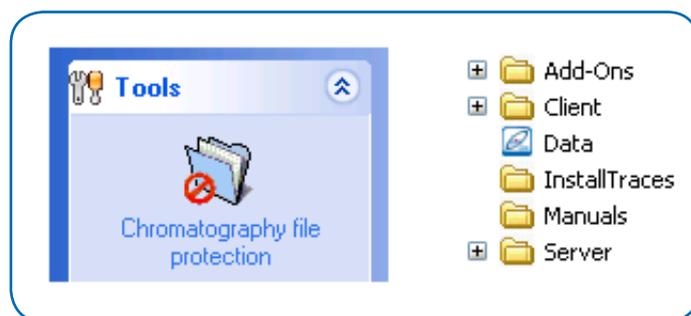


Data and results are critical to your laboratory's operation. CompassCDS provides the security to ensure the integrity of your results, regardless of industry or laboratory size. CompassCDS has a full set of access control and audit trail features to identify and limit who may operate chromatographs and manipulate data.

CompassCDS tracks all changes to methods and results through audit trails embedded in these files and can even protect your data from Windows® Explorer® to prevent files from being accidentally removed or deleted.

Summary Reports

Summary reports use results from any number of chromatograms, taken in a single sequence or over the past months, and calculate averages and standard deviations. Summary reports can also plot trends in data, detecting and flagging changes in chromatographic systems before these changes can compromise operation.



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INSTRUMENTS



SCION SQ Series GC-MS

- The Gas Chromatographers' Detector

SCION SQ™ GC-MS

The SCION SQ series GCMS systems combine innovative engineering with detailed customer requirements to produce a truly innovative system. The SCION SQ series. By designing the GC-MS systems to exceed the most critical performance and reliability needs of GC users, Scion Instruments has delivered systems that are especially for, and all about, the ultimate success of the GC users. The SCION SQ Select and Premium models are designed to meet many important user specific requirements – reliable performance, ease-of-use and simple maintenance – all in a small footprint that saves valuable bench space.



SCION SQ GC-MS Benefits

Easy to Use and Maintain

- Simple tuning due to "lens-free" ion-path design
- No multiplier calibration required

Robust

- An inert ion source that requires less frequent cleaning

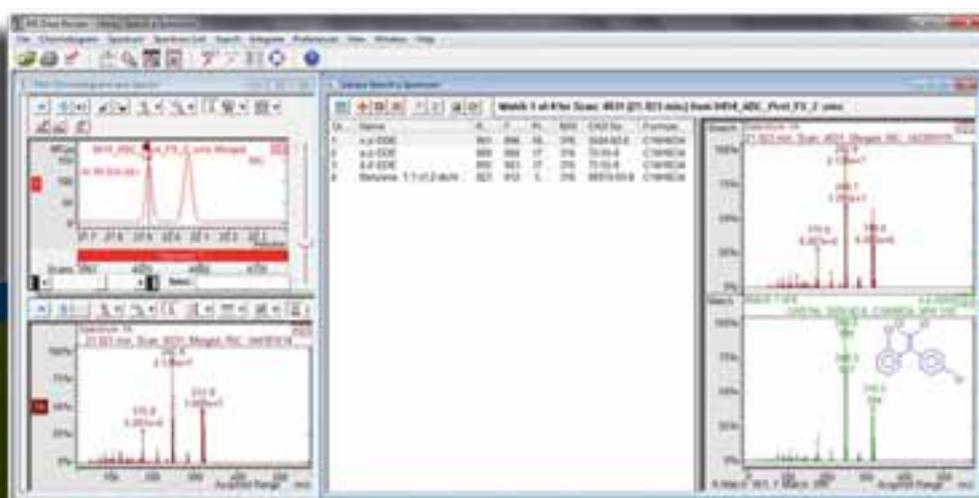
Sensitive

- Optional active-focusing qD uses He atoms to focus ions

• Enhanced Library Searching through Cleaner EI Spectra

The SCIION 50 models are designed to analyze thousands of samples from complex matrices. With an upper mass limit of m/z 1200, they are exceptionally capable of handling almost any GC application. The innovative lens-free design, combined with the robustness of the axial ion source, delivers unmatched stability and ultra-high sensitivity on a routine basis.

- One-click search of multiple spectral databases
- User-created spectral libraries and full support of spectral libraries such as NIST, Wiley, and Maurer/Pfleger/Weber (MPW)
- Adjustable spectral search parameters to streamline library searches
- Automated workflow to build a SIM method from a full scan data file
- Flexible and easy to use



Flexible and powerful library search showing matching of sample peak to o,p'-DDE in the NIST library



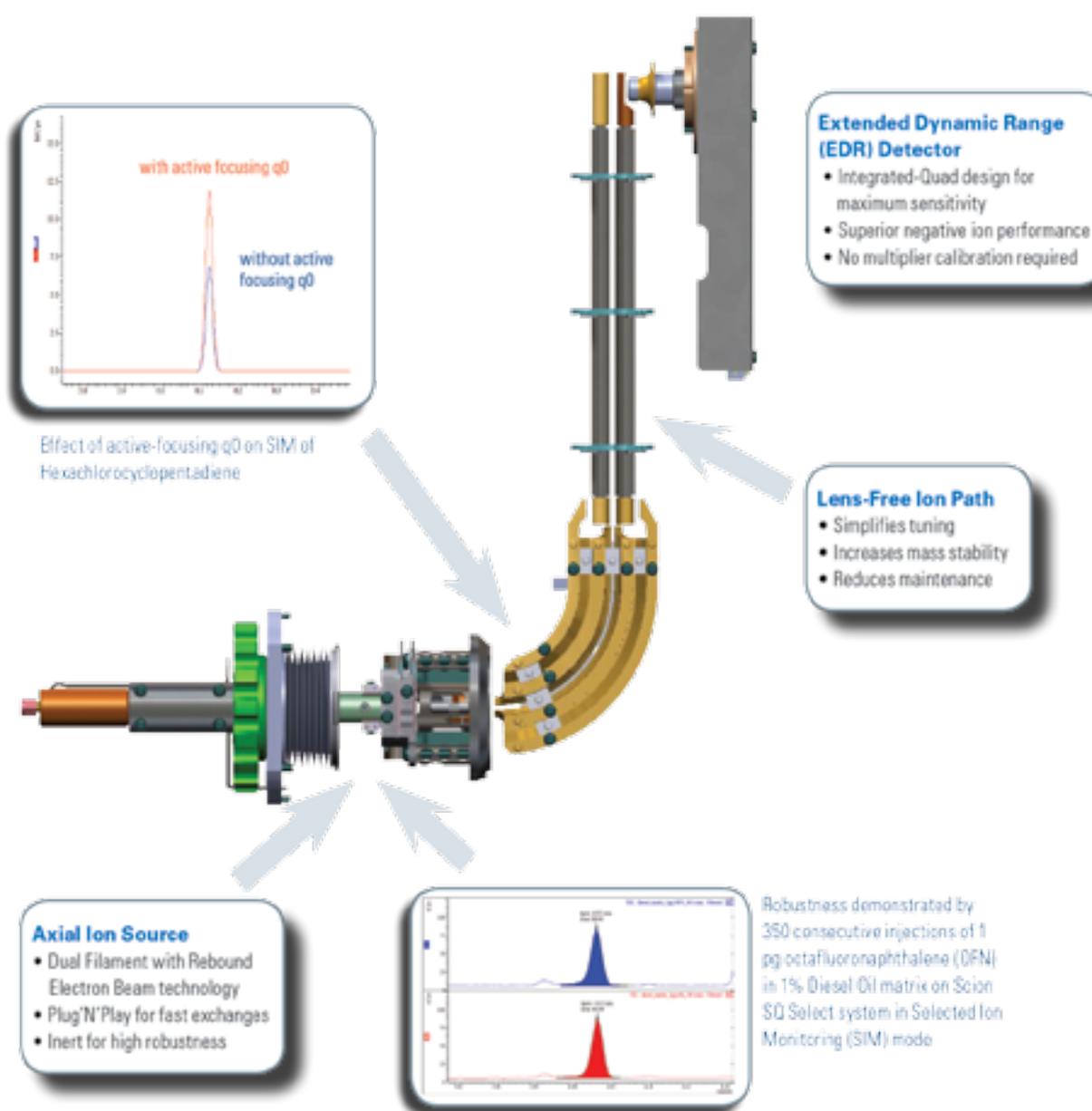
Why Choose the SCION SQ?

The SCION SQ series delivers exceptional performance for a single quadrupole mass spectrometer: a robust axial ion source, ultra-high sensitivity, cleaner spectra, and virtually-zero neutral noise. The series includes the SQ Select, Prime, and Premium GC-MS models.

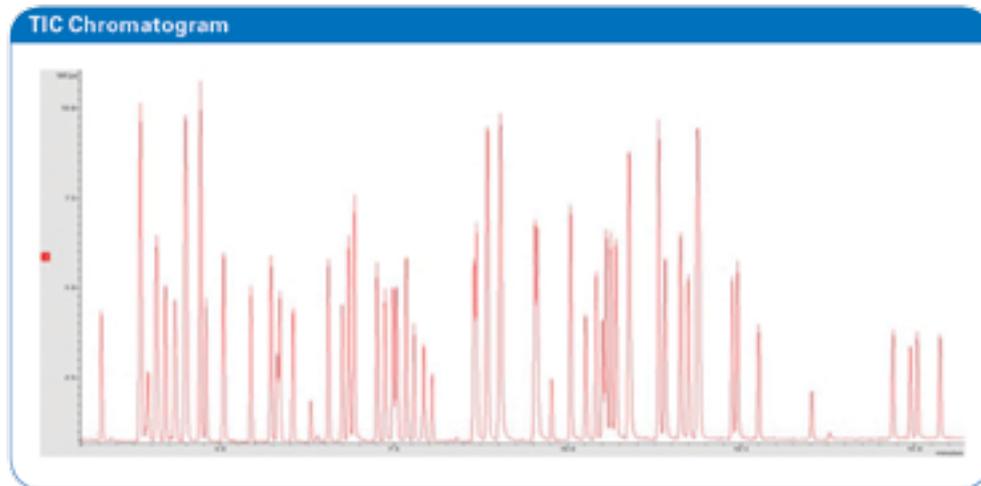
For routine EI-only applications, the **SCION SQ Select** delivers the best value. It comes with an industry leading 400 L/s high-capacity turbo pump that enables fast pump-down time for quick maintenance, and the use of high carrier gas flow for fast GC separations.

The EI-only **SCION SQ Prime** model comes with the split-flow, ultra-high capacity turbo pump (300-400 L/s) for added robustness and ease of use. It comes with the innovative active-focusing q0 optic that delivers enhanced sensitivity.

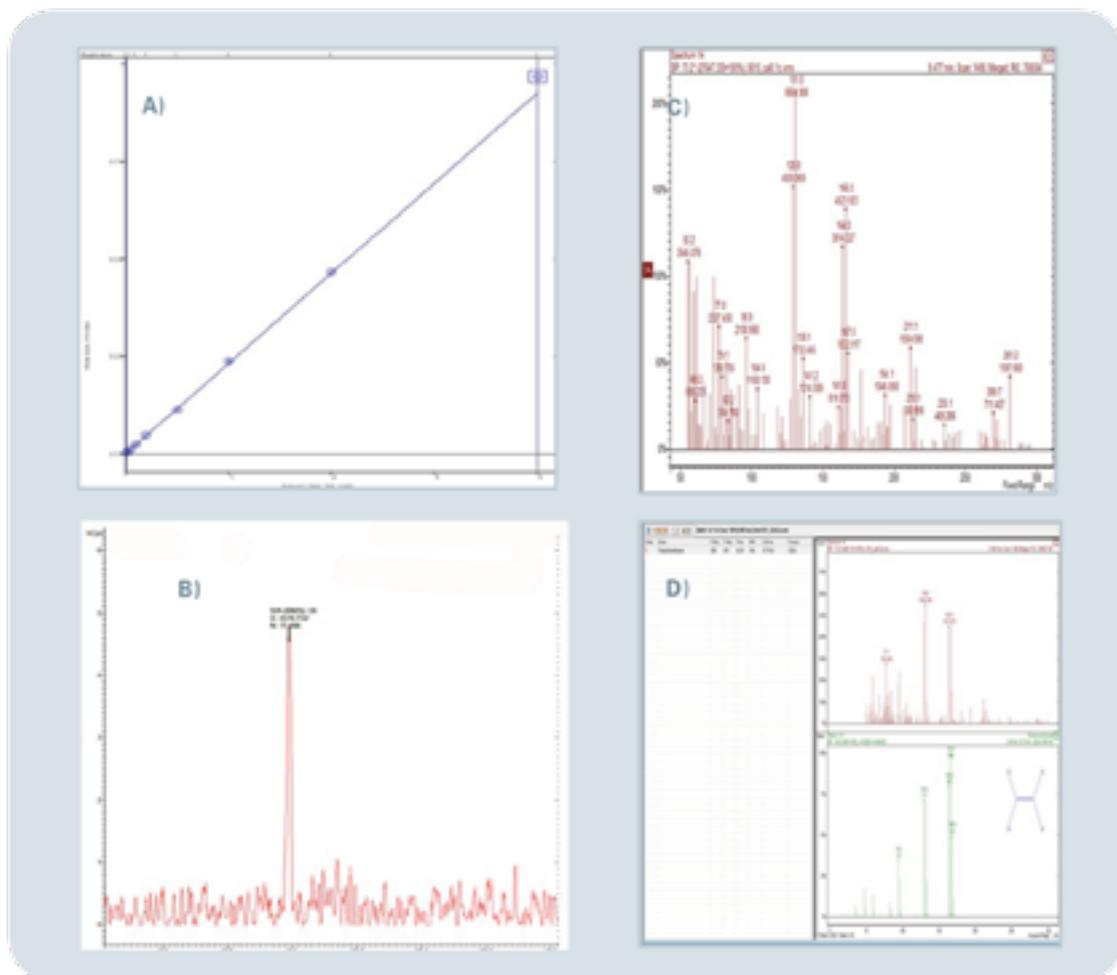
The **SCION SQ Premium** model comes CI-ready and therefore is the most versatile GC-MS SQ platform. The active-focusing q0 optic is heated for the demanding analysis that requires the ultimate robustness.



- **Linearity, Sensitivity, and Spectral Matching Using the SCION SQ for US EPA Methods**



TIC chromatogram of a 5 mL water sample containing 84 VOCs at 10 µg/l (ppb) by US EPA Method 524.3



Gas Chromatographs

An Infusion of Innovation with a Legacy of Reliability

The GC is a key part to the reliability, robustness, and sensitivity of any GC-MS analysis. Scion instrument's philosophy of innovation is highlighted by the introduction of two new GCs built to support the ultrasensitive SCION SQ. The compact SCION 436-GC and the versatile SCION 456-GC can accommodate two columns in the oven and are available with new backflush technology and the innovative ChromatoProbe™. The new GCs are also equipped with the multi-language touchpad display supporting 13 languages and enabling MS control.

SCION 436-GC

Compact design for those focused on routine applications requiring maximum throughput utilizing Split/Splitless (SSL) or Programmable Temperature Vaporization (PTV) injectors

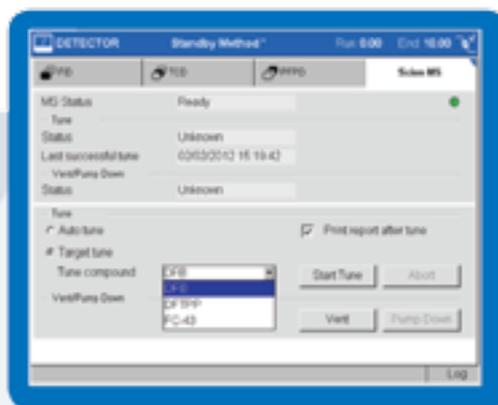
- Select up to 2 capillary inlets: SSL or PTV
- Support one GC detector and the mass spectrometer
- High precision electronic pressure control
- All temperature zones up to 450 °C
- Automated with Model 8400/8410 or CTC liquid autosampler

SCION 456-GC

Versatile design with additional injector and detector options for laboratories seeking multipurpose analysis utilizing both GC and GC-MS

- Support up to 3 injectors-SSL, PTV, cold On-column, and Purge-Packed
- Add up to 3 GC detectors-FID, ECD, TCD, PFPD, NPD (TSD)
- High precision electronic pressure control
- All temperature zones up to 450 °C
- Automated with Model 8400/8410 or CTC liquid autosampler

Scion Instruments has now set the industry standard for ease-of-use: Basic operation of SCION GC-MS systems can be directly controlled from the multi-language touchpad on the gas chromatograph. Automatic tuning, along with tune-to-target for meeting specific USEPA methods can be done with a touch of a button. And the MS can be vented and pumped down from the interface for the ultimate in easy maintenance.



SCION 436-GC



SCION 456-GC

● Additions to Enhance System Capability and Performance

SHS-40 Automated Headspace Sampler

- Perfect for analyzing VOCs in solid or liquid samples
- 40/125 sample capacity – Crimp cap or screw cap 10 or 20 mL vials
- 12 position oven for increased throughput
- 200 °C sample heating for extended range
- Injection with 1 mL sample loop, designed for EPC GCs MHE mode via

PTV Inlet with Back flush

The perfect addition for the SCION SQ PTV inlet is the backflush option.

Complex sample matrix can quickly ruin the chromatographic performance of your GC column. However, the PTV with Scion Instrument's "backflush" technique can reliably divert the higher boiling sample matrix away from the column. The benefits of this accessory are many:

- Run more samples per day – decrease analysis times as the heavy components are quickly eliminated
- Save time by eliminating column bakeout

ChromatoProbe™

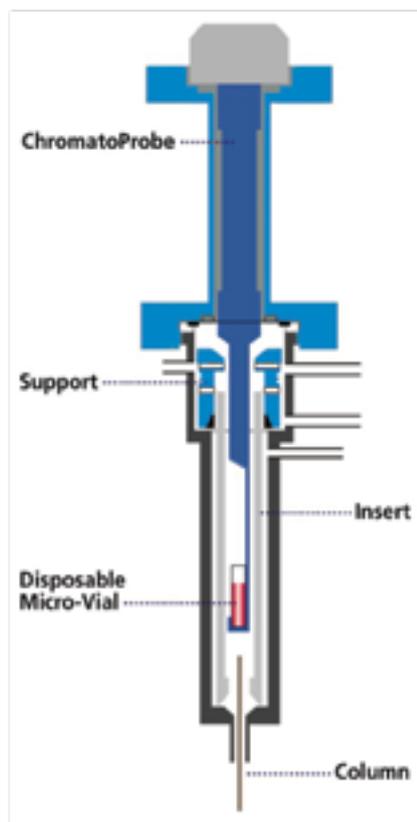
Added versatility for superior analysis of solids, liquids, and slurries

Compounds such as street drugs, industrial solids, synthetic organic products, and plant tissues that normally are not considered amenable to GC-MS analysis can be easily investigated with the ChromatoProbe.

Samples are introduced into the PTV injector via disposable micro-vials. Non-volatile or thermally degraded components from the sample remain in the micro-vial allowing the system to remain clean.

ChromatoProbe benefits:

- Increase uptime
- Minimize system contamination with disposable micro-vials
- Directly desorb samples in the PTV injector without added hardware





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Biodiesel Analyzers

- A family of Comprehensive Solutions

Scion GC Biodiesel Analyzers

Biodiesel is one of the most promising alternative fuel sources available today. Unlike conventional diesel fuel derived from crude oil, biodiesel comes from vegetable oils and animal fats. Compared to petroleum based diesel (petro-diesel), biodiesel is safe, renewable, biodegradable, cleaner burning and is compatible with today's diesel engines. However, like petro-diesel, biodiesel can present a significant analysis challenge.

- Extremely powerful, easy to use analysis solutions. Scion's flexible 456-GC or dual channel single detector 436-GC and compassCDS Chromatography Data Handling Software, combined with a broad range of application optimized columns and consumables provides the analytical power needed to perform all biodiesel analysis methods and ensures that conducting them is simple and straightforward.
- A complete range of GC based biodiesel solutions. Scion offers comprehensive solutions for the analysis of biodiesel by gas chromatography based on 'standard' methods. These include: EN-14105 (free and total glycerol, mono, di and tri-glycerides content), EN-14103 (total FAME and linolenic acid methyl esters), EN14106 (free glycerol), EN-14110 (residual methanol) and ASTM D6584 (free and total glycerin).
- Outstanding multi-channel flexibility of the 456-GC. Depending on your analysis and throughput requirements, two or more standard methods can be accommodated with a single 456-GC system. Both an automated liquid and headspace sampler can be installed on a single 456-GC. It is therefore possible to conduct more than one standard GC analysis method, on a single system without the need for any hardware reconfiguration or lengthy start up/ equilibration times.
- A broad range of application optimized consumables columns specifically designed for biodiesel analysis and long life time. All consumables included in these biodiesel analysis solutions have been carefully selected to ensure both high performance and reliability. Furthermore, a range of high performance capillary columns has been developed specifically for biodiesel applications, including the Scion Biodiesel inert steel column which provides superior separation performance, extremely low phase bleed performance and unequalled column lifetime.
- Single vendor analysis solutions. Scion is proud of its ability to provide a complete solution. All hardware, software, application optimization, documentation, installation and performance verification are provided and supported by Scion, or a trained and authorized Scion dealer.



Figure 1: The 456-GC and compassCDS chromatography workstation team to provide a powerful analysis platform for biodiesel analysis by GC.

● Key Benefits

Introduction

There are a variety of ways to determine biodiesel composition and quality. Both ASTM (American Standard and Testing Methods) and CEN (Comite Europeen de Normalisation) have implemented methods to characterize biodiesel and ensure it conforms to their standard specifications: EN-14214 or ASTM D6751. Of all biodiesel standard methods in use today, those employing GC are the most commonly used. These methods are listed and summarized:

Analysis of Free Glycerine and Total Glycerol; EN-14105, ASTM D6584
 The analysis of glycerine in biodiesel is extremely important because excessive amounts can make long term storage problematic, or cause the formation of unwanted deposits, leading to injector fouling and accelerated engine wear. The analysis of free glycerine and total glycerol requires the use of on-column injection with a high resolution capillary column operated at a very high temperature (>350 °C).

Although there are several factors which can negatively impact the performance of a GC, the most significant associated

Standard Method	Analytes	Injector Type	Recommended GC	Time per Analysis
ASTM D 6584	Free & total Glycerin	On-Column	456-GC or 436-GC	32 mins
EN-14103	Ester & linoleic acid methyl esters	Split / Splitless	456-GC or 436-GC	30 mins
EN-14105	Free and total Glycerin & Mono, Di & Tri-glycerides	On-Column	456-GC or 436-GC	35 mins
EN-14106	Free Glycerol	Split / Splitless	456-GC or 436-GC	10 mins
EN-14110	Methanol	Headspace with Split / Splitless	456-GC or 436-GC	10 mins

Table 1: Overview of Biodiesel methods.

with these particular methods is stress placed on the capillary column as it is subjected to repetitive high temperature thermal cycling of the oven. If conventional fused silica capillary columns are used for either of these methods, a severe reduction in useful column lifetime results. This is due to structural failure of the fused silica column coating itself leading to 'shattering'. To combat this problem, Scion is using a new specifically designed capillary column (Scion Biodiesel). The column is constructed of high tensile strength metal, eliminating the possibility of column failure. Furthermore, the column is extremely easy to install because it has the retention gap pre-coupled and thoroughly leak tested at the factory.

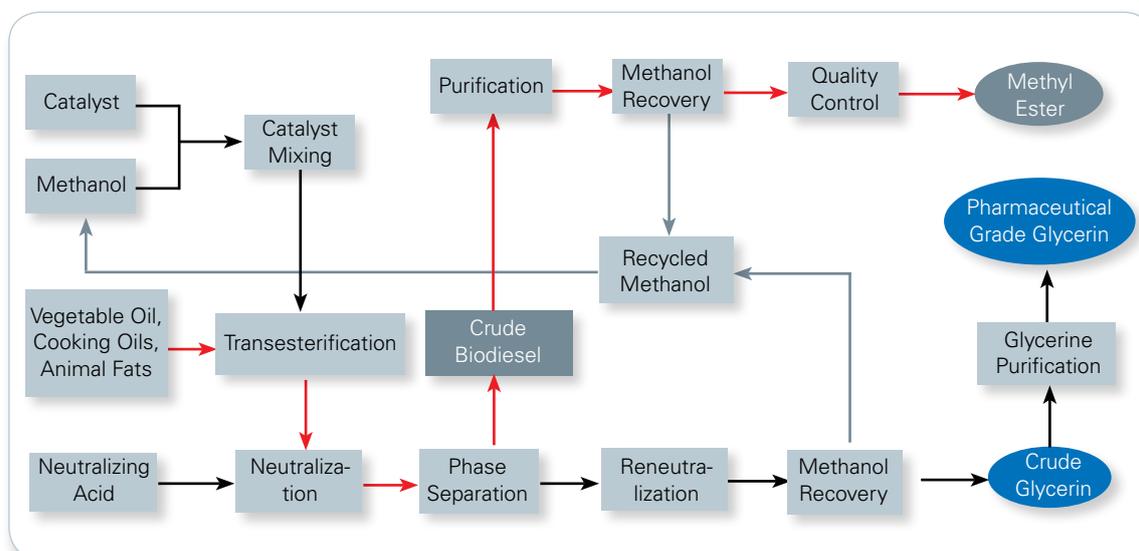


Table 2: Schematic overview of the Biodiesel production process.

● Scion GC Biodiesel Analyzers



Figure 2. The 456-GC configuration including the SHS-40 headspace sampler and CP-8410 liquid sampler for the analysis three Biodiesel methods.

The Scion Analyzers for Biodiesel are standard configured according each method. This maximizes the flexibility for your analytical needs and ensures maximum analysis capacity.

When several methods are required it is possible to combine these analysis into one GC. With the flexibility of the 456-GC, different methods to analyze biodiesel can be combined. This reduces the amount of equipment required and saves bench space. With the 456-GC, up to three methods (methanol, glycerin and Fames) can be configured using all three GC channels.

Automation is possible with the CP-8400 sampler for dual channel liquid injections in combination with the SHS-40 for the headspace analysis.

The glyceride analysis is used on a dedicated instrument only. The method requires an oven temperature that exceeds the columns' maximum allowed temperature of the other methods.

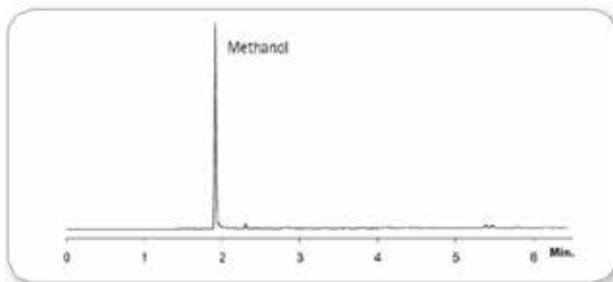


Figure 3. Chromatogram of methanol analysis according to EN-14110: The headspace injection of biodiesel prevents the heavy matrix to be injected onto the column.

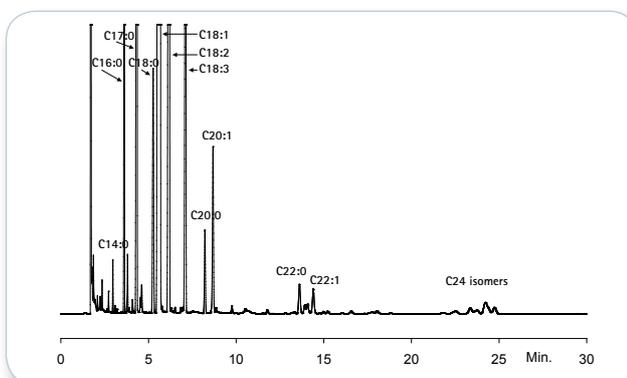


Figure 4. Chromatogram of B-100 biodiesel using the high resolution Scion Biodiesel for FAME column according to EN-14103 to separate the FAME isomers.

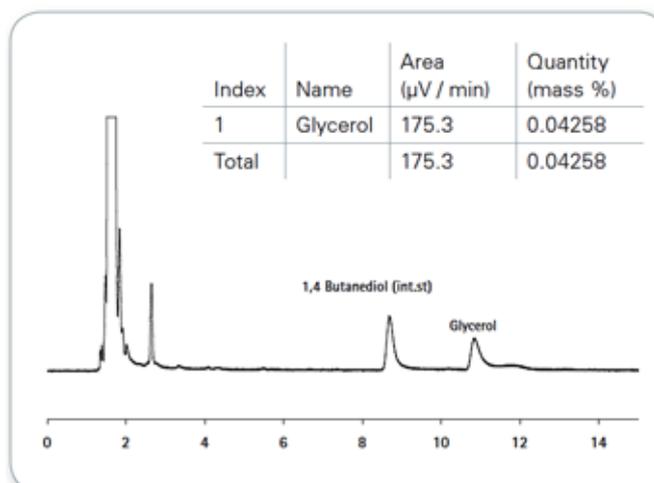


Figure 5. Chromatogram of free glycerol (glycerin) extracted from the biodiesel according ASTM D 6584.

● Scion GC Biodiesel Analyzers



Figure 6: A two method 436-GC Biodiesel system with SHS-40 headspace sampler and CP-8410 liquid sampler.

With the 436-GC, a combination of methods is available. Using the dual injection single FID configuration in combination at least two analyses (methanol, glycerin or Fames) are merged into the 436-GC. With the SHS-40 headspace sampler and the CP-8400 liquid sampler the system is fully automatable.

Including with the biodiesel analyzers, the powerful compassCDS is available to convert the analysis results into sample data. Especially the total glyceride content according to ASTM D 6584 and EN 14105, is calculated completely within the software by converting the areas of the peaks and peak groups into its glyceride content using the internal standards and glyceride contribution factors. Automatic summation results into the total glyceride content.

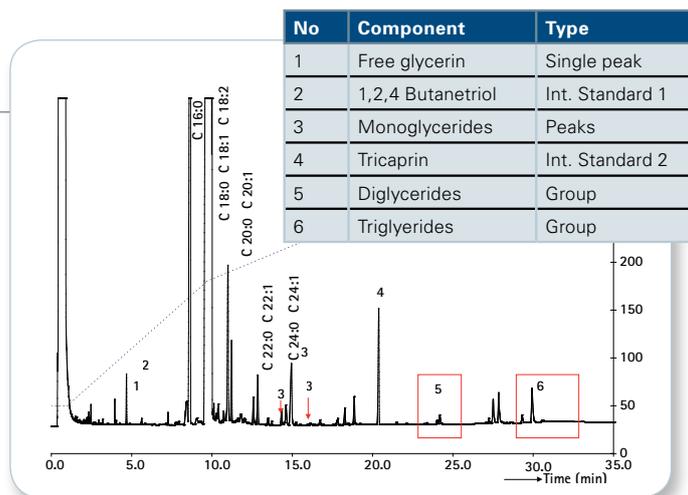


Figure 7: Chromatogram of B-100 biodiesel obtained via EN-14105.

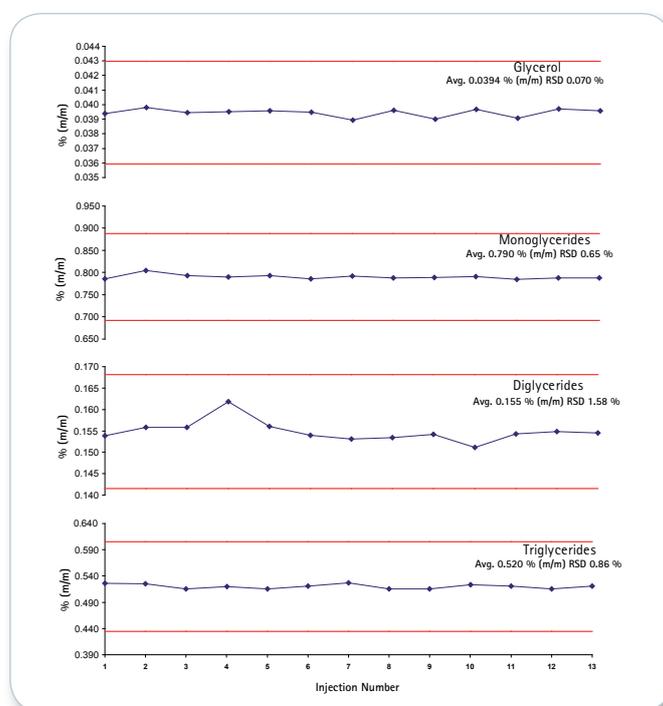


Figure 8: Typical repeatability results of 13 sequential injections of a spiked B-100 biodiesel sample, using the BR-Biodiesel column. Red lines represent the maximum allowed variation per standard method EN-14105.

Description	ASTM D 6584	EN-14103	EN-14105	EN-14106	EN-14110
	Free and total glycerin	Ester and Linoleic acid methyl esters	Free and total glycerin and Mono, Di and Tri-glycerides	Free Glycerol	Methanol
GC Columns					
BR-Biodiesel for Glycerides, 15 m x 0.32 mm x 0.1 µm + 2 m x 0.53 mm ID retention gap	BR29709				
BR-Biodiesel for Glycerides, 15 m x 0.32 mm x 0.1 µm	BR29707				
BR-Biodiesel for Glycerides, 10 m x 0.32 mm x 0.1µm + 2 m x 0.53 mm ID retention gap			BR29710		
BR-Biodiesel for Glycerides, 10 m x 0.32 mm x 0.1µm			BR29708		
BR-SWax for FAME Fused Silica, 30 m x 0.32 mm x 0.25 µm		BR89376			
BR-1 for Methanol Fused Silica, 30 m x 0.32 mm x 3.0 µm					BR89816
BR-Q PLOT, 10 m x 0.32 mm x 10 µm				BR8057-107	

Table 3: Recommended Scion columns.

Scion-Certified Consumables for Your SCION GC Series

Scion GC columns span a broad range of column diameters, stationary phases, and capillary column materials: Fused Silica (FS) and Inert Steel (IS). Ideal for either routine or research type analyses. Scion GC column offerings bridge across many important applications and include a number of offerings such as:

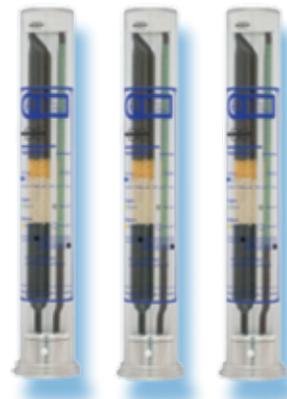
- Standard WCOT (Wall Coated Open Tubular)
- Solid Stationary Phase PLOT (Porous Layer Open Tubular)
- Inert Steel Micro-Packed and Packed



Super Clean™ Gas Filters

Scion Gas Purification Systems have the range to satisfy your needs from individual to combination filters, from Ultra purity combined with Ultra capacity, to all in one solution kits. Innovative features designed into the product yield extensive benefits to the user.

- Ultra-high capacity for long life, less change and improved productivity
- High-purity output ensures 99.9999% Pure Gas
- "Quick connect" fittings for easy, leak-tight filter changes
- Glass internals prevent diffusion; plastic externally for safety
- Easy-to-read indicators for planned maintenance and improved up-time



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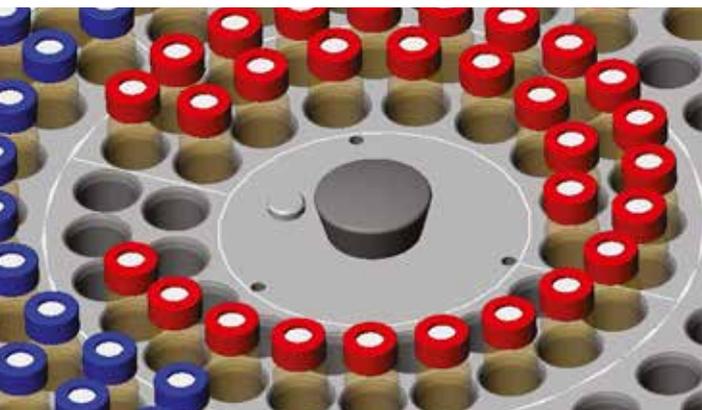


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DHA Analyzer Family

- Optimized Solutions for Detailed Hydrocarbon Analysis



The DHA Analyzer is a complete high resolution gas chromatography solution for the analysis of hydrocarbons in petroleum streams. It is capable of performing all of the standard methods including the analysis of light petroleum streams and crude oil light end.

Key Benefits include:

■ **Compliant with all industry standard methods**

Be confident using Scion's DHA Analyzers, which are configured in accordance with all the established standard methods including ASTM D6729, D6730, D6733, D5134, D6623, IP 344/DHA "Front End" and "Fast DHA"

■ **Complete and fully integrated solution**

DHA Analyzers come complete with everything you need to be up and running quickly.

■ **Powerful and easy-to-use analyzer**

With relatively little training, operators can generate outstanding analysis results day after day.

■ **Save time**

Easily generate reports with a few mouse clicks and reduce analysis time using "Fast DHA," increasing lab productivity.

■ **Single vendor solution**

Scion's GC analyzers are built and tested at Scion's factory, as well as installed and performance-verified on-site by Scion trained and certified engineers. Rest assured that our analyzers can meet or exceed your needs throughout the instrument's lifetime.



● DHA Analyzer Family

Detailed hydrocarbon analysis is often the preferred technique to fully characterize petroleum streams. The technique is based on the identification of individual components using high performance, high resolution capillary gas chromatography.

Software Ensures Accurate Identification

To successfully apply gas chromatography to detailed hydrocarbon analysis (DHA) the analyzer must be able to correctly identify a large number of components (many eluting very closely to one another) in a complex chromatogram. The identification is based on a comparison of their individual retention index values to those in a pre-established database. Therefore, it is extremely important that the analyzer functions in a highly repeatable manner.

Because the concentration of some of the individual components can vary considerably from stream to stream, the retention times for those peaks can shift slightly. This “shift” can lead to component misidentification, particularly with peaks that elute extremely close together or

those that may partially co-elute. Scion’s based DHA software includes a unique Peak Asymmetry Correction Algorithm to overcome this challenge. It accurately predicts the peak identity even if there is a large concentration change. This dramatically simplifies the operator’s job because a single analysis method/retention index database can be used for widely different streams.

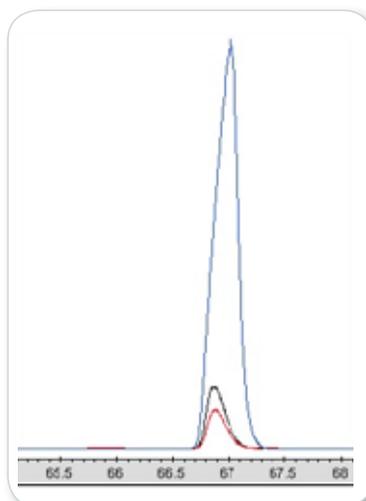


Figure 1: Column overloading has caused a retention time shift by almost 15 seconds. But with the unique peak asymmetry correction algorithm, the retention time is correctly predicted allowing the use of a single database

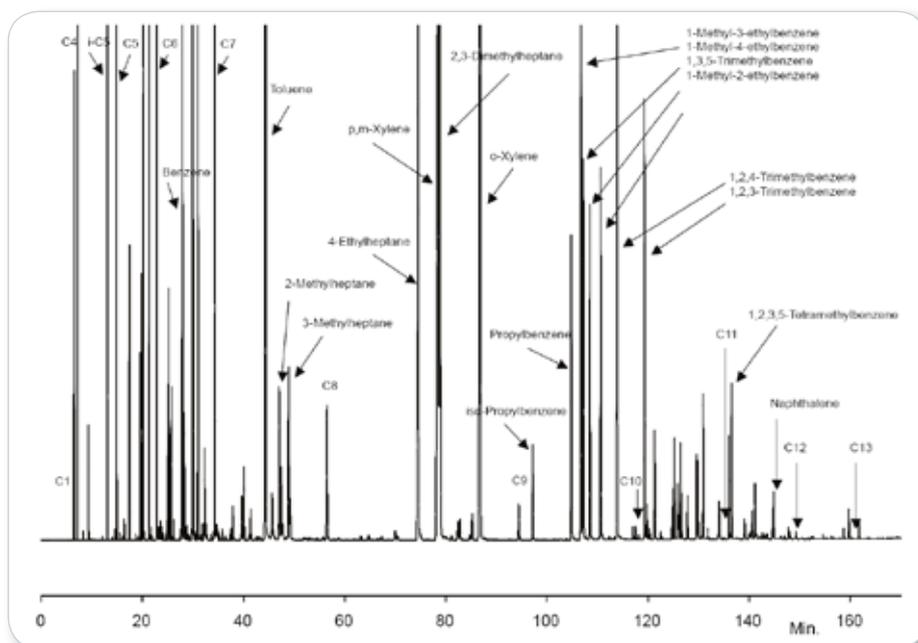


Figure 2: Detailed hydrocarbon analysis of a reformate sample showing aromatics identification according ASTM D6730

● Standard Methods

Selecting Individual Peaks and Updating the Database

The DHA software includes a Peak Select and Database Update function to make identification of unknown peaks as straightforward as possible. The system automatically provides the operator with detailed comparative retention index information for each "unknown" peak including a highlighted "best fit" indicator, making it easy for the operator to determine the ID.

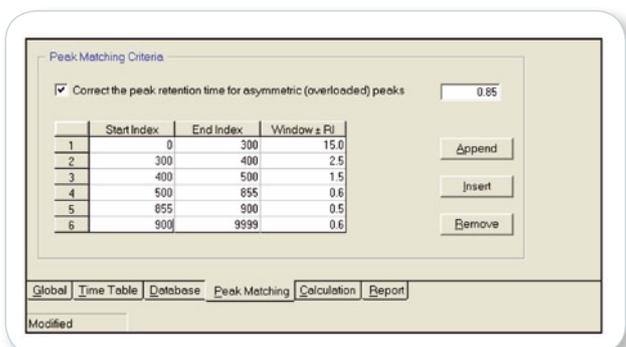


Figure 3: Assigning custom peak matching criteria is easy.

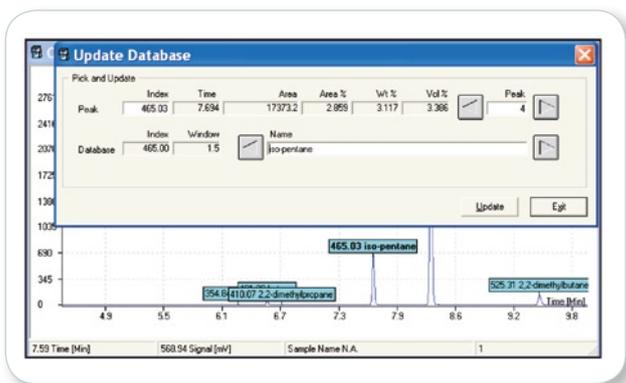


Figure 4: DHA provides an easy-to-use graphical means to select peaks and update the database

Integrated Standard Test Methods

Scion's DHA analyzers are compliant with the following methods:

- ASTM D6729
- ASTM D6623
- ASTM D6730
- "Fast" DHA
- ASTM D6733
- IP 344 "Front end"
- ASTM D5134

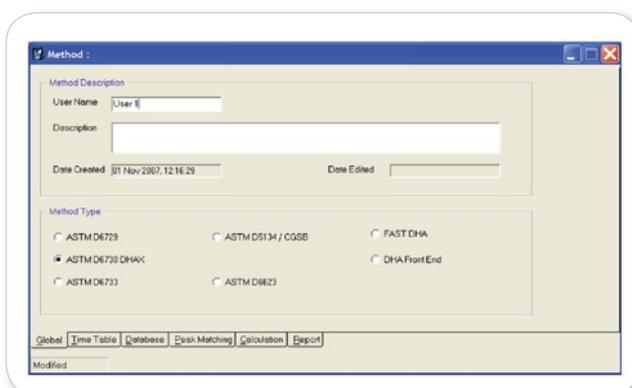


Figure 5: Choosing a preferred standard method is easy with the DHA software

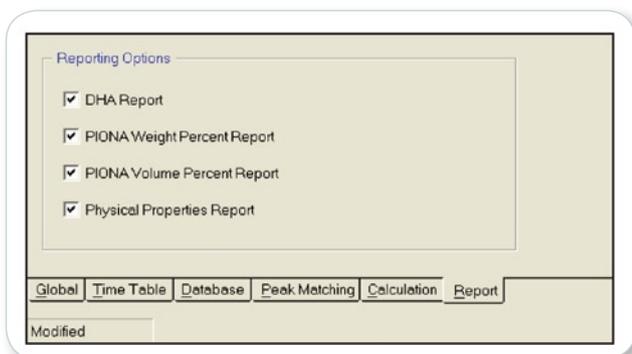


Figure 6: Choosing report options is simple

Although each DHA analyzer is configured, tested and certified at the factory for a standard method specified by the customer, the DHA software permits the operator to utilize any of the other popular standard methods as well. And, because of the outstanding performance and flexibility of the 450-GC and Galaxie software design, Scion is able to quickly modify the existing methods or add new ones if required as a result of the on-going "dynamic" industry standard processes.

Powerful Reporting is Built-in

Scion's DHA software includes several report options to accommodate the standard methods and/or to meet the customer's special needs. These include:

- Carbon number distribution
- PIONA report; (weight and volume percentage by hydrocarbon group)
- Physical properties calculations; specific gravity and molecular weight
- True distillation profile
- RON/MON specification

● Reduce Sample Analysis Time With “Fast DHA”

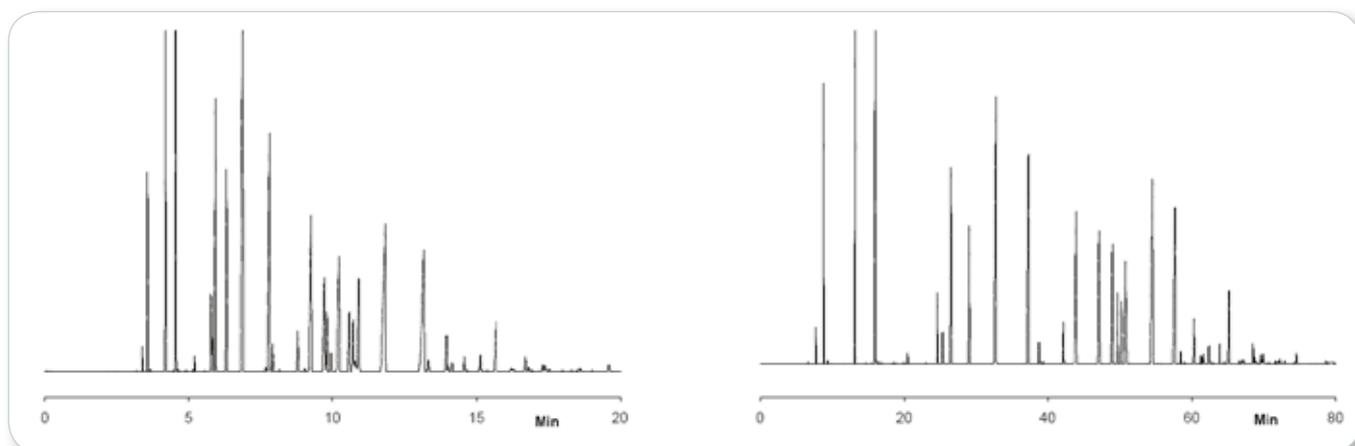


Figure 7: These chromatograms illustrate the decreased analysis time using the “Fast DHA” method. Chromatogram of a naphtha sample run on a 40 m X 0.10 mm X 0.2 µm film CP-Sil PONA CB using the “Fast DHA” method (left). Chromatogram of the same sample, but run on a 100 m X 0.25 mm ID X 0.5 µm film CP-Sil PONA CB column using standard method D6729 (right). Note reduced analysis time from ~80 minutes to ~20 minutes; almost four-fold.

DHA Analyzer Includes These Key Components

- Scion 456-GC high performance gas chromatograph equipped with:
 - Split/splitless capillary injection port
 - High performance capillary column (dependent on specified method on order)
 - Flame ionization detector (FID)
 - Full electronic flow control (EFC) of all gases
- State of the art backflush capabilities for the IP 344 „Front End” method
- CP-8400 or CP-8410 automatic liquid sampler
- compassCDS for system control, data acquisition and report generation
- Galaxie based DHA application software
- Computer/monitor
- Pre-loaded standard methods
- Factory test
- Reference chromatogram
- Reference standard for use in conducting on-site performance verification

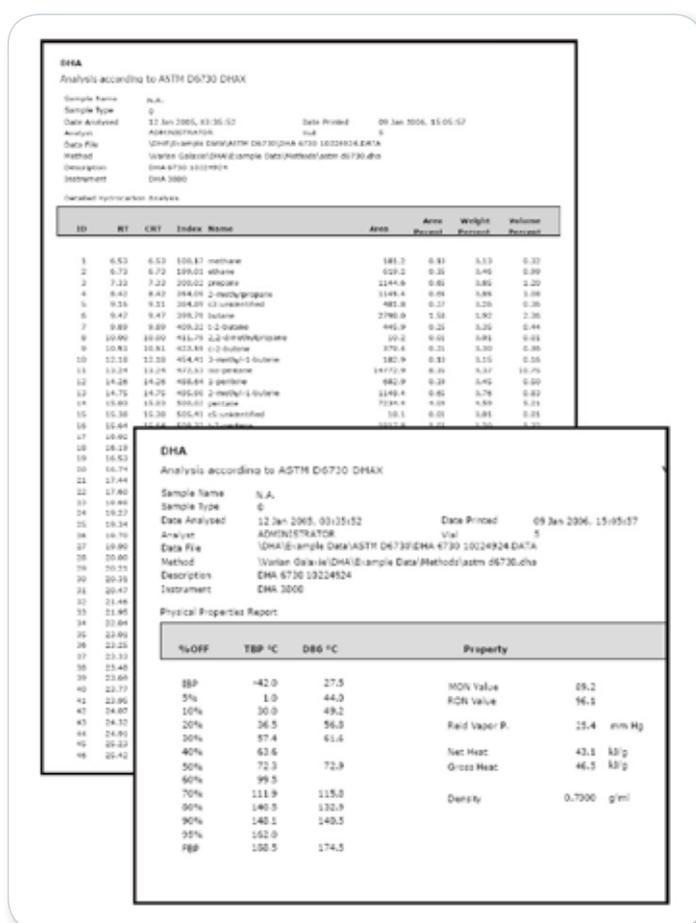


Figure 8: Physical properties and detailed hydrocarbon report

Chemical Analysis Solutions

GC quadrupole mass spectrometers

The Scion MS delivers the performance you've come to expect from an industry leader in quadrupole innovation. It features an 1200 Da mass range, superior negative ion sensitivity, and unmatched robustness in its performance class. The Scion MS delivers femtogram sensitivity and a wide array of chromatographic and ionization configurations to uniquely match your needs - all in less than 72 cm. (28 in.) of linear bench space!



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Greenhouse Gas Analyzer

- Analyze Greenhouse Gases in (Stack) Air, Water, Soil or Organic Materials



Scion Greenhouse Gas Analyzer

The Scion Greenhouse Gas Analyzer provides a turnkey solution for the analysis of the greenhouse gases methane, carbon dioxide and nitrous oxide in stack or ambient air. With the addition of a PAL Combi-xt Headspace sampler, water, soil or organic material can also be analyzed for greenhouse gases. The analyzer is configured with two chromatographic channels. Channel one is equipped with a TCD and an FID in series for the analysis of carbon dioxide and methane. The second channel is equipped with an ECD for the analysis of nitrous oxide. Optionally, this channel can be configured for the analysis of chlorofluorohydrocarbons and/or sulfur hexafluoride.

Optimized for the analysis of greenhouse gases methane, carbon dioxide and nitrous oxide

The system is configured and fully tuned and tested at the factory using all Scion components to ensure trouble-free operation. As a further guarantee, the system is installed and performance checked by Scion at the customer's site.

A turn-key solution

Scion's multi-channel 456-GC and state-of-the-art compassCDS chromatography software form a powerful combination, ensuring ease-of-use and fast and reliable results. In addition, this system does not require a high degree of operator skill.

Operational procedures fully documented

The Scion Greenhouse Gas Analyzer not only incorporates proven GC hardware and software but is also pre-loaded with analysis methods and documentation specific to the application. This makes the analyzer very efficient and cost-effective.

Flexibility

The standard configuration analyzes carbon dioxide, methane, and nitrous oxide in stack and ambient air. When a PAL Combi-xt Headspace sampler is used, the matrix can be extended with water, soil, and organic material. The application field can be extended to also analyze SF6 and CFCs.

Since the middle of the last century, the average temperature of the earth's surface has increased. This phenomenon is known as global warming, and is thought to be largely due to increasing concentrations of greenhouse gases in the atmosphere, such as methane, carbon dioxide and nitrous oxide. These gases make the earth atmosphere recycle the heat coming from its surface, creating a "green-house" effect.

Human activity is believed to increase the amount of greenhouse gases in the atmosphere, and so there is an increasing need to measure these gases routinely in air. Furthermore, studies are undertaken to analyze the gases emitting from other sources such as water, soil and organic materials. The Scion Greenhouse Gas Analyzer was developed to continuously measure greenhouse gases in these various matrices.





Analyzer Overview

The Scion Greenhouse Gas Analyzer is configured with two channels. In the first channel, the carbon dioxide and methane are separated from air and sent to a Thermal Conductivity Detector (TCD) and a Flame Ionization Detector (FID) in series.

The second channel separates the nitrous oxide from water and sends it to an Electron Capture Detector (ECD). The water is back flushed to vent. The application can be extended to chlorofluorohydrocarbons (CFCs) and/or sulfur hexafluoride (SF6). These components can also be analyzed on the second channel.

When other sources need to be analyzed, a PAL Combi-xt Headspace sampler is needed. Water, soil or organic material can be analyzed using this headspace sampler.

Specifications

Applicability: The determination of greenhouse gases - methane, carbon dioxide and nitrous oxide in air, water, soil or emitting from (organic) matter.

Analysis Time: Approximately 6 minutes.

Minimum Detectability: The minimum detection level is better than 50 ppb for methane and nitrous oxide and better than 10 ppm for carbon dioxide.

Repeatability: Better than 2% relative standard deviation (on peak area) at ambient concentration levels, measured over 20 consecutive runs.

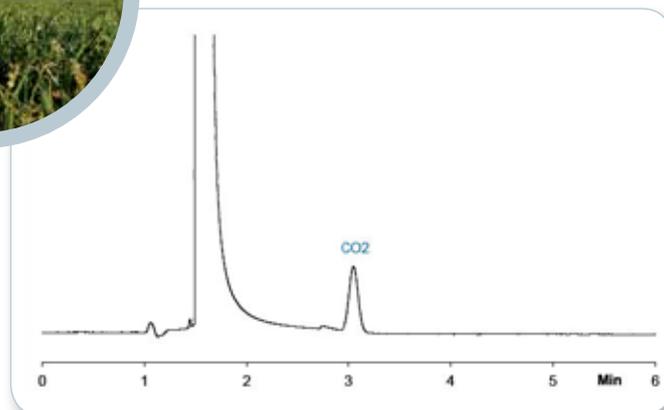


Figure 1: Carbon dioxide analyzed on the TCD.

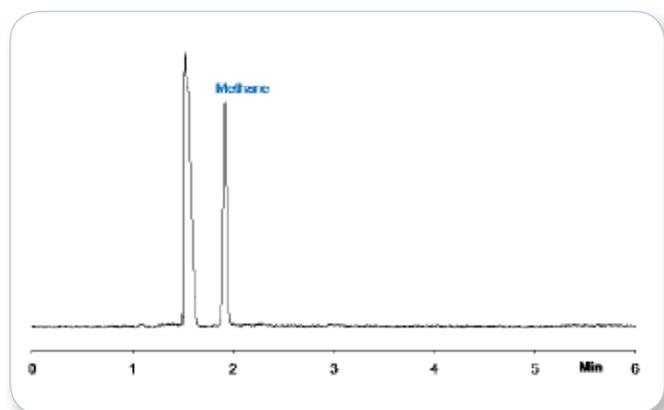


Figure 2: Methane analyzed on the FID.

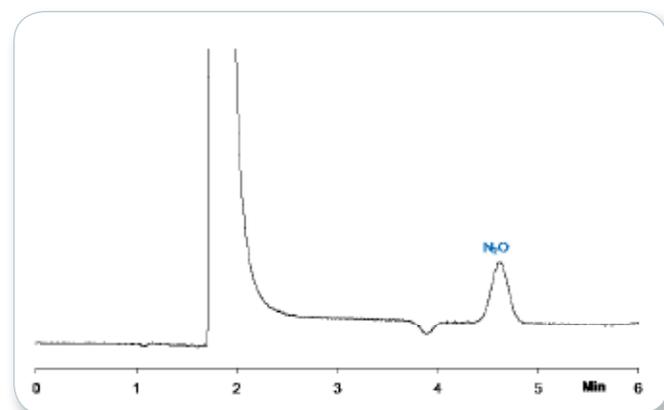


Figure 3: Nitrous oxide analyzed on the ECD.

Scion-Certified Consumables for Your SCION GC Series

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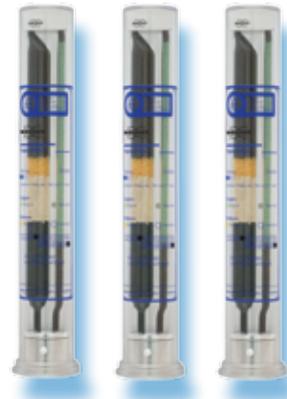
- Standard WCOT (Wall Coated Open Tubular)
- Solid Stationary Phase PLOT (Porous Layer Open Tubular)
- Inert Steel Micro-Packed and Packed



Super Clean™ Gas Filters

Scion Gas Purification Systems have the range to satisfy your needs from individual to combination filters, from Ultra purity combined with Ultra capacity, to all in one solution kits. Innovative features designed into the product yield extensive benefits to the user.

- Ultra-high capacity for long life, less change and improved productivity
- High-purity output ensures 99.9999% Pure Gas
- “Quick connect” fittings for easy, leak-tight filter changes
- Glass internals prevent diffusion; plastic externally for safety
- Easy-to-read indicators for planned maintenance and improved up-time



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Low Level Oxygenates Analyzer

- Trace Analysis of Oxygenates
in Hydrocarbon Matrices



The determination of sub to high ppm levels of ethers, alcohols, aldehydes and ketones in different hydrocarbon matrices is a recurring challenge in the petroleum refining and petrochemical industry. The Scion Low Level Oxygenates Analyzer is an easy to use 'turnkey' solution to meet this analysis challenge.

Key Benefits include:

- **Factory optimized to ensure excellent analysis results**

The system is configured, optimized and tested at the factory to analyze oxygenated components at concentrations as low as 100 ppb in streams with a final boiling point of up to 250 °C and no interference from the hydrocarbon matrix. An optimized system for the analysis of oxygenates at trace level.

- **A powerful GC solution, yet extremely easy to operate**

The Scion GC and compassCDS Chromatography Data Handling Software, is a very powerful combination and is key to the system's ability to generate reliable results. The system can be used successfully without a high level of operator skill.

- **A flexible, economical analysis solution**

The analysis system is configured to accommodate gas, liquid and liquified petroleum gas (LPG) streams. Configured with both a gas sampling valve and a liquid sampling valve, all three samples can be injected without the need to change the system configuration.

- **A cost effective, time-saving solution**

All Scion GC analyzers not only include proven GC hardware and software but are also pre-loaded with analysis methods and documentation specific to the method required. All operational procedures are fully documented.

- **A comprehensive, single vendor solution**

The hardware, software, application optimization, documentation, installation and support services are all provided by Scion, allowing analysis to be carried out quickly and efficiently by one dependable supplier.



Figure 1: The analyzer incorporates the Scion GC

● Low Level Oxygenates Analyzer

Oxygenated compounds can be present in hydrocarbon streams for a variety of reasons. For example, methanol is added to crude oil to reduce the formation of hydrates during transportation and storage. Unfortunately, the presence of methanol in downstream operations is highly problematic.

Clean-up processes like hydro-treating are used in an attempt to remove oxygenated compounds. But even at trace levels (sub ppm), oxygenates quickly degrade or destroy expensive process catalysts, (e.g. polymer production).

Most of the standard methods for the analysis of oxygenated compounds in use today (ASTM D4815, D5599, EN 13132, DIN 1601 and DIN 51413-7) are not suitable for the analysis of oxygenates at sub ppm levels. The Low Level Oxygenates Analyzer according to ASTM D7423 has been developed to meet this low level detection requirement.

Analyzer Overview

The Low Level Oxygenates Analyzer is designed and optimized to quantify ppm and sub ppm levels of ethers (e.g. DME, MTBE, ETBE, DIPE), alcohols (e.g. methanol, ethanol, propanol), ketones (e.g. acetone, MEK) and aldehydes in various hydrocarbon matrices. In general, all oxygenated components with a boiling point of up to 100 °C can be analyzed. The sample can be a gas, LPG or liquid under ambient conditions with a final boiling point up to 250 °C.

The system is comprised of a Scion 456 GC configured with gas and liquid sampling valves, two high performance capillary analysis columns, digitally controlled pneumatics including a 'fluidic' switch and Flame Ionization Detector (FID). An optional 'pressure station' can be added to eliminate the possibility of losing sample due to evaporation when analyzing LPG.

The GC is controlled via the Compass-CDS Chromatography Data Handling Software,

which acquires data, processes it and generates analyses reports. The gas or liquid sample is injected via a gas or liquid sampling valve onto the first of two columns (BR-1). A fluidic switch is positioned between the two columns. The 'lighter' fraction (containing the oxygenates) is separated from the rest of the stream components on the BR-1 column.

The 'heavier' components are then back-flushed to vent. The fraction containing the 'lighter' components is transferred onto the second column (LOWOX) using the fluidic switch. The LOWOX column is used to separate the individual oxygenated components from the bulk hydrocarbons. The LOWOX column was specially developed in the 1990s for the trace analysis of oxygenates in hydrocarbon streams. It offers unequalled selectivity for oxygenated compounds.

Due to its high selectivity, large amounts of sample can be introduced onto the LOWOX column which, through the use of the sensitive FID, enables the system to achieve the required low detection levels for oxygenated compounds.

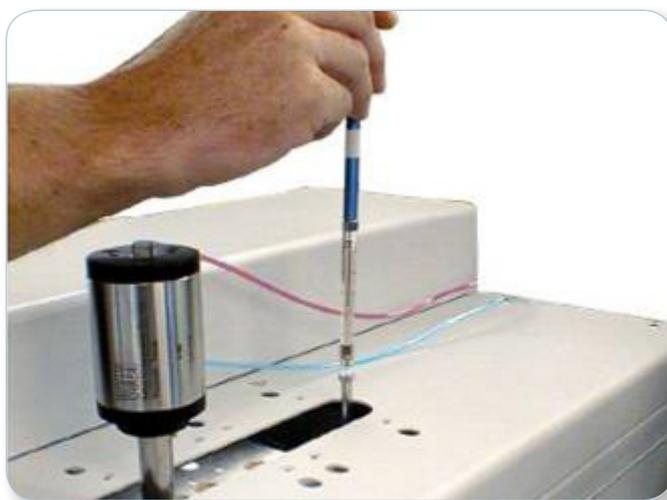


Figure 2: The analyzer includes both gas and liquid sample valves for maximum flexibility.

● Analysis of Oxygenates at sub-ppm Levels

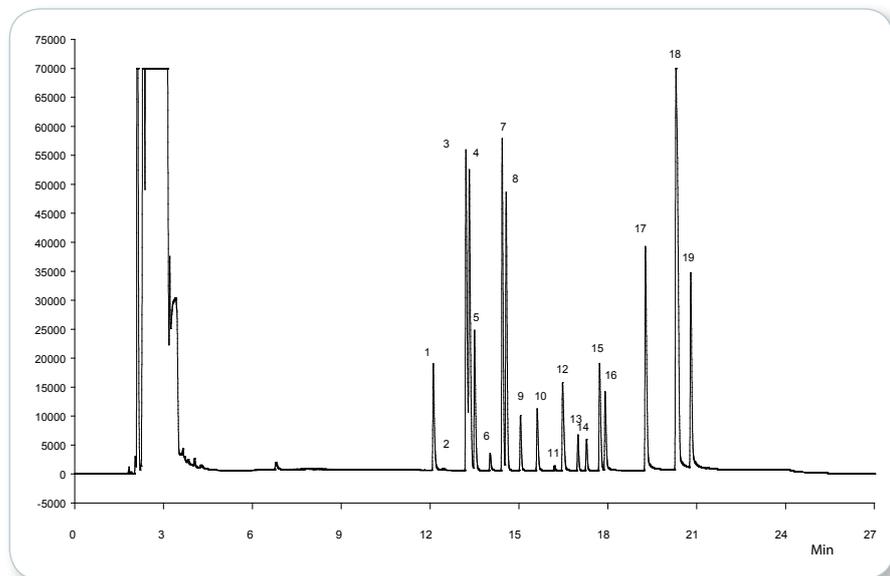


Figure 3: A typical chromatogram showing a wide range analysis of a liquid sample stream

Calibration is performed using several concentrations of a multi-component standard mixture with varying known/measured amounts of each oxygenated compound.

The system automatically generates a calibration curve for each analyte. The samples are analyzed, each of the oxygenates are automatically quantified and a report of the results is printed at the end of each analysis cycle. (Typical performance results are depicted in Table 1.)

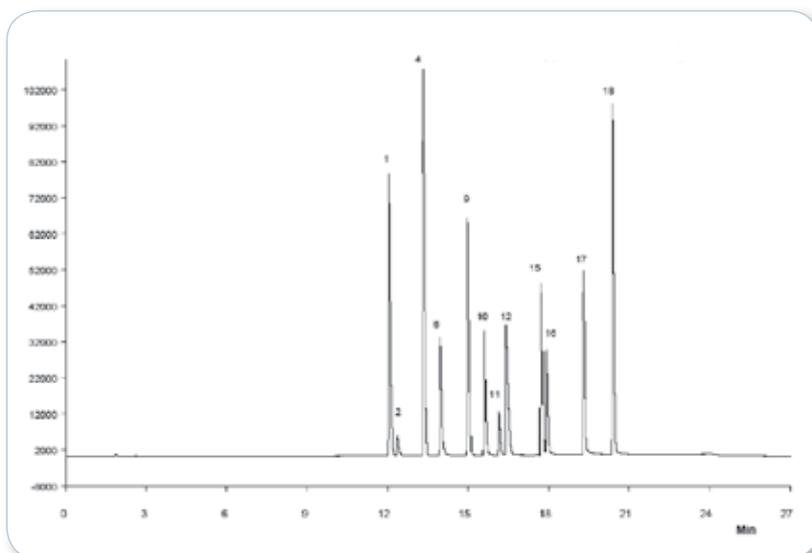


Figure 4: Typical chromatogram showing a MTBE range analysis of a gas stream.

Peak Identification

- | | |
|-----------------------------|-------------------------|
| 1. Diethylether | 11. Methanol |
| 2. Acetaldehyde | 12. Acetone |
| 3. Ethyl tert. Butyl ether | 13. Isovaleraldehyde |
| 4. Methyl pert. Butyl ether | 14. Valeraldehyde |
| 5. Diisopropylether | 15. 2-Butanone |
| 6. Propanal | 16. Ethanol |
| 7. Tert. amyl methyl ether | 17. 1-Propanol |
| 8. Propylether | 18. Tert. butanol |
| 9. Isobutyraldehyde | 19. 2-Methyl 1-propanol |
| 10. Butyraldehyde | 19. 1-Butanol |

● Typical Performance

Table 1. Typical repeatability in the analysis of oxygenates in a gas stream

	ETBE	MTBE	Methanol	Aceton	MEK	Ethanol	1-Propanol
Run 1	579554	490771	59388	224339	338840	129250	224083
Run 2	578838	491908	59310	224407	339195	129779	225111
Run 3	585890	481217	60135	225063	340116	131457	227596
Run 4	579313	491673	59663	224583	339338	130075	225695
Run 5	578500	492449	59897	224743	339606	131011	227058
Run 6	577796	493989	59766	224583	339482	130628	226651
Run 7	579844	490743	59879	224593	339622	130645	226309
Run 8	580253	487207	59800	224570	339470	130874	226918
Run 9	580592	484805	59856	224539	339556	130808	226284
Run 10	578693	487272	59946	224530	339893	131131	226630
N	10	10	10	10	10	10	10
Mean	579927	489203	59764	224595	339512	130566	226234
Std. Dev.	2142.60	3775.30	238.60	187.10	334.20	637.80	975.60
RSD %	0.37	0.77	0.40	0.08	0.10	0.49	0.43

Table 2. Typical analyzer performance.

Minimum Detection Level	100 ppb/Weight
System Linearity	100 ppb to 500 ppm
Typical RSD %	For gas streams; < 2 % for MTBE, methanol and ethanol. For liquid streams; < 2.5 % for MTBE, methanol and ethanol.

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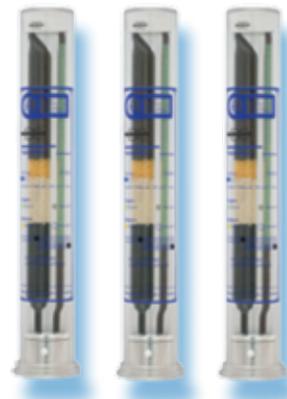
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Refinery Gas Analyzer

- Optimized GC Analysis Solutions

Scion Refinery Gas Analyzer

The source and composition of refinery gases varies considerably. Measuring gas composition precisely and accurately is a significant challenge in today's refinery operations. Scion's refinery gas analyzers are designed to deliver superior, reliable results for a wide range of sources and analysis throughput requirements.

- A range of refinery gas analysis (RGA) solutions. Scion offers RGA solutions to meet the broadest range of stream sample types and throughput requirements.
- A powerful, easy to use GC solution. Scion's 456-GC and compassCDS chromatography software is a very powerful combination designed to achieve the best possible results. In addition, these systems do not require a high degree of operator skills.
- A highly flexible solution for analysis. The Scion RGA solutions can optional be configured to analyze high pressurized gas and liquefied petroleum gas (LPG) through the use of a fully integrated Micro-Gasifier, giving the flexibility to accommodate a wide range of stream types.
- Operational procedures are fully documented. Scion RGA analyzers not only incorporate proven GC hardware and software but also arrive pre-loaded with analysis methods, and include documentation specific to the application required.
- A comprehensive, single vendor solution. Scion provides complete solutions. The hardware, software, application optimization, documentation, installation and performance verification are all provided by Scion, offering an all inclusive, convenient analysis solution.



Figure 1: The Scion 456-GC RGA has outstanding flexibility, analytical power and robustness.

● Key Benefits

Scion solutions for refinery gas analysis

Typical sources for refinery gases include atmospheric or FCC overheads, ethylene, propylene production, fuel gas, stack gas and off gas from desulfurization. The physical stream types range from gas to highly pressurized gas or liquefied gases. Scion's refinery gas analyzers (RGA) are 'turnkey' systems pre-configured and tuned at the factory to conform to industry standard methods including: UOP 539, DIN-51666 and ASTM D2163. The RGA systems are based on the Bruker 456-GC. To perform good analysis, the RGA is optional equipped with an integrated micro-gasifier. This sample conditioning device ensures complete vaporization of LPGs and high pressures samples to prevent any sample discrimination prior injection.

The Analyzers employ a proven and optimized multi-channel approach. They determine the concentration of individual saturated and unsaturated hydrocarbon components up to and including C5 (C6 and higher components as a composite peak) and all permanent gases, including hydrogen and hydrogen sulfide in a single analysis. Included in every system is Scion's powerful compassCDS chromatography software to provide complete analyzer control, data acquisition and flexible report generation.

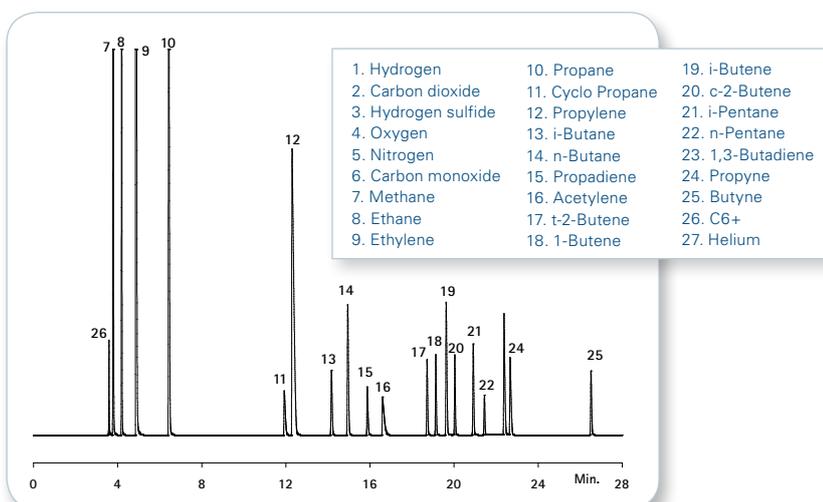


Figure 2: The separation of light hydrocarbons using the Standard RGA.

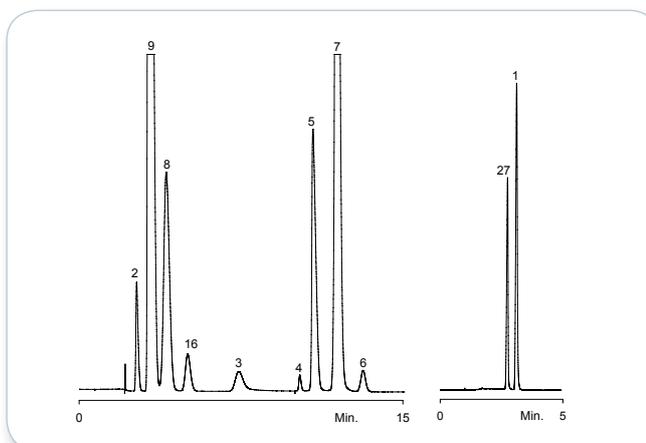


Figure 3: The analysis of the permanent gases and hydrogen (and helium) using the Standard RGA.



Figure 4: The RGA analyzers are applicable to a variety of different hydrocarbon streams.

● Scion Refinery Gas Analyzers

Scion offers two RGA systems to meet the widest range of analysis requirements:

- Standard RGA:** A three channel 456-GC with a multi-valve design using both capillary and packed columns. The first channel is optimized for the analysis of permanent gases, the second is designed for light hydrocarbons, and the third specifically for hydrogen. The system is configured and fully tested in accordance with industry standard methods. Total analysis time for all components is less than 25 minutes.

The standard RGA analyzer is the most powerful tool to analyze the widest range of RGA type streams. This includes sample streams with a high % level of components as in ethylene, propylene and butylene streams.

- Rapid RGA:** A three channel 456-GC that utilizes a multi-valve design in which the packed columns used in the Standard RGA are replaced by micro-packed columns in both the hydrogen and permanent gas channels. Since the micro-packed columns are installed in a separate heated zone, the capillary columns located in the GC oven can be temperature programmed in a more aggressive manner. For high sample analysis demand, the Rapid RGA Analyzer concept provides a substantial reduction in overall analysis time of 5 minutes (7 minutes with H₂S) compared to the 25 minutes with the standard RGA.

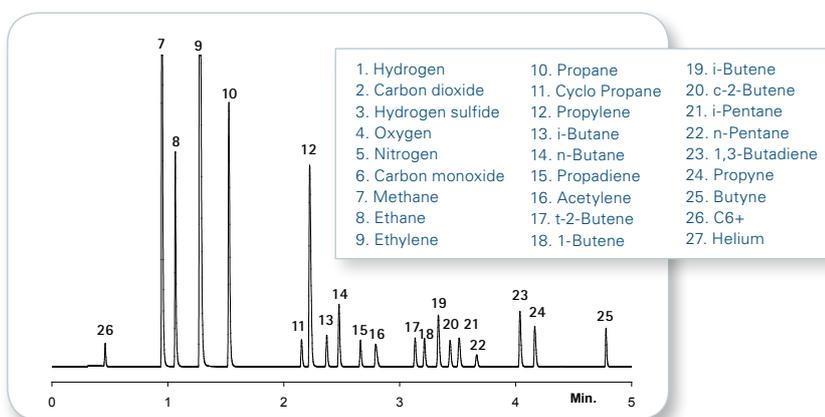


Figure 5: The analysis of light hydrocarbons using the Rapid RGA, with complete separation in less than five minutes.

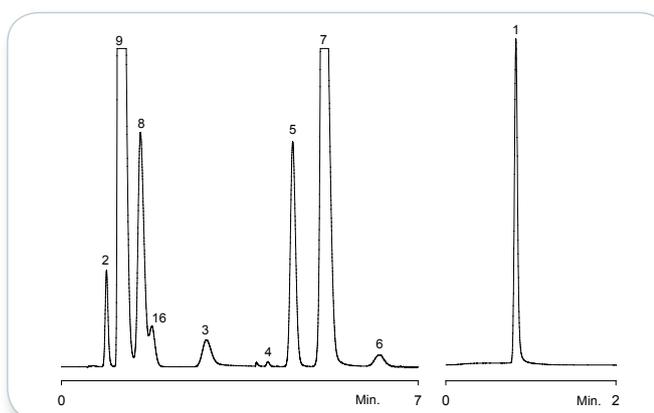


Figure 6: The analysis of permanent gases and hydrogen using the Rapid RGA.

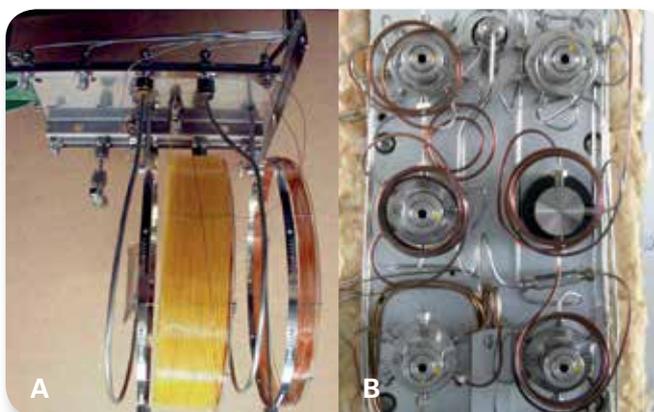


Figure 7: A shows a 'traditional' RGA with all columns mounted in oven. B shows the micro-packed columns mounted in the separate heated zone in the Rapid RGA.

● Scion Refinery Gas Analyzers

Table 1: RGA Analyzer Characteristics.

Characteristics	Standard RGA	Rapid RGA
No. of Channels/Detectors Used	3	3
No. of Column Ovens	1	2
Analysis Time	25 min	5 min (7 min with H ₂ S)
Repeatability	<1%	<1%
Linear Bench Space Required	66 cm/26 in.	66 cm/26 in.
Minimum Component Detection Level	0.01% all components except H ₂ S = 0.05%	0.01% all components except H ₂ S = 0.05%
Suitability		
Typical Refinery Gas	Excellent	Excellent
Impurities in Bulk Ethylene	Excellent	Excellent
Impurities in Bulk Propylene	Excellent	Good
Impurities in Bulk C4	Good	Good

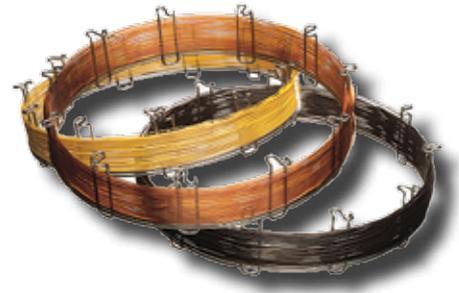
Table 2: Multiple channels of data are conveniently combined into a single analysis report.

Peak No.	Peak Name	Channel	RT (min.)	Result (g/l)	Norm. (%)	Area (uV/Sec.)
1	Hydrogen	Middle (TCD)	1.6967	36.0300	22.7681	390257
2	Carbon dioxide	Front (TCD)	2.6000	0.1000	0.0632	13376
3	Hydrogen sulfide	Front (TCD)	-	0.0000	0.0000	0
4	Oxygen	Front (TCD)	9.9200	0.0000	0.0000	37325
5	Nitrogen	Front (TCD)	10.3267	1.1990	0.7577	2122071
6	Carbon monoxide	Front (TCD)	-	0.0000	0.0000	0
7a	Methane	Front (TCD)	11.1917	11.9900	7.5767	1394584
7b	Methane	Rear (FID)	3.7350	11.9900	7.5767	1492388
8a	Ethane	Front (TCD)	3.5367	17.9900	11.3682	2867688
8b	Ethane	Rear (FID)	4.1283	17.9900	11.3682	4480322
9a	Ethylene	Front (TCD)	2.9550	29.9800	18.9449	4139442
9b	Ethylene	Rear (FID)	4.7217	29.9800	18.9449	7411134
10	Propane	Rear (FID)	6.1933	0.1990	0.1258	71402
11	Cyclo Propane	Rear (FID)	-	0.0000	0.0000	0
12	Propylene	Rear (FID)	-	0.0000	0.0000	0
13	i-Butane	Rear (FID)	-	0.0000	0.0000	0
14	n-Butane	Rear (FID)	-	0.0000	0.0000	0
15	Propadiene	Rear (FID)	-	0.0000	0.0000	0
16a	Acetylene	Front (TCD)	5.0283	0.5020	0.3172	49786
16b	Acetylene	Rear (FID)	16.4331	0.5020	0.3172	121300
17	t-2-Butene	Rear (FID)	18.5050	0.0990	0.0626	138647
18	1-Butene	Rear (FID)	-	0.0000	0.0000	0
19	i-Butene	Rear (FID)	19.5167	0.0990	0.0626	44492
20	cis-2-Butene	Rear (FID)	-	0.0000	0.0000	0
21	1,3-Butadiene	Rear (FID)	22.1367	0.0000	0.0000	16165
22	Propyne	Rear (FID)	-	0.0000	0.0000	0
23	C5+	Rear (FID)	2.9217	0.1000	0.0632	58164
Totals				158.2480	100.0000	

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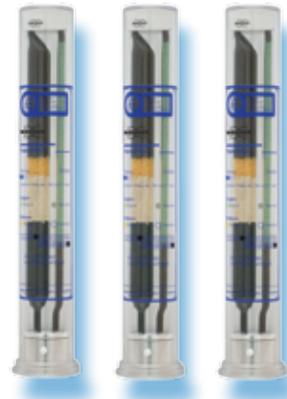
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Simulated Distillation Analyzers

- A Full Range of Simulated Distillation Analysis Solution

Scion Simulated Distillation Analyzers

Scion's range of Simulated Distillation Analyzers provide boiling point distribution up to 750°C. Designed to meet all industry standard methods, Scion's analyzer software includes both ASTM D86 and ASTM D1160 correlations.

A gas chromatographic (GC) technique, Simulated Distillation (SimDist) reproduces the physical distillation of petroleum materials and products by determining boiling point distribution. Used for controlling refinery operations, Scion's range of SimDist analyzers deliver fast, accurate standard test method results. Scion's highly automated Scion GC, compassCDS Chromatography Data Handling Software, and integrated SimDist software are also designed to meet worldwide industry standard test methods.

Key Benefits include:

- **Accurate boiling point distribution up to 750 °C**
The Scion SimDist Analyzer range tests a variety of distillates, blends, fuels, residues and crude oil, ranging from carbon number C1 to C120 and higher. This enables refinery processes to be monitored and controls product quality with fast, accurate boiling points versus mass distributions up to 750 °C.
- **Complete control from initial setup to final report**
The SimDist analyzers are controlled by Scion's advanced CompassCDS Chromatography Data System software package. The SimDist software is fully integrated into compassCDS for system operation, automation, calibration and report generation.
- **Integrated standard test methods**
Scion offers SimDist analyzers with built-in applications to help monitor refinery processes and control product quality. These applications fully comply with ASTM, IP, EN, DIN and ISO standard test methods used globally. New methods will be added to Scion's SimDist software as they are approved and released.
- **ASTM D86 and ASTM D1160 correlation**
SimDist software includes both ASTM D86 and ASTM D1160 correlations. Dedicated to a sample type, these correlations are crude independent. With the fully automated Scion SimDist Analyzer, data is generated rapidly and with increased precision, making ASTM D86 and ASTM D1160 correlations faster and easier.
- **Complete, single vendor solution**
Scion's single vendor approach includes the latest hardware, software and column technologies. Our solutions maximize uptime and increase the speed of data generation.



Figure 1a: Scion456-GC SimDist Analyzer.

● Operational Simplicity For All SimDist Methods



Figure 1b: 436-GC with Sampler

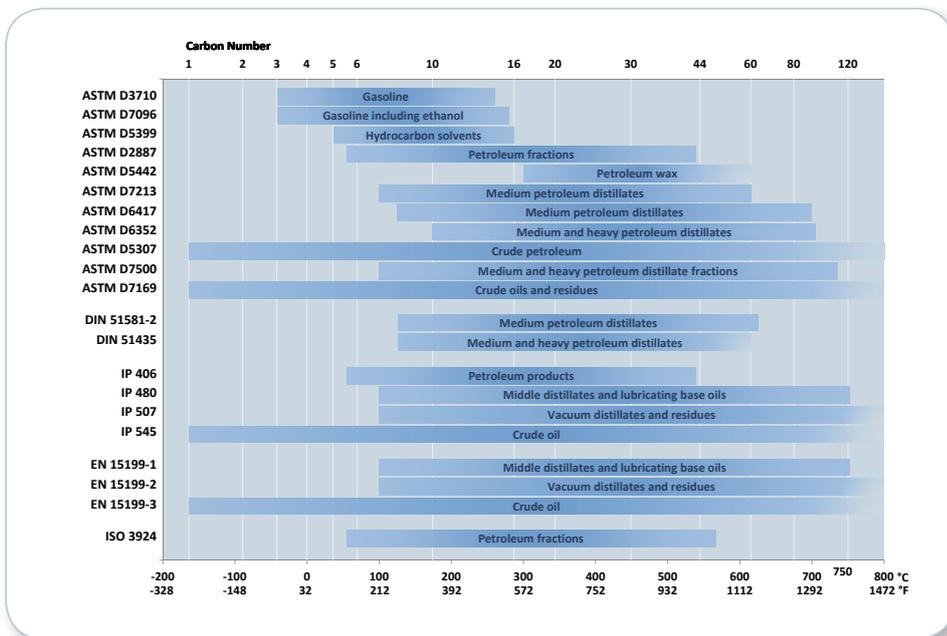


Figure 2: Standard Test Methods.

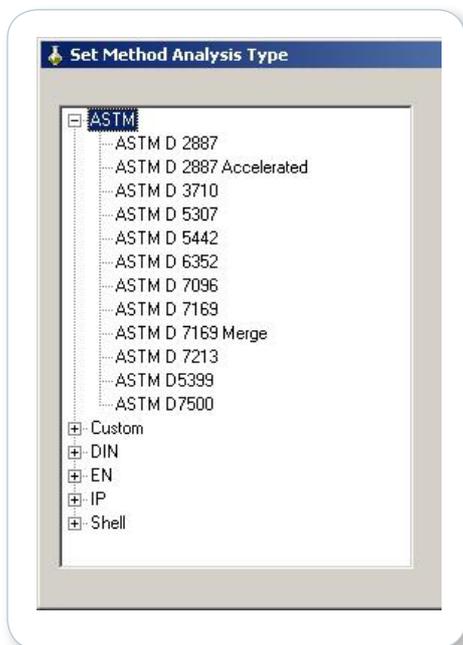


Figure 3: Easy to use industry standard test methods are fully integrated into Scion's SimDist software.

Scion's compassCDS Software has pre-programmed settings which streamline instrument setup, analysis and reporting to ensure outstanding data precision and reproducibility.

Once the preferred industry standard method is selected, Scion's SimDist software automatically corrects any offset to the baseline signal and removes the blank analysis to provide a corrected net area and precise boiling point data. In addition, users may choose to modify individual settings to suit their specific requirements.

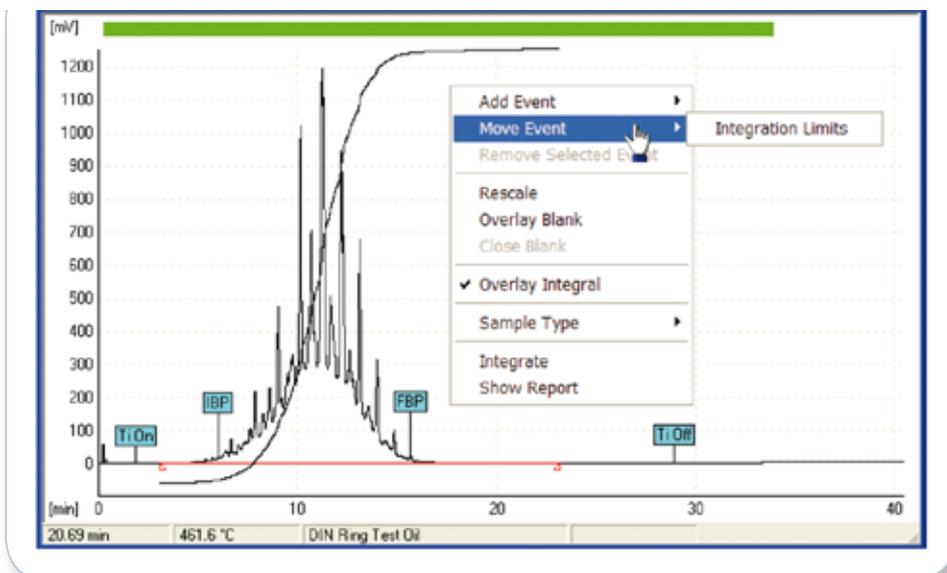


Figure 4: The intuitive interface enables easy adjustments of integration parameters and result overlays.

● Modification of Variables

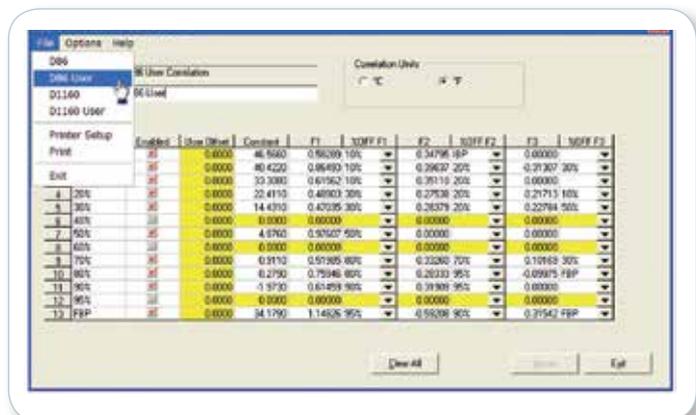


Figure 5: ASTM D86 and ASTM D1160 Correlation Editor to customize and add correlation data.

ASTM D86 and ASTM D1160 Correlation Editor

As the composition of raw materials and intermediates change, the ability to modify the ASTM D86 or ASTM D1160 variables to improve correlation results is required. The Correlation Editor also allows new correlation data at the 40 %, 60 % and 95 % cut-off points to be generated for additional information between the two methods.

Built-in Reports

Scion's SimDist software provides a wide variety of report options to meet specific lab requirements including:

- Chromatogram with merged corrected blank analysis and IBP/FBP marks versus retention time
- Table with boiling point versus percentage of sample
- Table and plot with retention time versus boiling point

- Table with D86 and D1160 correlations
- DIN Noak and motor oil volatility reports
- Table with cutpoints and fractions
- Residue analysis with recovery calculation up to C120

Simplified, Accurate Analysis

In order to obtain accurate results:

- Run a blank to obtain an accurate baseline signal
- Run a standard configuration to obtain retention time versus boiling point calibration
- Run the reference sample to validate system performance and to determine the response factor
- Analyze samples

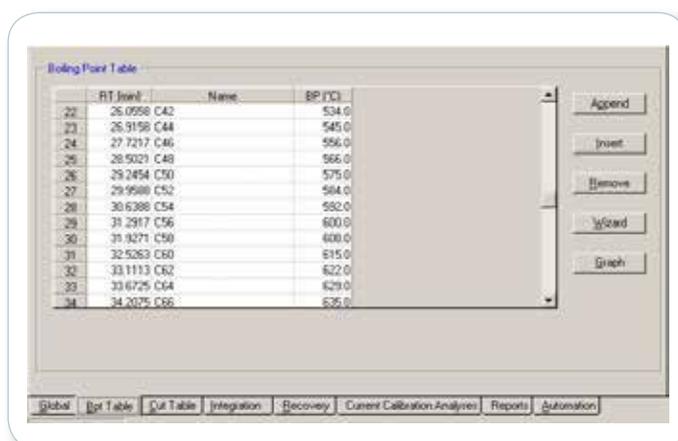


Figure 6: Accurate Analysis.

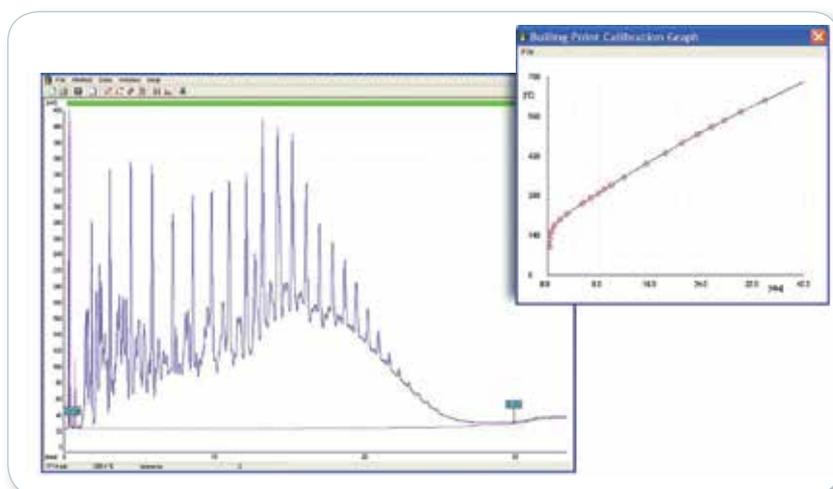


Figure 7: The integrated CompassCDS SimDist software for calibration and analysis.

● SimDist Analyzer Specifications

Hardware

- Gas chromatograph: Scion 456-GC or 436-GC
- Injector: temperature controlled on-column injector for capillary columns full Electronic Flow Control (EFC)
- Column oven: with CO₂ sub ambient cooling
- Detector: FID with EFC control
- Computer: with monitor
- Autosampler: CP-8400 or CP-8410 autosampler with carousel heating and cooling plate

Software

- Scion's compassCDS system control, data acquisition and report generation software
- Scion's compassCDS SimDist application software (fully integrated)

SimDist Analyzer Configurations Applicability

Six standard configurations are available for the test methods listed in Table 1. Optional mode kits are available to facilitate the change from one standard configuration to another. Each mode kit contains all the necessary components, including column and standard samples for a given configuration.

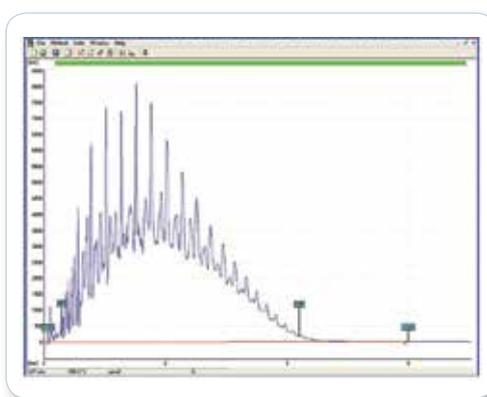


Figure 8: Diesel fuel analysis with fast 2887 SimDist method reduces analysis time by a factor of five compared to ASTM D2887 (6 rather than 30 minutes).

Sample Type	Configuration	Method	Min IBP	Max FBP
Gasoline	1	ASTM D3710	--	260°C
		ASTM D7096	--	280°C
Hydrocarbon solvents	1	ASTM D5399	37°C	285°C
Petroleum fractions	2	ASTM D2887, ISO 3924 and IP 406	55°C	538°C
	4	ASTM D 2887 accelerated	55°C	538°C
Middle distillates and lubricating base stocks	4	ASTM D7213	100°C	615°C
		ASTM D6417	126°C	615°C
		DIN 51581-2	160°C	635°C
		DIN 51435	165°C	615°C
	5	EN15199-1	100°C	750°C
		IP 480	100°C	750°C
Medium and heavy petroleum distillate fractions	5	ASTM D6352	174°C	700°C
		ASTM D7500	100°C	750°C
Petroleum wax	6	ASTM D5442	302°C	>548°C*
Vacuum distillates and residual fuels	5	EN15199-2 and IP 507	100°C	>750°C*
Crude petroleum	3	ASTM D5307	--	>538°C*
	5	EN15199-3 and IP 545	--	>750°C*
Crude oils and residues	5	ASTM D7169	--	>720°C*

* The method allows sample types that have a higher final boiling point than the maximum temperature analyzed by the method. This higher boiling fraction is reported as "greater than".

Table 1: Analyzer configurations for standard SimDist methods.

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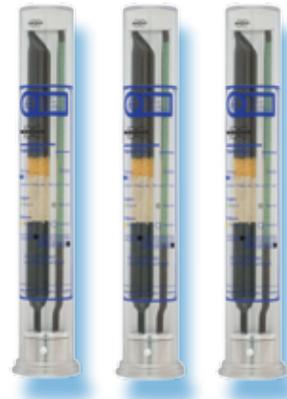
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Application Note #281753

A Complete Solution for the Analysis of Volatile Organic Compounds (VOCs) in Water

The Scion SQ single quadrupole mass spectrometer, in combination with the Tekmar Atomx™ automated purge-and-trap sample concentrator, demonstrates excellent performance for routine analysis of volatile organic chemicals (VOCs) in drinking water. The Scion SQ's high sensitivity, scan speed, and easy to use software are distinct advantages enabling laboratories to easily meet and/or exceed EPA Method 524.3 requirements.



Introduction

Demand for lower detection limits of volatile organic compounds in drinking water requires the use of a mixed mode GC/MS analysis, i.e. simultaneous full scan and selected ion monitoring (SIM) for low-level quantitation. USEPA Method 524.3 requires a full scan analysis in addition to SIM for specified target analytes.

The Scion SQ has a unique feature known as Compound Based Scanning (CBS) for easy automated setup and optimization of complex mixed mode methods. CBS makes use of libraries that store all the essential information about a compound, such as retention time, time window, qualifier and quantifier ions. Compounds are loaded directly into a method, scan times are optimized, and data acquisition and processing tables are synchronized. Managing large numbers of SIMs in mixed mode is easy.

Experimental

The Scion SQ was set up with a Tekmar Atomx™ purge-and-trap sample concentrator to achieve a highly automated and robust solution for VOC analysis.

The purge-and trap and GC parameters are listed in Tables 1a-c. The purge-and-trap conditions for drinking water come factory installed on the Atomx. A split ratio of 1:100 was used on the gas chromatographic inlet along with a BR-624 ms column (20 m x 0.25 mm x 1.0 um).

Table 1a

Variable	Value	Variable	Value
Valve Oven Temp.	150°C	Sample Preheat Time	1.00 min.
Transfer Line Temp.	150°C	Preheat Temp.	40°C
Sample Mount Temp.	60°C	Purge Time	11.00 min.
Condenser Ready Temp.	40°C	Purge Flow	40 mL/min
Condenser Purge Temp.	20°C	Dry Purge Time	0.00 min.
Pre-purge Flow	40 mL/min.	Desorb Preheat Temp	245°C
GC Start	Start of Desorb	Desorb Time	1.00 min.
Bake Time	7.00 min.	Desorb Temp	250°C
Bake Temp.	260°C	Desorb Flow	100 mL/min.
Bake Flow	300 mL/min.	Condenser Bake Temp.	200°C

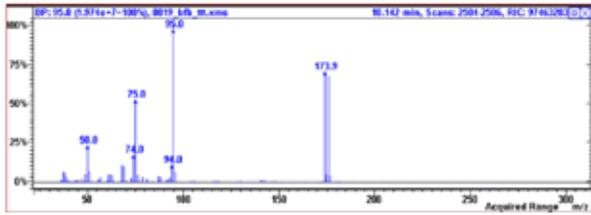
Tekmar Atomx conditions.

Table 1b

Oven	Rate	Hold	Total
Temperature, °C	°C/min	(min)	(min)
35.0	0.00	2.00	2.00
170.0	10.00	0.00	15.50
240.0	50.00	1.00	17.90

GC oven program.

Figure 1



m/z	Acceptance Criteria	Value	PASS/FAIL
50	15-40% of mass 95	23.0	PASS
75	30-80% of mass 95	52.4	PASS
95	Base peak	100.0	PASS
96	5-9% of mass 95	6.1	PASS
173	< 2% of mass 174	0.5	PASS
174	> 50% of mass 95	70.0	PASS
175	5-9% of mass 174	6.1	PASS
	> 95% but < 101% of mass 174	97.3	PASS
176	5-9% of mass 95	6.1	PASS
177			PASS

Bromofluorobenzene (BFB) Tuning Criteria after Tune-to-Target.

Calibration standards were prepared at 0.1, 0.5, 1, 2, 5, 10, 20, and 40 ppb with the method preservative. Standards at lower concentrations were required for SIM, typically in the range of 5-100 ppt. A 5 mL sample size was used for the analysis as required by the method.

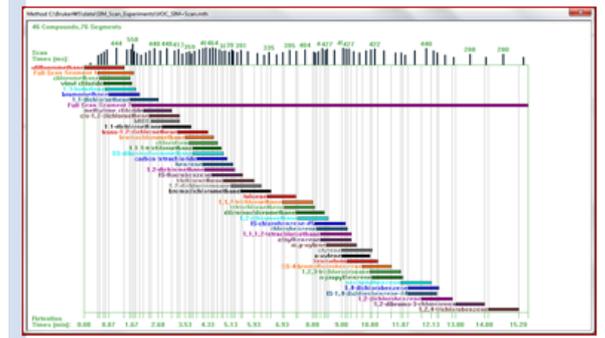
Results

The Scion SQ was tuned to meet method requirements for spectral resolution for Bromofluorobenzene (BFB-Fig. 1) using target ion ratio tuning that is built directly into the software.

Using CBS, a mixed-mode method was created by loading the compounds from a library containing all of the associated SIM ions. A Compound Graph was created, based upon the retention time and retention time window. CBS optimizes the placement of the SIM ions throughout the run for maximum sensitivity and optimal dwell times. As can be seen from Figure 2 below, the full scan segment is also shown in the Compound Graph.

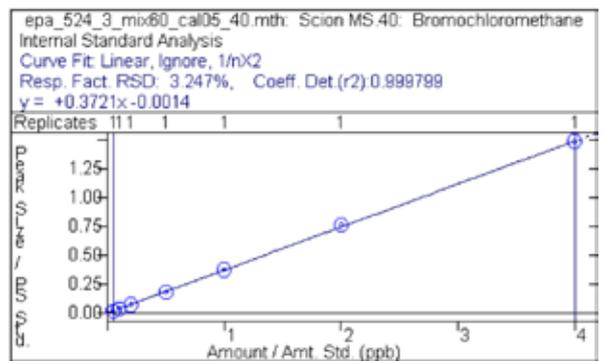
A calibration range from 0.1 to 40 ppb was used for routine quantitative analysis for all target analytes. Average calibration percent RSD and correlation coefficient for Method 524.3 analytes were 7.35% and 0.9991 respectively. Example curve for Bromochloromethane shown in Figure 3. Table 2 has calibration statistics for select compounds in the method.

Figure 2



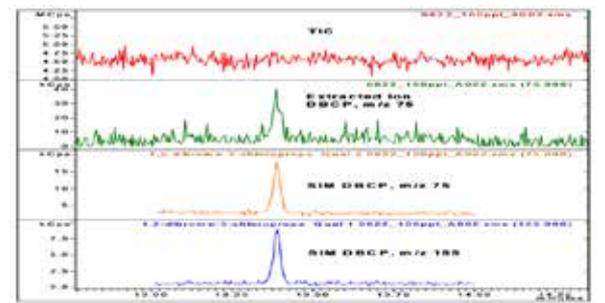
Compound Graph for mixed-mode analysis. Total scan time is represented by a bar graph on the top of the chart.

Figure 3



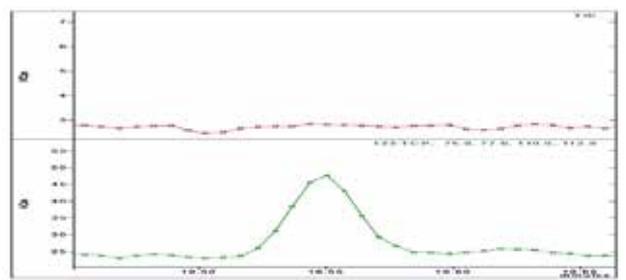
Bromochloromethane calibration curve 0.5-40 ppb full Scan.

Figure 4



SIM analysis during full scan acquisition for DBCP. SIM provides excellent sensitivity for ions m/z 75 and m/z 155 at 100 ppt.

Figure 5



TIC (full scan) for top trace, SIM ions m/z 75, 77, and 110 in bottom trace for 1,2,3-Trichloropropane at 5 ppt.

Table 2

Compound Name	Corr. Coeff.	Avg. RRF	% RSD
Dichlorodifluoromethane	0.9869	0.0733	13.56
Chloromethane	0.9972	0.2829	8.05
Vinyl chloride	0.9986	0.2507	4.81
Bromomethane	0.9968	0.3446	22.04
Chloroethane	0.9982	0.2173	5.38
Trichlorofluoromethane	0.9979	0.2719	6.66
1,1-Dichloroethene	0.9991	0.6014	3.38
Methylene chloride	0.9995	0.3270	1.86
cis-1,2-dichloroethene	0.9996	0.7666	3.49
1,1-Dichloroethane	0.9996	0.6523	2.68
2,2-Dichloropropane	0.9986	0.3364	3.63
trans-1,2-dichloroethene	0.9996	0.9507	2.15
Bromochloromethane	0.9998	0.3645	3.25
Chloroform	0.9999	1.0559	4.64
1,1,1-Trichloroethane	0.9996	0.4772	8.20
Carbon Tetrachloride	0.9994	0.6219	16.29
1,1-Dichloropropene	0.9991	0.5530	5.69
Benzene	0.9995	1.2990	1.68
1,2-Dichloroethane	0.9997	0.7074	2.52
Trichloroethene	0.9996	0.9517	4.30
1,2-Dichloropropane	0.9998	0.5672	2.80
Dibromomethane	0.9999	0.1636	8.64
Bromodichloromethane	0.9993	0.6403	15.27
trans-1,3-dichloropropene	0.9995	0.4606	13.60
Toluene	0.9997	1.3013	2.40
cis-1,3-dichloropropene	0.9993	0.4375	15.05
1,1,2-trichloroethane	0.9999	0.3189	2.74
Tetrachloroethene	0.9997	0.6481	4.38
1,3-dichloropropane	0.9999	0.6297	2.13
Dibromochloromethane	0.9968	0.2336	21.61

Compound Name	Corr. Coeff.	Avg. RRF	% RSD
1,2-Dibromoethane (EDB)	0.9999	0.6577	9.37
Chlorobenzene	0.9999	1.1600	2.51
1,1,1,2-Tetrachloroethane	0.9995	0.6940	10.42
Ethylbenzene	0.9999	2.6085	4.04
m,p-Xylene	0.9993	4.0190	5.29
o-Xylene	0.9999	2.3739	5.24
Styrene	0.9993	1.0651	11.14
Bromoform	0.9945	0.1715	22.75
Isopropylbenzene	0.9993	1.7781	6.58
Bromobenzene	0.9999	1.3909	3.64
1,1,2,2-Tetrachloroethane	0.9997	1.0195	14.87
1,2,3-Trichloropropane	0.9993	1.0271	7.95
trans-1,4-Dichlorobutene	0.9995	0.8606	10.12
n-Propylbenzene	0.9993	2.3916	8.03
2-Chlorotoluene	1.0000	1.6695	6.19
4-Chlorotoluene	1.0000	2.0170	6.16
1,3,5-Trimethylbenzene	0.9999	1.4536	9.45
tert-Butylbenzene	0.9997	1.2789	6.37
1,2,4-Trimethylbenzene	0.9993	1.3999	8.65
sec-Butylbenzene	0.9993	1.7028	9.23
1,3-Dichlorobenzene	1.0000	0.6896	4.52
p-Isopropyltoluene	0.9999	3.1700	5.76
1,4-Dichlorobenzene	0.9999	1.8504	3.67
1,2-Dichlorobenzene	1.0000	1.8145	2.97
n-Butylbenzene	0.9993	3.6870	8.03
1,2-Dibromo-3-chloropropane (DBCP)	0.9951	0.5154	17.88
1,2,4-Trichlorobenzene	0.9998	1.6642	3.14
Hexachlorobutadiene	0.9997	0.4132	6.25
Naphthalene	0.9996	3.0391	6.65
1,2,3-Trichlorobenzene	1.0000	1.6357	3.14

Method detection limits (MDLs) in Tables 3a and 3b were obtained by performing seven replicate injections and using the following equation from Section 9.2.6 of EPA Method 524.3:

$$DL = S \times t(n-1, 1 - \alpha)$$

Where $t(n-1, 1 - \alpha)$ = Student's t value for the 99% confidence level with n-1 degrees of freedom (for seven replicate determinations, the Student's t value is 3.143 at a 99% confidence level), n = number of replicates, and S = standard deviation of replicate analyses.

Table 3a

Parameter	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	mean	sd	Calculated MDL	Target concentration
Dichlorodifluoromethane	0.202	0.203	0.186	0.209	0.201	0.199	0.189	0.198	0.00812	0.026	0.2
Chloromethane	0.175	0.211	0.200	0.202	0.180	0.203	0.182	0.193	0.01995	0.044	0.2
Vinyl chloride	0.201	0.216	0.193	0.206	0.207	0.195	0.180	0.200	0.01166	0.037	0.2
Bromomethane	0.210	0.211	0.253	0.239	0.216	0.215	0.219	0.223	0.01632	0.051	0.2
Chloroethane	0.092	0.073	0.094	0.101	0.090	0.058	0.111	0.088	0.01769	0.056	0.1
Trichlorofluoromethane	0.082	0.119	0.131	0.081	0.110	0.124	0.121	0.110	0.02026	0.084	0.1
1,1-Dichloroethene	0.068	0.078	0.079	0.055	0.073	0.078	0.078	0.073	0.00875	0.028	0.1
Methylene chloride	0.091	0.089	0.096	0.078	0.100	0.096	0.098	0.093	0.00748	0.024	0.1
cis-1,2-dichloroethane	0.074	0.100	0.086	0.090	0.104	0.105	0.103	0.095	0.01166	0.037	0.1
1,1-Dichloroethane	0.081	0.088	0.107	0.094	0.081	0.087	0.102	0.091	0.01008	0.032	0.1
2,2-Dichloropropane	0.089	0.107	0.134	0.081	0.146	0.117	0.099	0.108	0.02748	0.086	0.1
trans-1,2-dichloroethene	0.094	0.155	0.120	0.111	0.125	0.105	0.106	0.117	0.01977	0.062	0.1
Bromochloromethane	0.094	0.091	0.080	0.089	0.091	0.087	0.083	0.085	0.00658	0.027	0.1
Chloroform	0.077	0.110	0.099	0.109	0.103	0.109	0.086	0.099	0.01285	0.040	0.1
1,1,1-Trichloroethane	0.084	0.109	0.109	0.103	0.095	0.112	0.080	0.096	0.01791	0.056	0.1
Carbon Tetrachloride	0.071	0.118	0.100	0.115	0.107	0.110	0.111	0.105	0.01588	0.050	0.1
1,1-Dichloropropene	0.077	0.097	0.082	0.104	0.121	0.106	0.109	0.099	0.01546	0.049	0.1
Benzene	0.078	0.107	0.110	0.086	0.095	0.090	0.094	0.094	0.01127	0.035	0.1
1,2-Dichloroethane	0.103	0.088	0.096	0.102	0.098	0.102	0.096	0.096	0.00524	0.016	0.1
Trichloroethene	0.077	0.107	0.081	0.087	0.087	0.109	0.085	0.090	0.01253	0.039	0.1
1,2-Dichloropropane	0.087	0.091	0.088	0.081	0.114	0.105	0.112	0.094	0.01726	0.054	0.1
Dibromomethane	0.093	0.096	0.081	0.102	0.112	0.104	0.110	0.100	0.01072	0.034	0.1
Bromodichloromethane	0.071	0.113	0.103	0.088	0.105	0.105	0.091	0.097	0.01421	0.045	0.1
trans-1,3-dichloropropene	0.076	0.080	0.079	0.086	0.073	0.089	0.080	0.078	0.00709	0.022	0.1
Toluene	0.087	0.094	0.093	0.095	0.102	0.098	0.091	0.091	0.01136	0.036	0.1
cis-1,3-dichloropropene	0.090	0.096	0.104	0.082	0.094	0.101	0.100	0.095	0.0075	0.024	0.1
1,1,2-Trichloroethane	0.084	0.079	0.093	0.097	0.086	0.123	0.108	0.093	0.01892	0.059	0.1
Tetrachloroethene	0.088	0.112	0.098	0.093	0.127	0.127	0.119	0.106	0.02146	0.087	0.1
1,3-dichloropropane	0.082	0.078	0.097	0.110	0.097	0.087	0.093	0.092	0.01077	0.034	0.1
Dibromochloromethane	0.111	0.104	0.107	0.110	0.125	0.109	0.105	0.110	0.00703	0.022	0.1

Calibration statistics for select VOCs by EPA 524.3 on Scion SQ.

Table 3a

Parameter	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	mean	sd	Calculated MDL	Target concentration
1,2-Dibromoethane (EDB)	0.103	0.106	0.121	0.100	0.092	0.092	0.110	0.103	0.01026	0.032	0.1
Chlorobenzene	0.090	0.109	0.117	0.113	0.106	0.112	0.099	0.107	0.00929	0.029	0.1
1,1,1,2-Tetrachloroethane	0.094	0.117	0.124	0.125	0.120	0.113	0.104	0.114	0.01131	0.036	0.1
Ethylbenzene	0.066	0.094	0.106	0.097	0.099	0.097	0.086	0.092	0.01298	0.041	0.1
m,p-Xylene	0.070	0.110	0.102	0.105	0.118	0.109	0.100	0.102	0.01531	0.048	0.1
o-Xylene	0.100	0.113	0.125	0.121	0.103	0.104	0.108	0.111	0.0095	0.030	0.1
Styrene	0.083	0.090	0.093	0.083	0.101	0.101	0.069	0.089	0.01137	0.036	0.1
Bromoform	0.091	0.099	0.091	0.086	0.080	0.074	0.093	0.088	0.00844	0.027	0.1
Isopropylbenzene	0.067	0.113	0.114	0.098	0.104	0.112	0.098	0.101	0.01642	0.052	0.1
Bromobenzene	0.113	0.109	0.099	0.099	0.117	0.124	0.115	0.111	0.00928	0.029	0.1
1,1,2,2-Tetrachloroethane	0.117	0.106	0.108	0.099	0.120	0.102	0.084	0.105	0.01201	0.038	0.1
1,2,3-Trichloropropane	0.085	0.081	0.068	0.075	0.081	0.078	0.055	0.074	0.01016	0.032	0.1
trans-1,2-Dichloroethane	0.093	0.101	0.094	0.096	0.111	0.106	0.081	0.097	0.00978	0.031	0.1
n-Propylbenzene	0.087	0.114	0.107	0.103	0.112	0.124	0.106	0.108	0.01139	0.036	0.1
2-Chlorotoluene	0.066	0.089	0.090	0.087	0.075	0.093	0.076	0.079	0.01121	0.035	0.1
4-Chlorotoluene	0.073	0.104	0.084	0.081	0.081	0.102	0.093	0.088	0.01166	0.037	0.1
1,3,5-Trimethylbenzene	0.088	0.095	0.104	0.099	0.090	0.120	0.104	0.100	0.01082	0.034	0.1
tert-Butylbenzene	0.083	0.114	0.109	0.116	0.116	0.128	0.111	0.111	0.01376	0.043	0.1
1,2,4-Trimethylbenzene	0.082	0.099	0.096	0.096	0.108	0.100	0.086	0.095	0.00877	0.028	0.1
sec-Butylbenzene	0.062	0.130	0.104	0.110	0.111	0.115	0.093	0.104	0.02147	0.067	0.1
1,3-Dichlorobenzene	0.084	0.122	0.111	0.104	0.098	0.105	0.084	0.101	0.01386	0.044	0.1
p-Isopropyltoluene	0.080	0.143	0.128	0.121	0.108	0.136	0.125	0.120	0.02089	0.066	0.1
1,4-Dichlorobenzene	0.093	0.107	0.085	0.097	0.097	0.094	0.090	0.095	0.00685	0.022	0.1
1,2-Dichlorobenzene	0.091	0.109	0.124	0.113	0.123	0.115	0.093	0.110	0.01323	0.042	0.1
n-Butylbenzene	0.081	0.107	0.119	0.090	0.129	0.124	0.104	0.108	0.01774	0.056	0.1
1,2-Dibromo-3-chloropropane (DBCP)	0.151	0.132	0.107	0.177	0.113	0.118	0.170	0.138	0.02809	0.088	0.1
1,2,4-Trichlorobenzene	0.069	0.072	0.118	0.105	0.105	0.099	0.106	0.096	0.01853	0.058	0.1
Hexachlorobutadiene	0.078	0.108	0.077	0.095	0.095	0.127	0.144	0.103	0.02487	0.078	0.1
Naphthalene	0.083	0.092	0.108	0.098	0.117	0.097	0.096	0.099	0.01098	0.035	0.1
1,2,3-Trichlorobenzene	0.085	0.082	0.099	0.083	0.072	0.099	0.090	0.087	0.00972	0.031	0.1

1,2-Dibromoethane (EDB), 1,2-Dibromo-3-chloropropane (DBCP), and 1,2,3-trichloropropane were quantitated in SIM mode. Figure 4 shows a DBCP analysis for a 100 ppt standard. Extraction of select ions from the TIC result in more noise and higher detection limits when compared to the SIM trace.

1,2,3-Trichloropropane was easily detected at 5 ppt in SIM during mixed-mode acquisition (Figure 5). 1,2,3-Trichloropropane (TCP) requires very low detection limits because it is suspected to be a human carcinogen and is used widely as a chemical intermediate in polymer production and extraction solvents.

Reporting of the samples and quality control is the final step that must be completed by the lab. Scion offers EnviroPro™, a Microsoft Access database that will generate all of the required reports for EPA Method 524, as well as several other methods. Example reports are tune criteria, method detection limit calculations, initial calibration reports, and continuing calibration checks. There are several graphic options available for printing chromatograms and target compounds, as well as unknown peaks (non-target analytes).

Conclusions

The Tekmar Atomx purge-and-trap sample concentrator with the Scion SQ is a total solution for EPA VOC methods. Method set-up for full scan and scan/SIM experiments is easy with unique CBS software. Performance of the system is excellent and exceeds the requirements of EPA method 524.3.

Acknowledgements

Special thanks to Teledyne Tekmar for providing the Atomx™ automated purge-and-trap sample concentrator.

Figure 6



The EnviroPro™ software package for environmental methods in Microsoft Access 2010.

References:

[1] <http://www.epa.gov/ogwdw000/methods/pdfs/methods/met524-3.pdf>

Authors:

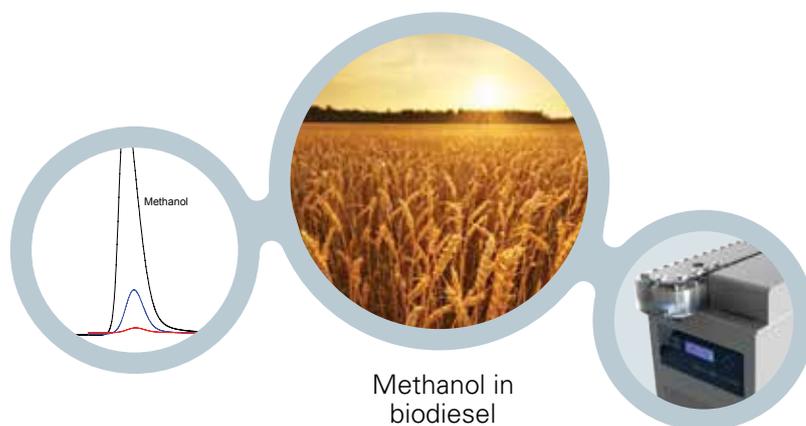
Ed George

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Application Notes #283027

Determination of Methanol Content in Biodiesel using Gas Chromatograph with Headspace Sampling According to EN-14110

The popularity and interest in biodiesel is significantly growing in many areas of the world and has become a commonly sought after alternative fuel source for use with diesel engines. Biodiesel is produced from vegetable oils or animal fats via transesterification using methanol to yield Fatty Acid Methyl Esters (FAME) and glycerine. The yield, pure FAME (once the glycerine and the residual methanol has been recovered/removed) is called B-100. In order for biodiesel to be used as a motor fuel or blended with petroleum diesel, it must conform to standard specifications (ASTM D 6751 or EN-14214). There are GC methods in use today to determine whether biodiesel conforms to the standard specifications. One of these methods, EN-14110, is used to determine the methanol content. EN-14110 is applicable for a concentration range from 0.01% (m/m) to 0.5% (m/m) methanol*.

Instrumentation

436-GC

- Injector: Split / splitless 1177 S/SL, full EFC control
- Detector: FID, full EFC control

Headspace Sampler

- SHS-40 Headspace Analyzer

GC control and data handling

- compassCDS software

Materials and Reagents

Column

- GC Care Column: BR-1 fs, 30m x 0.32mm, df= 3.0 μ m. PN BR89816
- Fatty Acid Methyl Ester mixture (FAME) with a methanol content of <0.001%

*The method is not applicable to mixtures of FAME which contain other low boiling components.

Sample Preparation

Calibration solutions

- Solution A: 0.5% (m/m) methanol in FAME
- Solution B: 0.1% (m/m) methanol in FAME
- Solution C: 0.01% (m/m) methanol in FAME

A 1mL aliquot was accurately weighed, and transferred into a 20mL vial and then immediately capped.

Sample

A 1mL sample was accurately weighed then transferred into a 20mL vial and immediately capped.

Conditions

GC conditions

- Injector: 250°C
- Split ratio: 50:1
- Detector: 275°C, FID
- Oven: 80°C (0.5 min. isothermal)
@ 20°C/min. to 160°C (2 min)
- Carrier gas: 2.0mL/min. const. flow, Helium

SHS-40

- Sample loop: 1mL
- Vial/heating: 80°C
- Equilibrium time: 45 min.

Results and Discussion

All three calibration solutions were analyzed twice and a calibration curve was obtained. See Figure 1 for an overlay of the methanol peaks of the different calibration solutions. The calibration curve (Figure 2) shows excellent correlation with the method.

Figure 1. Overlay traces of calibration solutions.

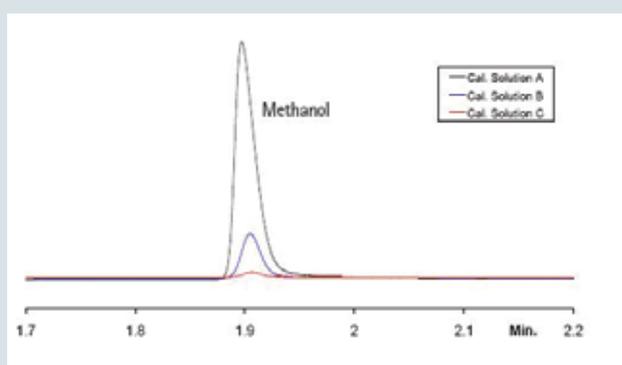
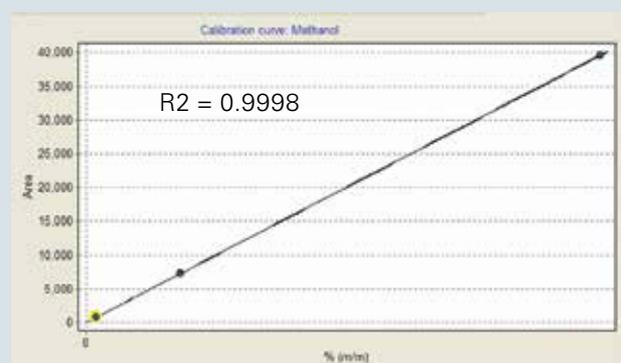


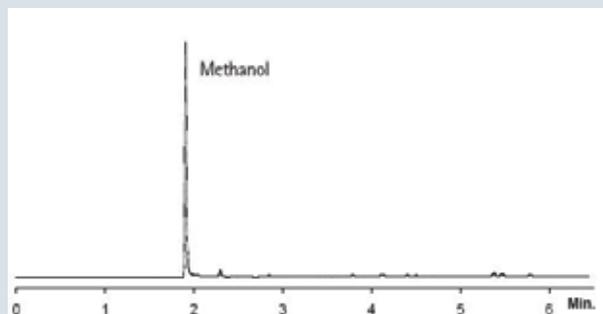
Figure 2. Calibration curve.



The correlation coefficient should be > 0.95 . In this case the correlation coefficient was determined to be 0.9998.

A typical chromatogram of a biodiesel sample is shown in Figure 3.

Figure 3. Typical headspace chromatogram of biodiesel.

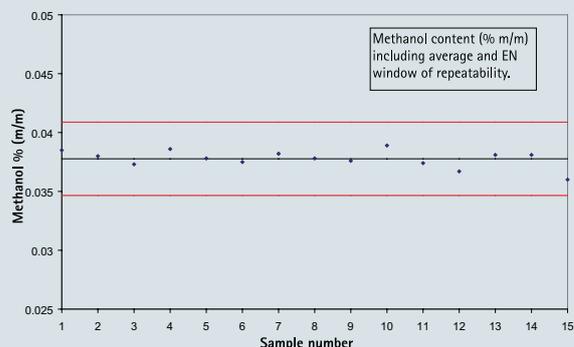


Since biodiesel generally does not contain volatile components, other than methanol, identification and quantification is quite straightforward.

Figure 4. Repeatability figures.

	Methanol (mass %)
N	15
Average	0.038
St. dev.	0.0007
RSD (%)	1.96

Figure 5. Repeatability values are within the specification boundaries established in EN-14214 as indicated by the red lines in the chart.



The methanol content of the biodiesel was 0.038% (m/m) thus meeting the specifications set in EN-14214, (methanol content <0.2%). Furthermore the repeatability figures indicated that the system was properly optimized for the analysis as seen in Figure 5, where the analyses trend line is well within the repeatability “window” set forth in the EN-14110 method. In Figure 5 this is visualized by adding the average line and the window of repeatability set in the EN-14110 method.

Conclusion

The GC Headspace system (Scion 436-GC Gas Chromatograph and a SHS-40 Headspace Sampler) was shown to be well suited for the determination of methanol content in biodiesel according to specifications outlined in EN-14110, and the biodiesel tested in this application note meets the specifications on methanol content set forth in EN-14214.

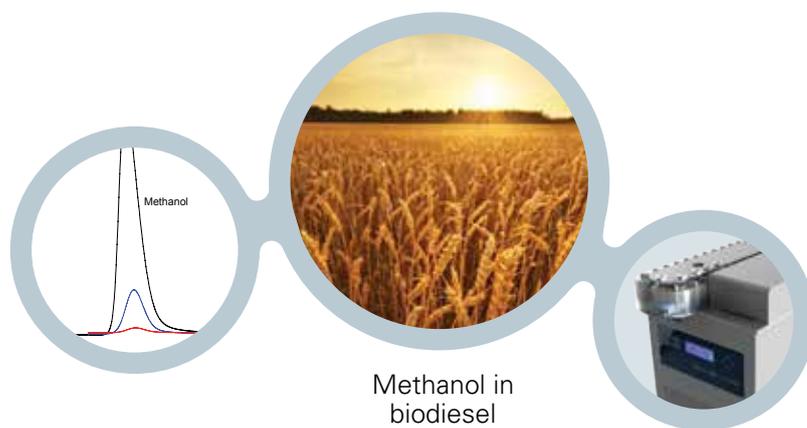
References

- EN-14110 Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of methanol content.
- EN-14214 Automotive fuels – Fatty Acids Methyl Esters (FAME) for diesel engines – requirements and test methods.

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Application Notes #283027

Determination of Methanol Content in Biodiesel using Gas Chromatograph with Headspace Sampling According to EN-14110

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Instrumentation

436-GC

- Injector: Split / splitless 1177 S/SL, full EFC control
- Detector: FID, full EFC control

Headspace Sampler

- SHS-40 Headspace Analyzer

GC control and data handling

- compassCDS software

Materials and Reagents

Column

- GC Care Column: BR-1 fs, 30m x 0.32mm, df= 3.0 μ m. PN BR89816
- Fatty Acid Methyl Ester mixture (FAME) with a methanol content of <0.001%

*The method is not applicable to mixtures of FAME which contain other low boiling components.

Sample Preparation

Calibration solutions

- Solution A: 0.5% (m/m) methanol in FAME
- Solution B: 0.1% (m/m) methanol in FAME
- Solution C: 0.01% (m/m) methanol in FAME

A 1mL aliquot was accurately weighed, and transferred into a 20mL vial and then immediately capped.

Sample

A 1mL sample was accurately weighed then transferred into a 20mL vial and immediately capped.

Conditions

GC conditions

- Injector: 250°C
- Split ratio: 50:1
- Detector: 275°C, FID
- Oven: 80°C (0.5 min. isothermal)
@ 20°C/min. to 160°C (2 min)
- Carrier gas: 2.0mL/min. const. flow, Helium

SHS-40

- Sample loop: 1mL
- Vial/heating: 80°C
- Equilibrium time: 45 min.

Results and Discussion

All three calibration solutions were analyzed twice and a calibration curve was obtained. See Figure 1 for an overlay of the methanol peaks of the different calibration solutions. The calibration curve (Figure 2) shows excellent correlation with the method.

Figure 1. Overlay traces of calibration solutions.

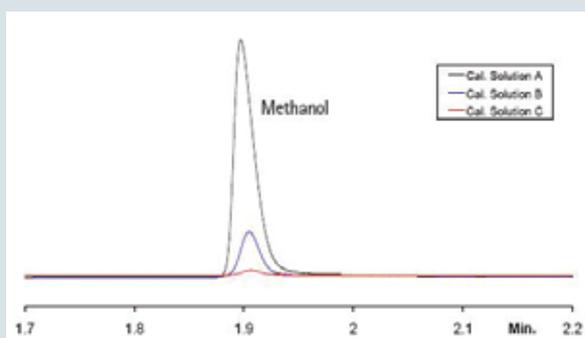
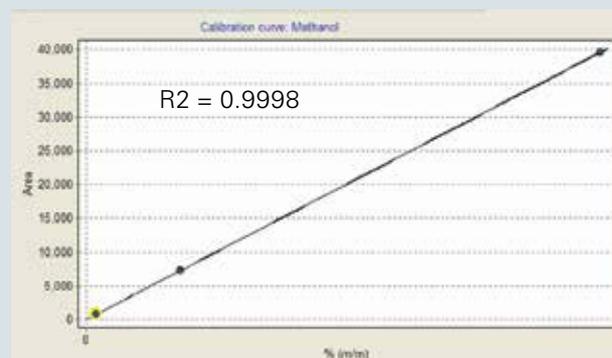


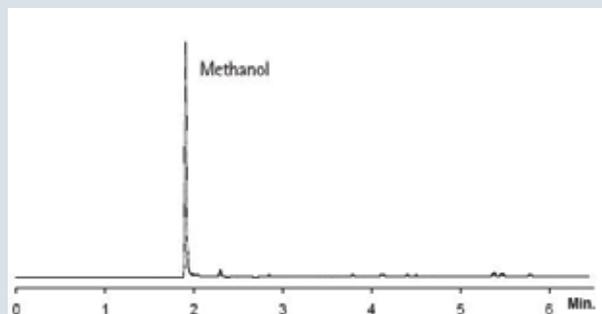
Figure 2. Calibration curve.



The correlation coefficient should be > 0.95 . In this case the correlation coefficient was determined to be 0.9998.

A typical chromatogram of a biodiesel sample is shown in Figure 3.

Figure 3. Typical headspace chromatogram of biodiesel.

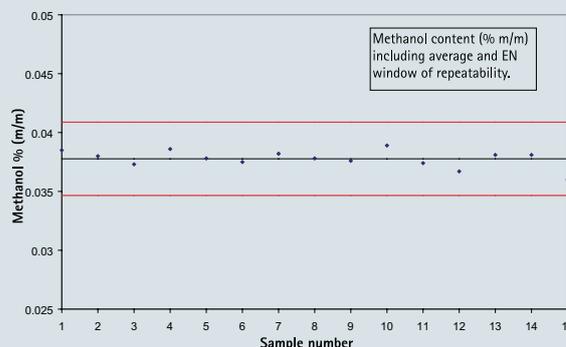


Since biodiesel generally does not contain volatile components, other than methanol, identification and quantification is quite straightforward.

Figure 4. Repeatability figures.

	Methanol (mass %)
N	15
Average	0.038
St. dev.	0.0007
RSD (%)	1.96

Figure 5. Repeatability values are within the specification boundaries established in EN-14214 as indicated by the red lines in the chart.



The methanol content of the biodiesel was 0.038% (m/m) thus meeting the specifications set in EN-14214, (methanol content <0.2%). Furthermore the repeatability figures indicated that the system was properly optimized for the analysis as seen in Figure 5, where the analyses trend line is well within the repeatability “window” set forth in the EN-14110 method. In Figure 5 this is visualized by adding the average line and the window of repeatability set in the EN-14110 method.

Conclusion

The GC Headspace system (Scion 436-GC Gas Chromatograph and a SHS-40 Headspace Sampler) was shown to be well suited for the determination of methanol content in biodiesel according to specifications outlined in EN-14110, and the biodiesel tested in this application note meets the specifications on methanol content set forth in EN-14214.

References

EN-14110 Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of methanol content.

EN-14214 Automotive fuels – Fatty Acids Methyl Esters (FAME) for diesel engines – requirements and test methods.

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USP <467> residual solvent analysis

Application Notes #283029

The Analysis of Residual Solvents By Headspace Sampling and GC According to USP <467>

For pharmacopeial purposes, residual solvents in pharmaceuticals are defined as organic volatile chemicals that are consumed or produced in the manufacture of drug products.

Appropriate selection of the solvent for the synthesis of a drug substance may enhance the yield, or determine characteristics such as crystal form, purity and solubility. Therefore, the solvent may sometimes be a critical element in the synthetic process. Residual solvents are not completely removed by practical manufacturing techniques. The United States Pharmacopeia (USP) <467> specifies the gas chromatographic conditions¹ for the analysis of these organic volatile impurities (OVI).

This application note describes the gas chromatographic analysis of these residual solvents combined with the headspace sampling and injection technique.

Instrumentation

456-GC Gas Chromatograph

- Injector: Split/Splitless, 1177 S/SL, full EFC control
- Detector: FID, full EFC control

Headspace Sampler

- SHS-40 Headspace Analyzer

GC control and data handling

- compassCDS software

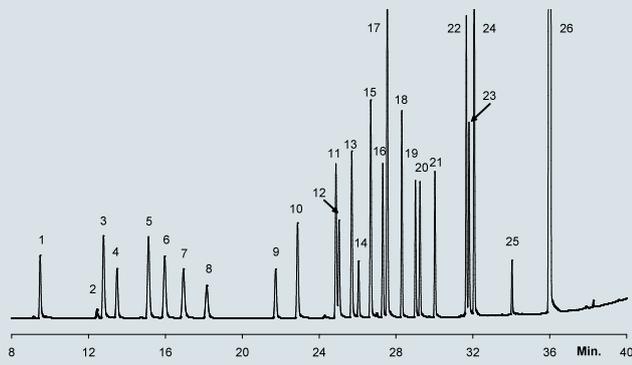
Sample Preparation

Solvents ranging in concentration from 100 to 500 ppm (v/v) from a stock solution were added to de-ionized water and serial dilutions prepared from the resulting mixture were used to calibrate the system.

Table 1. Peak Identification.

#	Component	#	Component
1	Methanol	14	Trichloromethane
2	Pentane	15	Cyclohexane
3	Ethanol	16	Benzene
4	Diethylether	17	2-Methylpropanol
5	Acetone	18	Heptane
6	2-Propanol	19	Trichloroethylene
7	Acetonitrile	20	1-Butanol
8	Dichloromethane	21	1,4-Dioxane
9	Hexane	22	Methylisobutylketone (MIBK)
10	1-Propanol	23	Pyridine
11	Methylethylketone (MEK)	24	Toluene
12	Ethylacetate	25	N,N-Dimethylformamide
13	Tetrahydrofuran (THF)	26	Dimethylsulfoxide (DMSO)

Figure 1. Chromatogram of a water sample spiked with "residual solvents".



Instrument Operational Conditions

SHS-40 Automated Headspace Sampler

Type: Sample loop

Oven: 80°C

Valve: 180°C

Tube: 180°C

Precond. time: 60 min. shaking

Run time: 45 min.

450-GC Gas Chromatograph

Column: GC Care Column: BR-624ms fs,
60m x 0.32mm, df= 1.80 µm
PN: BR86128

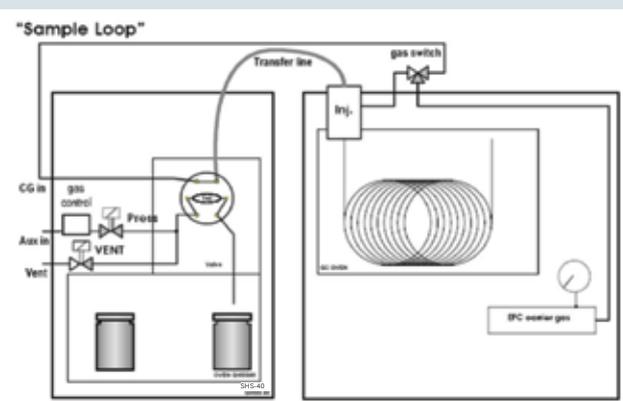
Injector: 200°C, Split ratio 1:10

Detector: FID, 270°C

Carrier Gas: He, 1mL/min. constant flow

Oven: 40°C, 20 min. isothermal @
10°C/min. to 250°C

Figure 2. Connection scheme of the SHS-40 and the 450-GC.



Results and Discussion

The system is validated using five independently spiked water samples at different concentrations using the 26 component standard/stock solution used to prepare the calibration standard. A typical chromatogram of the 26 components is shown in Figure 1. The hardware configuration is shown schematically in Figure 2.

Concentrations range between 100 and 500 ppm per component. Calibration curves were then generated for each individual component. Figures 3 – 6 below show several selected solvents with different polarity that represent examples of the different safety classes stated in the method.

Figure 3. Calibration curve of Pentane.

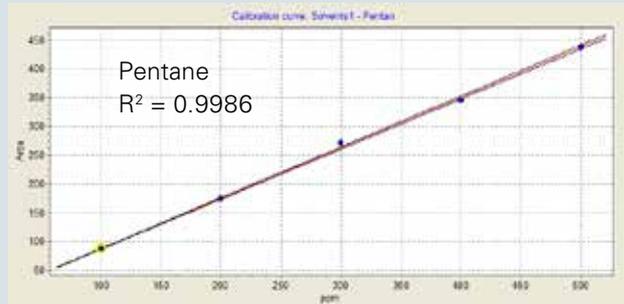


Figure 4. Calibration curve of Benzene.

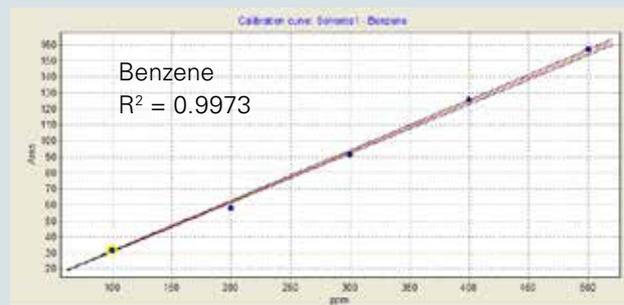


Figure 5. Calibration curve of Dichloromethane.

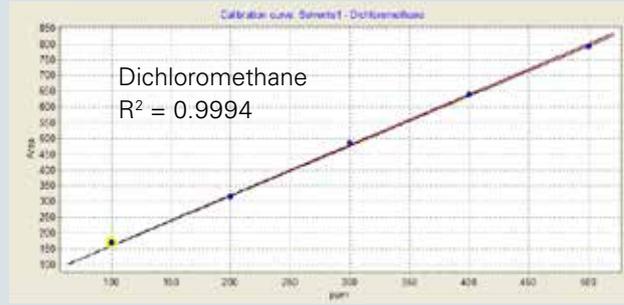
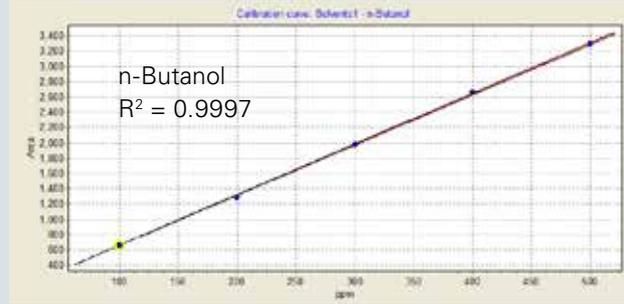


Figure 6. Calibration curve of n-Butanol.



The calibration curves shown above clearly indicate that the system is well suited for the analysis of the different solvent types.

Table 2. Correlation Coefficient per compound.

Safety Class 1	
Compound	Correlation coefficient
Benzene	0.9973
Safety Class 2	
Compound	Correlation coefficient
1,4-Dioxane	0.9903
Acetonitrile	0.9887
Cyclohexane	0.9996
Dichloromethane	0.9994
Methanol	0.9979
n-Hexane	0.9982
Pyridine	0.9954
Toluene	0.9997
Trichloroethylene	0.9957
Trichloromethane	0.9996
Safety Class 3	
Compound	Correlation coefficient
1-Propanol	0.9905
2-Methylpropanol	0.9997
2-Propanol	0.9867
Acetone	0.9870
Diethylether	0.9995
Ethanol	0.9916
Ethylacetate	0.9970
Methylethylketone (MEK)	0.9989
Methylisobutylketone (MIBK)	0.9992
n-Butanol	0.9997
n-Heptane	0.9996
Pentane	0.9986
Tetrahydrofuran (THF)	0.9766

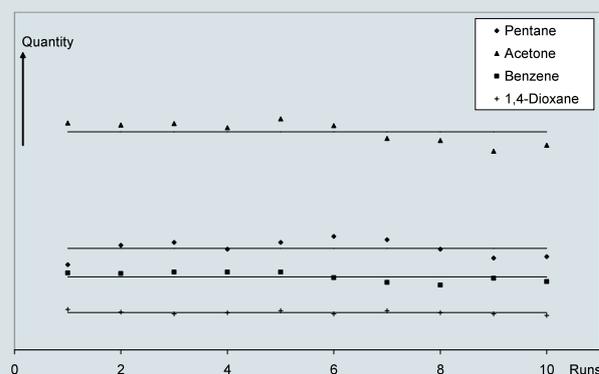
Correlation coefficients are close to 1.0 indicating a linear relationship between component concentration in the sample and detector signal. This indicates that the full system including headspace sampling, injection, separation and detection worked correctly.

Excellent correlation coefficients were achieved over a wide range of polarity including a-polar solvents like pentane, polar solvents like n-butanol, chlorinated solvents like dichloromethane and aromatic solvents like toluene.

However, further analysis improvements can be achieved if a salt like sodium sulfate is added to the samples. This ensures super saturation and forces the maximum amount of analyte into the headspace.

To further validate the system analysis to analysis, repeatability was tested. Some of the data is summarized in Chart 1. As before, the selected components are indicative of the different chemical classes and safety classes outlined in the method. The repeatability data shown in Chart 1 is excellent. Relative standard deviation is typically <5% for all components.

Chart 1. Repeatability data for selected components at 100 ppm level.



Conclusion

The combination of the Scion 456-GC Gas Chromatograph and Scion SHS-40 headspace sampler is well suited for the determination of residual solvents according to the United States Pharmacopeia (USP) <467>.

All components listed in the method show linear response in the analysis range and excellent system repeatability.

References

1. Organic Volatile Impurities / Residual Solvents <467>, United States Pharmacopeia USP 32-NF 18.

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Residual Solvent
Analysis

Application Notes #283030

Determination of Class I USP Residual Solvents and TICs in Dietary Supplements and Pharmaceutical Products by GC/MS

Residual solvents in pharmaceuticals are defined as organic volatile chemicals that are consumed or produced in the manufacture of drug products. It is known that these residual solvents are not completely removed by practical manufacturing techniques. The United States Pharmacopeia (USP) <467> specifies the gas chromatographic (GC) conditions for the analysis of these organic volatile impurities. Static headspace analysis coupled with gas chromatography mass spectrometry (GC/MS) is the ideal technique for the analysis, because target and unknown compounds can be analysed simultaneously with complete confidence in the identification and quantification of the results. The new Scion automated static headspace auto sampler, the SHS-40, is combined with the SCION SQ mass spectrometer for the analysis of Class I residual solvents in pharmaceutical and dietary supplement products. Non-target compounds are also determined by automated library search.

Introduction

Class 1 residual solvents should not be employed in the manufacture of drug substances, excipients, and drug products because of the unacceptable toxicities or deleterious environmental effects of these residual solvents (1). Table 1 provides a list of the compounds and the concentration limits, along with the general concern for each.

Table 1. Class I Residual Solvents.

Compound	Limit (ppm)	Hazard
Benzene	2	Carcinogen
Carbon Tetrachloride	4	Toxic Env Hazard
1,2-Dichloroethane	5	Toxic Env Hazard
1,1-Dichloroethene	8	Toxic Env Hazard
1,1,1-Trichloroethane	1500	Toxic Env Hazard

The sample is typically analysed by headspace coupled with gas chromatography (non-MS detectors such as FID). The SCION SQ can be operated in a single ion monitoring (SIM) mode and SCAN mode simultaneously, which provides very low limits of quantitation for the target residuals along with identification of other compounds that may be present in the sample.

A unique feature of SCION is the ability to set up methods rapidly using Compound Based Scanning (CBS). CBS makes use of libraries that contain all scan and retention time information for a given set of compounds that are loaded directly into an acquisition method and data handling compound table in one easy step. Figure 1 shows the Class I Residual Solvents and a full scan segment in a library that are easily loaded into the method. Figure 2 is a graph of the compound acquisition windows that have been automatically created by CBS for optimal sensitivity and quantitative analysis.

Figure 1. A CBS Library of USP Class I Residual Solvents.

Name	CAS No.	MW	Boiling Point	Density	Refractive Index
Acetone	67-64-1	58.08	56.0	0.791	1.359
Diethyl ether	109-87-1	74.12	34.6	0.714	1.311
Methanol	67-58-0	32.04	64.7	0.791	1.329
Ethanol	64-17-5	46.07	78.4	0.789	1.362
Isopropanol	67-73-0	60.10	82.6	0.786	1.377
n-Butanol	71-36-3	74.12	117.7	0.810	1.399
tert-Butanol	75-97-5	74.12	82.5	0.781	1.382
2-Propanol	67-63-0	60.10	82.6	0.786	1.377
1-Propanol	71-23-8	60.10	97.2	0.804	1.393
1,2-Dichloroethane	107-06-2	98.96	83.7	1.250	1.424
1,1-Dichloroethane	75-34-8	98.96	47.5	1.250	1.424
1,1,1-Trichloroethane	70-13-8	131.39	34.5	1.412	1.461
1,1,2-Trichloroethane	70-12-7	131.39	54.3	1.412	1.461
1,2-Dichlorobenzene	90-43-3	147.03	180.4	1.310	1.505
1,4-Dichlorobenzene	95-50-1	147.03	176.2	1.310	1.505
1,3-Dichlorobenzene	95-49-7	147.03	181.0	1.310	1.505
1,2-Dichlorobenzene	95-49-7	147.03	181.0	1.310	1.505
1,1-Dichloroethane	75-34-8	98.96	47.5	1.250	1.424
1,1,1-Trichloroethane	70-13-8	131.39	34.5	1.412	1.461
1,1,2-Trichloroethane	70-12-7	131.39	54.3	1.412	1.461
1,2-Dichlorobenzene	90-43-3	147.03	180.4	1.310	1.505
1,4-Dichlorobenzene	95-50-1	147.03	176.2	1.310	1.505
1,3-Dichlorobenzene	95-49-7	147.03	181.0	1.310	1.505
1,2-Dichlorobenzene	95-49-7	147.03	181.0	1.310	1.505

Figure 2. Optimized Compound acquisition summary with simultaneous Full Scan and SIMs.



Experimental

A vitamin dietary supplement, a common pain reliever and an allergy medication were prepared for static headspace analysis by dissolving 250mg of the finished product in 25mL of water. Five milliliters (5mL) was transferred to a 20mL headspace vial with a screw-cap teflon-faced septum. Two grams of sodium sulfate anhydrous was added to each vial, along with 1.0mL deionized reagent water.

Standards containing the Class I USP residual solvents were prepared such that all compounds were at the same final concentrations of 0.1, 1.0, and 10ppm. The 0.1 and 1.0ppm standards are well below the individual compound required concentration limits shown in Table 1. A volume of 1.0mL was transferred to 5mL deionized reagent water, along with 2g sodium sulfate anhydrous.

The samples were placed on the Scion SHS-40 headspace auto sampler, with the conditions listed in Table 2.

Figure 3. Scion SHS-40 Headspace Autosampler with SCION GC/MS.



Table 2. SHS-40 Sampling Conditions.

Parameter	Set Point
Oven Temp	85°C
Valve/Loop Temp	160°C
Transfer line Temp	125°C
Pressure	500psi
Loop Volume	1mL
PC (incubation) Time	30 min
GC/MS Run Time	20 min
Shake option	ON

The SCION GC/MS column, oven program, and injector conditions:

Column: BR-624ms, 20M x 0.18mm x 1.0um
 Injector: BR-1079, PTV injector with 3.4mm single goose-neck open split liner set at 200°C
 Injector split ratio: 1:20
 Column flow: 1mL/min.
 Oven program: Initial 35°C hold 2 min; program to 170°C at 10°C/min; hold 0; program to 250°C at 50°C/min, hold 1 min, (total run time 17.9 min.)

Table 3. Synchronous SIM/SCAN parameters set up using CBS for Class 1 USP Residual Solvents.

Compound Name	Retention Time (RT)	RT Window	Scan Mode, Ions monitored	Dwell Time (ms)
1,1-Dichloroethene	2.10	1.0	SIM, 61, 96, 98	49
1,1,1-Trichloroethane	4.23	1.0	SIM, 97, 99	49
Carbon tetrachloride	4.38	1.0	SIM, 117, 119	49
Benzene	4.59	1.0	SIM, 77, 78	49
1,2-Dichloroethane	4.67	1.0	SIM, 62, 64	49
Full Scan	NA	2.0-17.9	Full (m/z 35-300)	300

Results

The instrument in the SIM/Scan mode provided excellent sensitivity as seen in Figure 4. All of the compounds are easily detected at the 0.1ppm level, with excellent peak shape due to the 1:20 split. Since qualifier ions are also monitored, unambiguous results are obtained. The correlation coefficient (r(2)) for all curves were greater than 0.999. An example calibration curve for 1,1,1-trichloroethane is shown in Figure 5.

None of the Class 1 Residual Solvents were detected in any of the test samples. However, other compounds showed up in the full scan data. The Scion MSWS 8 software is capable of automated peak detection and library searching of unknown peaks. The tentatively identified compounds (TICs) can be reviewed to identify potential problems that may be occurring in the manufacturing process. The pain reliever had a good match for several compounds (reverse fit match greater than 800 for top 10 hits). The compound was not detected in the blank or control samples (Figure 6).

Figure 4. Measurement of USP Class I Residual Solvents at 0.1 ppm.

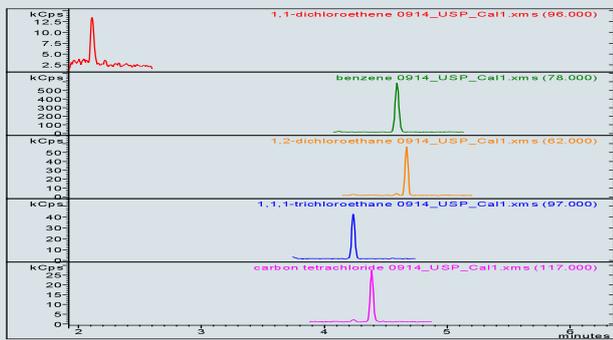


Figure 6. TIC detected in pain reliever.

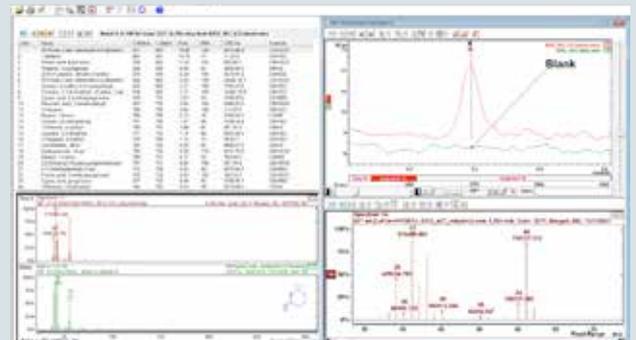
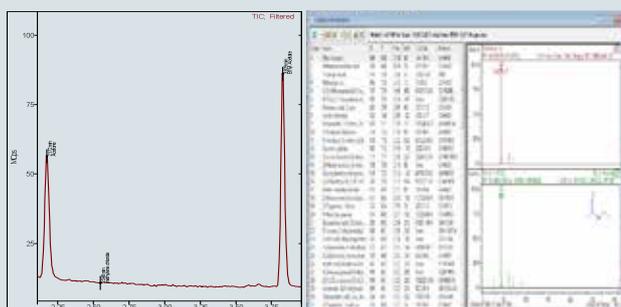


Figure 5. Calibration curve, from 0.1 to 10 ppm of 1,1,1-trichloroethane.



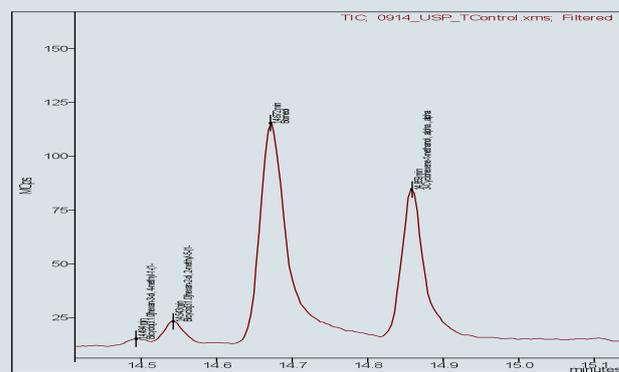
These are part of a list that includes solvents that are not known as human health hazards at levels normally accepted in pharmaceuticals. The allergy medication had relatively high concentrations of ethyl acetate and acetone detected in the sample as indicated in Figure 7. These are Class 3 Residual solvents. However, there are no long-term toxicity or carcinogenicity studies for many of the residual solvents in Class 3. Available data indicate that they are less toxic in acute or short-term studies and negative in genotoxicity studies (2).

Figure 7. Residual Class 3 solvents, acetone and ethyl acetate, found as TICs in the allergy medication.



The multi-vitamin (also advertised as having weight-control benefits) had some peaks eluting near the end of the chromatographic run. Borneol and other terpenes were detected in the sample.

Figure 8. Multi-vitamin/weight loss control dietary supplement with TIC borneol detection.



Conclusion

The Scion SHS-40 headspace auto sampler coupled with the SCION GC/MS provided excellent detection limits and quantitative data for the Class I USP Residual Solvents. Compound Based Scanning (CBS) makes it easy to set up optimized acquisition and data handling methods directly from compound libraries with a single click. None of the target residual solvents were detected in the three products studied, however other TICs were found by examining the SCAN data. This additional data can alert the quality control manager of potential contamination in the manufacturing process, or be used to evaluate other non-regulated compounds present in the product.

References

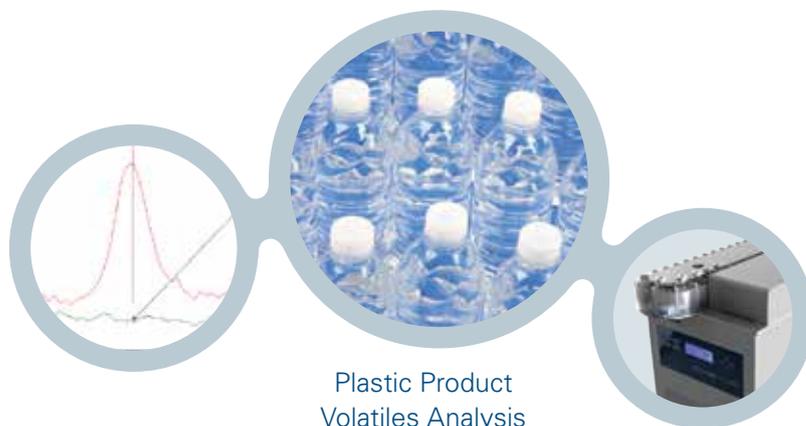
(1) and (2): Chemical Tests: General Chapter (USP) <467> Residual Solvents, Organic Volatile Impurities, July 2007

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Plastic Product
Volatiles Analysis

Application Notes #283078

Direct Analysis of Finished Plastic Products by Heated Head Space and GC/MS

A sample of raw or finished material is directly analysed for volatile components using an automated head space auto sampler. Little or no sample preparation is required, making it an ideal technique for high throughput quality control testing in manufacturing. When operated in a synchronous SIM/Scan mode, the SCION SQ single quadrupole mass spectrometer provides quantitative data, along with tentatively identified compounds (non-target compounds).

Introduction

Material testing for outgassing volatile organic chemicals is required in many industries to ensure consumers are not being exposed to harmful contaminants. This is especially important when a material such as a plastic is exposed to excess heat with little or no ventilation. A good example would be plastic materials in a car such as dash boards, which are exposed to very high temperatures in direct sunlight. Volatile chemicals are also common in carpeting and polyvinyl chloride (PVC) piping, and may pose a risk in poorly ventilated homes.

Chlorinated solvents that outgas from materials are generally considered harmful, because they are classified as being carcinogenic or environmental hazards. Other compounds detected may be uncharacterized with little known about

their toxicity. Phthalate esters, for example, have been linked to endocrine disruption in some animal species, and regulations are emerging to control these substances in plastic bottles (1).

Experimental

Samples of polypropylene from a car manufacturer and PVC pipe were cut into small pieces and placed directly into 20mL head space vials. About 500mg-1g was added. The Scion SHS-40 Auto sampler conditions used are cited in Table 1.

Table 1. SHS-40 Sampling Conditions.

Parameter	Set Point
Oven Temp	70°C
Valve/Loop Temp	160°C
Transfer line Temp	125°C
Pressure	500psi
Loop Volume	1mL
PC (incubation) Time	30 min.
GC/MS Run Time	20 min.
Shake option	ON

Figure 3. Scion SHS-40 Headspace Autosampler with SCION GC/MS.



The SCION GC/MS column, oven program, and injector conditions:

Column: BR-624ms, 20M x 0.18mm x 1.0um
 Injector: BR-1079, PTV injector with 3.4mm single goose-neck open split liner set at 200°C
 Injector split ratio: 1:20
 Column flow: 1mL/min
 Oven program: Initial 35°C hold 2 min; program to 170°C at 10°C/min; hold 0; program to 250°C at 50°C/min, hold 1 min, (total run time 17.9 min.)

Results

The polypropylene dash board sample was analysed in full scan. Figure 2 shows all compounds that were tentatively identified using an automated library search against the NIST 08 library.

Figure 2. Full Scan Identification of Compounds in Polypropylene Sample.

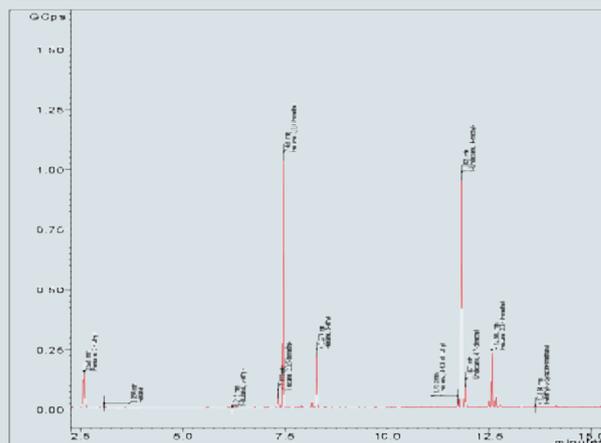


Table 2. Compounds detected in polypropylene dash board sample.

RT (min)	Peak Name	Result Type	Amt	Match	Result	Library
2.581	Pentane, 2-Methyl-	TIC	450552256	1	933	NIST
3.066	Hexane	TIC	43213484	1	903	NIST
6.216	1-Butanol, 2-Ethyl-	TIC	10450404	1	877	NIST
6.766	Toluene	TIC	10159705	1	901	NIST
7.333	Hexane, 2,3,5-Trimethyl-	TIC	139044608	1	925	NIST
7.455	Hexane, 2,3,4-Trimethyl-	TIC	1922249344	1	922	NIST
7.919	2,4-Dimethyl-1-Heptene	TIC	16034614	1	858	NIST
8.16	Nonane, 4-Ethyl-5-Methyl-	TIC	48203884	1	909	NIST
8.279	Hexane, 3-Ethyl-	TIC	418871424	1	921	NIST
11.732	1-Nonene, 4,6,8-Trimethyl-	TIC	85041800	1	876	NIST
11.821	1-Undecene, 4-Methyl-	TIC	1740321664	1	884	NIST
11.921	Undecane, 4,7-Dimethyl-	TIC	208282336	1	883	NIST
12.482	Octane, 2,3,6,7-Tetramethyl-	TIC	48176440	1	878	NIST
12.565	Hexane, 2,3,4-Trimethyl-	TIC	417671808	1	877	NIST
12.657	Hexane, 2,3,4-Trimethyl-	TIC	92484168	1	889	NIST
12.775	Undecane, 4-Methyl-	TIC	21904856	1	870	NIST
13.634	6-Methyl-2-Pyrazinylmethanol	TIC	15546811	1	766	NIST
14.128	Oxalic Acid, Isobutyl Nonyl Ester	TIC	11080139	1	885	NIST
15.271	1-Lodo-2-Methylundecane	TIC	40731856	1	882	NIST
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Full Scan	NA	2.0-17.9	Full (m/z 35-300)	300

Table 4. Tentatively Identified compounds in PVC sample.

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1.283	Hydrazinecarboxamide	TIC	6.61E+06	6613686	1	762	NIST
1.389	Ethyne, Fluoro-	TIC	546473	546473	1	665	NIST
2.159	Acetone	TIC	4.23E+06	4234303	1	867	NIST
3.116	Thiirane	TIC	4.68E+08	4.68E+08	1	907	NIST
3.583	Butane, 2-Nitro-	TIC	1.32E+07	13169822	1	760	NIST
5.659	Pentanal	TIC	5.34E+06	5341163	1	796	NIST
5.705	2-Propenoic Acid, 2-Methyl-, Methyl Este	TIC	5.40E+06	5398963	1	806	NIST
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12.224	Hexane, 2,4-Dimethyl-	TIC	2.94E+07	29438408	1	817	NIST
12.554	Hexane, 1,1-Dichloro-3-Methyl-	TIC	8.90E+06	8901146	1	656	NIST
13.033	Ethanone, 2,2-Dihydroxy-1-Phenyl-	TIC	1.32E+07	13228981	1	879	NIST
13.282	Nonanal	TIC	2.19E+07	21900612	1	865	NIST
13.367	Benzenemethanol, .Alpha.,.Alpha.-Dimethy	TIC	8.96E+06	8960782	1	727	NIST
13.63	6-Methyl-2-Pyrazinylmethanol	TIC	9.97E+06	9966499	1	741	NIST
14.874	2-Decen-1-Ol	TIC	1.31E+07	13070243	1	888	NIST

The PVC sample was run in SIM/Scan mode. Figure 3 shows detection of 1,2-dichloroethane, a target compound with qualifier ion.

Figure 3. SIM/Scan analysis of PVC sample.

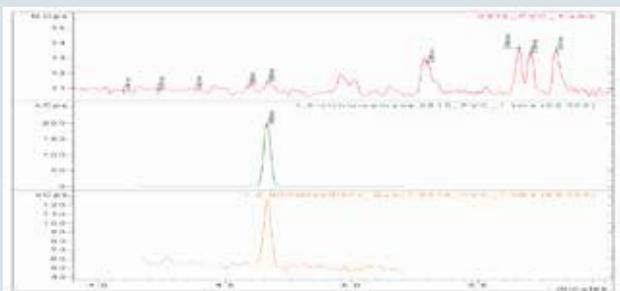
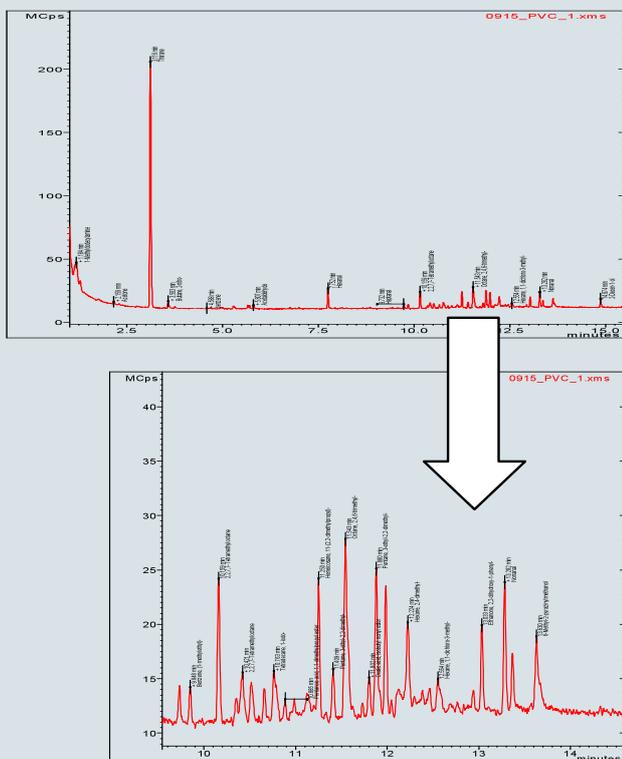


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The compounds were tentatively identified and are listed in Table 3. Lower display is magnification of peaks eluting between 10 and 14 min.

Conclusion

The Scion SHS-40 headspace coupled with the SCION GC/MS is an excellent tool for qualitative and quantitative identification of volatile compounds in raw materials. Using SIM, compounds can be selectively quantitated at very low concentrations. Full scan data can be interrogated for TICs and used for quality control "fingerprints".

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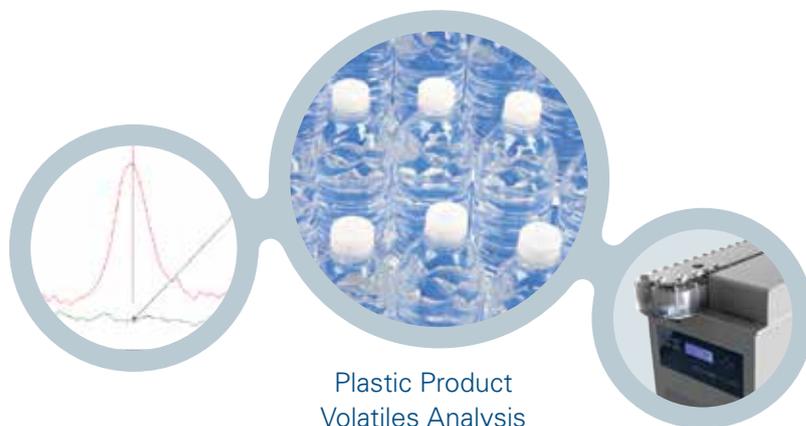
(1) Developmental Effects of Endocrine-disrupting Chemicals in Wildlife and Humans; T. Colborn, F. S. vom Saal, and A. M. Soto, W. Alton Jones Foundation, Washington, DC 20037.

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Plastic Product
Volatiles Analysis

Application Notes #283078

Direct Analysis of Finished Plastic Products by Heated Head Space and GC/MS

A sample of raw or finished material is directly analysed for volatile components using an automated head space auto sampler. Little or no sample preparation is required, making it an ideal technique for high throughput quality control testing in manufacturing. When operated in a synchronous SIM/Scan mode, the SCION SQ single quadrupole mass spectrometer provides quantitative data, along with tentatively identified compounds (non-target compounds).

Introduction

Material testing for outgassing volatile organic chemicals is required in many industries to ensure consumers are not being exposed to harmful contaminants. This is especially important when a material such as a plastic is exposed to excess heat with little or no ventilation. A good example would be plastic materials in a car such as dash boards, which are exposed to very high temperatures in direct sunlight. Volatile chemicals are also common in carpeting and polyvinyl chloride (PVC) piping, and may pose a risk in poorly ventilated homes.

Chlorinated solvents that outgas from materials are generally considered harmful, because they are classified as being carcinogenic or environmental hazards. Other compounds detected may be uncharacterized with little known about

their toxicity. Phthalate esters, for example, have been linked to endocrine disruption in some animal species, and regulations are emerging to control these substances in plastic bottles (1).

Experimental

Samples of polypropylene from a car manufacturer and PVC pipe were cut into small pieces and placed directly into 20mL head space vials. About 500mg-1g was added. The Scion SHS-40 Auto sampler conditions used are cited in Table 1.

Table 1. SHS-40 Sampling Conditions.

Parameter	Set Point
Oven Temp	70°C
Valve/Loop Temp	160°C
Transfer line Temp	125°C
Pressure	500psi
Loop Volume	1mL
PC (incubation) Time	30 min.
GC/MS Run Time	20 min.
Shake option	ON

Figure 3. Scion SHS-40 Headspace Autosampler with SCION GC/MS.



The SCION GC/MS column, oven program, and injector conditions:

Column: BR-624ms, 20M x 0.18mm x 1.0um
 Injector: BR-1079, PTV injector with 3.4mm single goose-neck open split liner set at 200°C
 Injector split ratio: 1:20
 Column flow: 1mL/min
 Oven program: Initial 35°C hold 2 min; program to 170°C at 10°C/min; hold 0; program to 250°C at 50°C/min, hold 1 min, (total run time 17.9 min.)

Results

The polypropylene dash board sample was analysed in full scan. Figure 2 shows all compounds that were tentatively identified using an automated library search against the NIST 08 library.

Figure 2. Full Scan Identification of Compounds in Polypropylene Sample.

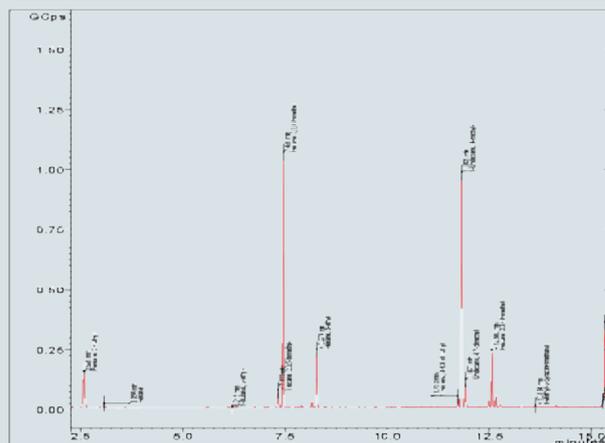


Table 2. Compounds detected in polypropylene dash board sample.

RT (min)	Peak Name	Result Type	Amt	Match	Result	Library
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3.066	Hexane	TIC	43213484	1	903	NIST
6.216	1-Butanol, 2-Ethyl-	TIC	10450404	1	877	NIST
6.766	Toluene	TIC	10159705	1	901	NIST
7.333	Hexane, 2,3,5-Trimethyl-	TIC	139044608	1	925	NIST
7.455	Hexane, 2,3,4-Trimethyl-	TIC	1922249344	1	922	NIST
7.919	2,4-Dimethyl-1-Heptene	TIC	16034614	1	858	NIST
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11.821	1-Undecene, 4-Methyl-	TIC	1740321664	1	884	NIST
11.921	Undecane, 4,7-Dimethyl-	TIC	208282336	1	883	NIST
12.482	Octane, 2,3,6,7-Tetramethyl-	TIC	48176440	1	878	NIST
12.565	Hexane, 2,3,4-Trimethyl-	TIC	417671808	1	877	NIST
12.657	Hexane, 2,3,4-Trimethyl-	TIC	92484168	1	889	NIST
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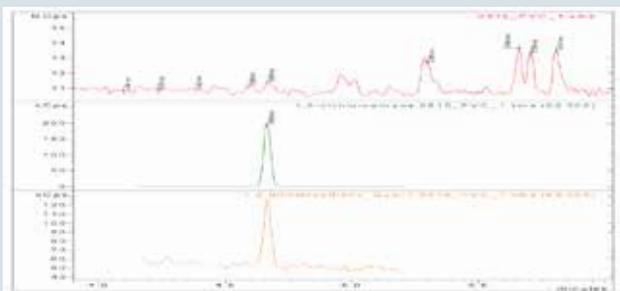
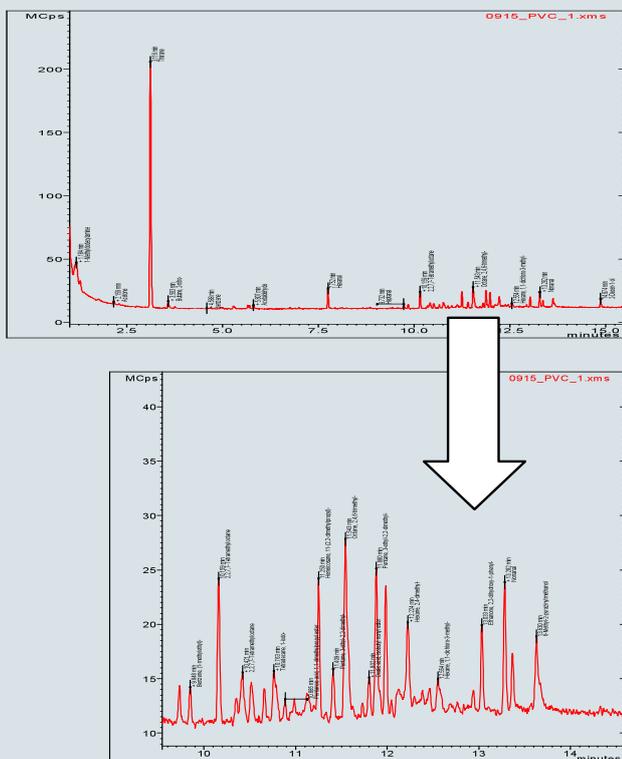


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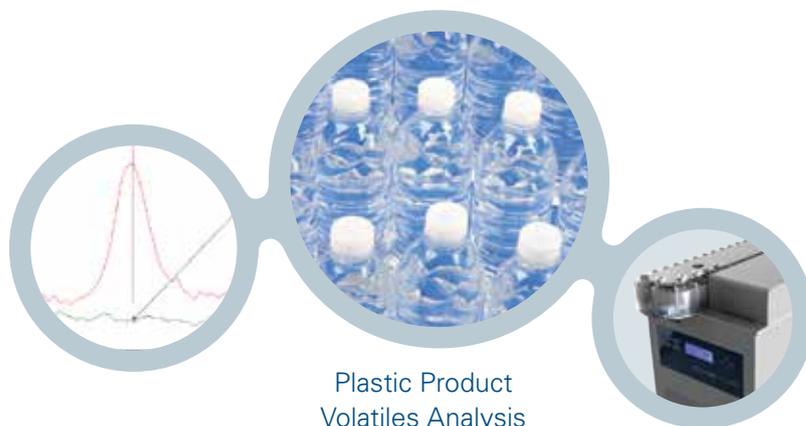
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Volatiles Analysis

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Transfer line Temp	125°C
Pressure	500psi
Loop Volume	1mL
PC (incubation) Time	30 min.
GC/MS Run Time	20 min.
Shake option	ON

Figure 3. Scion SHS-40 Headspace Autosampler with SCION GC/MS.



The SCION GC/MS column, oven program, and injector conditions:

Column: BR-624ms, 20M x 0.18mm x 1.0um
 Injector: BR-1079, PTV injector with 3.4mm single goose-neck open split liner set at 200°C
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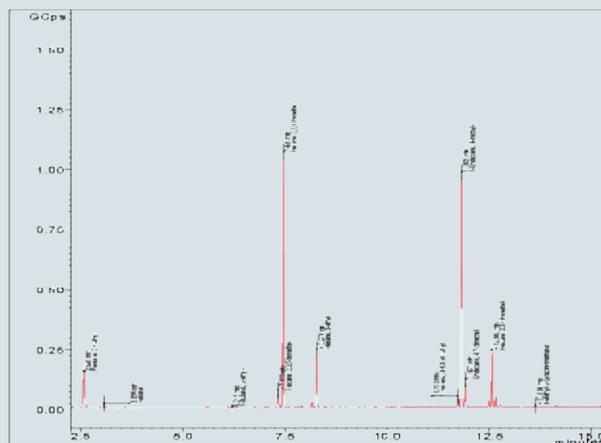


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11.919	Heneicosane, 11-(2,2-Dimethylpropyl)-	TIC	2.79E+06	2789403	1	795	NIST
11.983	Oxalic Acid, Isobutyl Octyl Ester	TIC	2.44E+07	24356798	1	886	NIST
12.224	Hexane, 2,4-Dimethyl-	TIC	2.94E+07	29438408	1	817	NIST
12.554	Hexane, 1,1-Dichloro-3-Methyl-	TIC	8.90E+06	8901146	1	656	NIST
13.033	Ethanone, 2,2-Dihydroxy-1-Phenyl-	TIC	1.32E+07	13228981	1	879	NIST
13.282	Nonanal	TIC	2.19E+07	21900612	1	865	NIST
13.367	Benzenemethanol, .Alpha.,.Alpha.-Dimethy	TIC	8.96E+06	8960782	1	727	NIST
13.63	6-Methyl-2-Pyrazinylmethanol	TIC	9.97E+06	9966499	1	741	NIST
14.874	2-Decen-1-ol	TIC	1.31E+07	13070243	1	888	NIST

The PVC sample was run in SIM/Scan mode. Figure 3 shows detection of 1,2-dichloroethane, a target compound with qualifier ion.

Figure 3. SIM/Scan analysis of PVC sample.

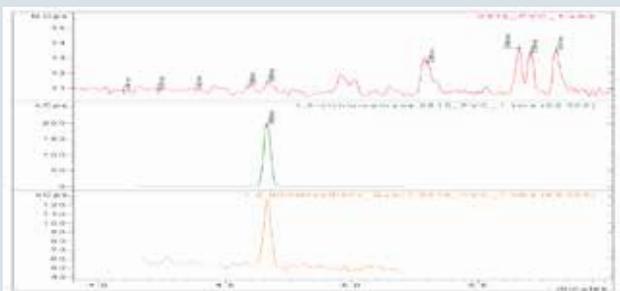
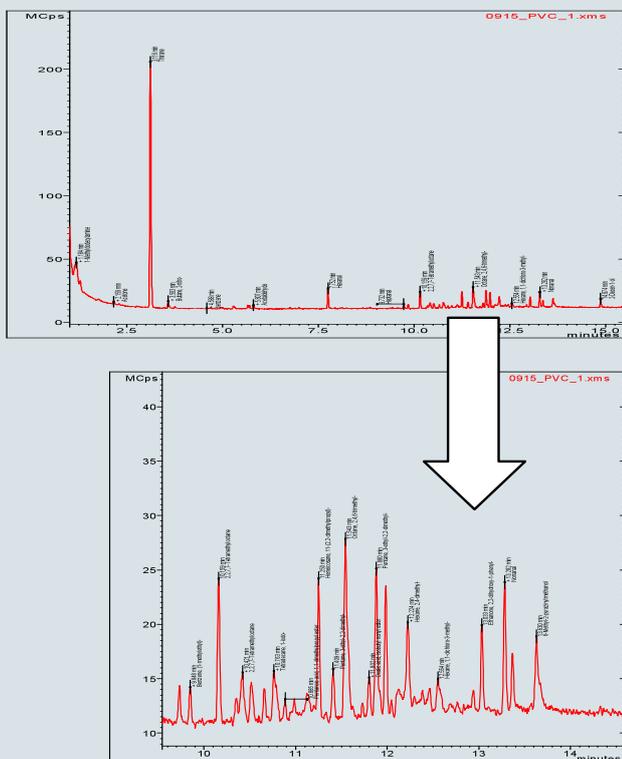


Figure 4. RIC of PVC sample.



The compounds were tentatively identified and are listed in Table 3. Lower display is magnification of peaks eluting between 10 and 14 min.

Conclusion

The Scion SHS-40 headspace coupled with the SCION GC/MS is an excellent tool for qualitative and quantitative identification of volatile compounds in raw materials. Using SIM, compounds can be selectively quantitated at very low concentrations. Full scan data can be interrogated for TICs and used for quality control "fingerprints".

References

(1) Developmental Effects of Endocrine-disrupting Chemicals in Wildlife and Humans; T. Colborn, F. S. vom Saal, and A. M. Soto, W. Alton Jones Foundation, Washington, DC 20037.

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Application Note # CA 284780

Meeting Challenging Laboratory Requirements for USEPA Method 8270 Using a Highly Sensitive, Robust, and Easy-to-Use GC/MS

Abstract

The analysis of semi-volatile organic compounds (SOCs) using EPA Method 8270 presents challenges due to the wide variety of acids, bases, and neutrals that must be analysed in extremely complex sample extracts. Laboratories are under pressure from their customers to provide lower detection limits, faster sample turn-around-time, and detailed reports that contain all the validated quality control data. In addition, these labs want a GC/MS system that is easy to set up and manage, because constant requests for new target analytes increase the overall burden of data analysis and processing when changes to the existing method are required. The Scion Single Quadrupole (SQ) Mass Spectrometer is designed to meet these new challenges and provides a total solution to laboratories for USEPA Method 8270.

Introduction

Traditionally, USEPA Method 8270 has been used to analyze a variety of complex sample matrices using full scan GC/MS. Most labs analyze a subset of the compounds listed in the method, typically 75 to 100, at a calibration concentration range of 1 to 200 ppm. Newer versions of the method allow for the use of selected ion monitoring (SIM), which

can significantly lower detection limits. Mixed mode scan methods, such as SIM/Scan, have the benefits of lowering detection limits and simultaneously providing full scan data for library search confirmation of target compounds and tentative identification of any unknown peaks in a chromatogram.

To take full advantage of this mixed mode approach the mass spectrometer must be capable of fast acquisition speed, especially if large numbers of compounds are added to the SIM component of the method. Secondly, the analytical system must have excellent sensitivity in the full scan mode, because library search results will be used for confirmation in many cases. Finally, the data acquisition and processing must be easy to maintain and manage in the mixed mode to meet the demand for the ever-increasing number of target compounds.

The Scion SQ meets these challenges by providing an inert ion source and revolutionary ion optics to obtain part-per-billion sensitivity in full scan analysis. In addition, software known as Compound Based Scanning (CBS) makes it easier than ever before to set up, optimize, and maintain complex mixed mode methods. Additional tools

such as tune-to-target and custom EPA reporting templates round out a complete solution for USEPA Method 8270.

Experimental

The following conditions were used for set up of the gas chromatograph and full scan mass spectrometry components of Method 8270. A pulsed-split injection was used for the analysis, which provided excellent sensitivity with a minimized amount of sample entering the column. The Scion SQ under these conditions is capable of easily detecting and quantitating concentrations of most analytes down to 75 ppb or lower in full scan, which is 10-100 times less than what is required in the method.

In the mixed scan mode, all 100 components were analyzed in full scan, along with 57 compounds in SIM mode. See Table 1 for analytes and SIM ions monitored for each.

Calibration standards were prepared at 0.075, 0.15, 1, 2, 5, 10, 30 ppm. Seventeen very dirty sludge extracts were provided by a local environmental laboratory, see Figure 1 showing an example extract.

451-GC Gas Chromatographic Conditions

Column:	BR-5ms, 30 m x 0.25 mm x 0.25 um
Column Flow Rate:	1.0 mL/min constant flow
Injector:	Scion Split/Splitless injector, with 4 mm ID Siltek Fritted Liner (Part No.)
Injector Conditions:	40 psi pulsed split set 1:50 for 0.30 min at 250 °C
Column Temperature Program:	45 °C, hold 3 min, ramp to 120 at 30 °C/min, hold 1 min; ramp to 310 °C at 10 °C/min, hold 5 min.
Injection:	1.0 uL using Bruker 8400 auto sampler

Scion SQ Full Scan MS Conditions

Scan Range:	45-450, unit mass resolution
Scan rate:	200 ms, positive ion polarity
Ion Source Temperature:	300 °C
Transferline Temperature:	280 °C
Filament Delay time:	2 min
Filament Emission Current:	80 uA

Compound Name	Retention Time	SIM Ion
N-Nitrosodimethylamine	3.639	74
2-Fluorophenol	5.177	112
Phenol-d5	6.106	99.1
Phenol	6.119	94.1
bis(2-chloroethyl)ether	6.212	93
2-Chlorophenol	6.290	128
1,4-Dichlorobenzene-d4	6.542	152
N-nitroso-di-n-propylamine	7.071	70
Nitrobenzene-d5	7.312	82
2,4-Dimethylphenol	7.892	107
2,4-Dichlorophenol	8.237	162
Naphthalene	8.524	128
4-Chloro-3-methylphenol	9.597	107
2-Methylnaphthalene	9.867	142.2
1-Methylnaphthalene	10.071	142.2
2,6-Dimethylnaphthalene	10.248	156
2,4,6-Trichlorophenol	10.491	196
2,4,5-Trichlorophenol	10.563	196
2-Fluorobiphenyl	10.673	172
2,3,4-Trichlorophenol	10.698	196
Biphenyl	10.885	154
Dimethylphthalate	11.633	163
Acenaphthylene	11.841	152.2
Acenaphthene	12.242	153.2
2,3,5-Trimethylnaphthalene	13.139	170
Diethylphthalate	13.278	149.1
Fluorene	13.444	149.1

Compound Name	Retention Time	SIM Ion
Azobenzene	13.853	77
2,4,6-Tribromophenol	14.023	330
Hexachlorobenzene	14.731	284
Pentachlorophenol	15.240	266
Phenanthrene-d10	15.658	188.1
Phenanthrene	15.714	178.2
Anthracene	15.834	178.2
Carbazole	16.281	167
1-Methylphenanthrene	17.188	192
Di-n-butylphthalate	17.195	149.1
Fluoroanthene	18.555	202.2
Pyrene	19.089	202.2
Terphenyl-d14	19.544	244.3
Butylbenzylphthalate	20.784	149.1
Chrysene	21.975	228.2
Benzo(a)anthracene	22.062	228.2
Bis(2-ethylhexyl)phthalate	22.176	149.1
Di-n-octylphthalate	23.649	149.1
Benzo(b)fluoranthene	24.376	252.2
Benzo(k)fluoranthene	24.436	252.2
Benzo(e)pyrene	24.945	252.2
Benzo(a)pyrene	25.047	252.2
Perylene-d12	25.171	264.2
Perylene	25.224	252.2
Indeno(123-cd)pyrene	27.469	276.3
Dibenz(ah)anthracene	27.531	278.3
Benzo(ghi)perylene	28.149	276.2

Table 1: SIM ions monitored in mixed mode on the Scion SQ

CBS is therefore a powerful tool to easily manage a large number of SIM ion acquisitions for a complex method like 8270. No need to worry about too many ions in a single segment, segment breaks, or whether one compound needs to be in more than one segment.

CBS also automatically creates the data handling method that is associated with the acquisition method, linking the tables. Therefore, if there is a change in the acquisition method, ie a compound added or deleted, the change will be reflected in the data handling method table as well. This saves the operator time because only one list of method compounds and conditions needs to be managed.

The mixed scan mode in CBS provides excellent sensitivity, as shown in the example chromatogram in Figure 8. Benzo(ghi)perylene is a PAH that elutes late in the chromatographic run, often with excessive column bleed. SIM monitoring provides clean baseline and enhanced signal-to-noise resulting in lower detection limits.

checks (CCC) are used to monitor the recovery of internal standards before and after sample extracts are run to ensure that there is minimal loss in sensitivity. An example of internal and surrogate standard peaks monitored in the CCC before and after 17 sludge extracts is shown in Table 3.

Standard	Perylene-d12	Phen-	Terphenyl-d14	Phenol-d5	Nitrobenzene-d5
Peak Area 1st CCC	93230000	230600000	36960000	34380000	18170000
Peak Area Last CCC	71140000	240900000	36540000	36900000	20110000
% Difference	23.69	4.47	1.14	7.33	10.68

Table 3: Peak areas in the CCC of internal and surrogate standards before and after sludge extracts. Peak shape and response were maintained in spite of over 30 injections of the sludge extracts, as shown in Figure 9 below.

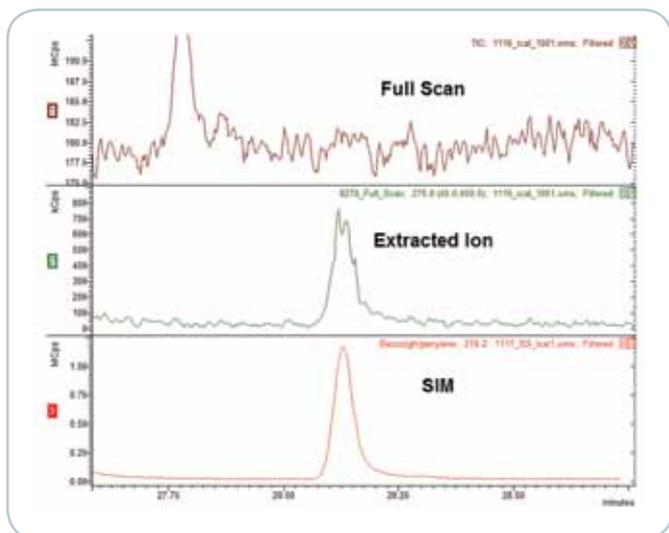


Figure 8: Mixed mode acquisition using CBS of 0.075 ppm calibration standard. Ion at m/z 276 can be extracted from the full scan data (green), but SIM run (red) simultaneously give best signal-to-noise and peak shapes to be used for low-level quantitation.

DFTPP can also be checked and will pass method criteria when the Scion SQ is operated in the mixed mode. This is because of the high speed scan rate combined with the optimal placement of the SIM scans in the acquisition. Another important aspect in the routine operation of the method is the ability to maintain good data quality while analysing dirty sample extracts. Since the Scion SQ is very sensitive, a pulsed-split injection can be used instead of a pulsed-splitless injection. Pulsed-split decreases the total amount placed onto the column, therefore increases the life of the column, liner, and thus improves throughput. Laboratories that analyse sludge extracts like that shown in Figure 1 often see loss of internal standards and poor peak shape after only a few injections. Continuing calibration

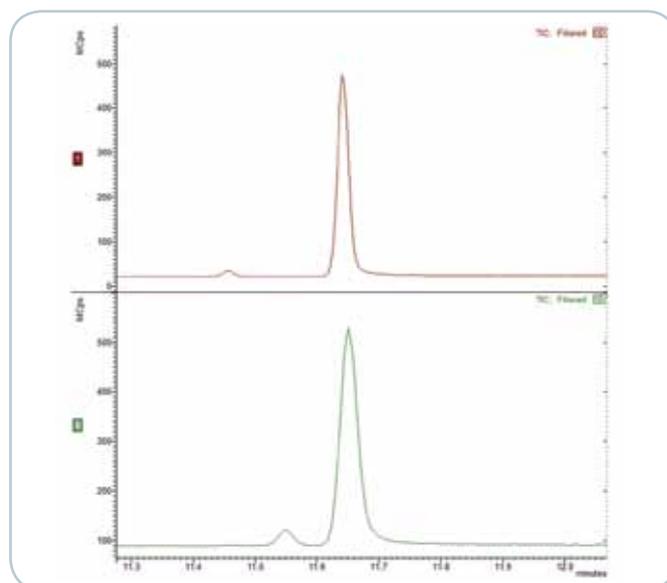


Figure 9: Excellent peak shape and response for pentachlorophenol is still maintained after 30 injections of sludge extracts. (Top- PCP peak before injection of extracts, Bottom-PCP peak after injection of extracts).

Reporting of the samples and quality control is the final step that must be completed by the lab. Scion offers EnviroPro™, a Microsoft Access database that will generate all of the required reports for EPA Method 8270, as well as several other methods. Example reports are tune criteria, method detection limit calculations, initial calibration reports, and continuing calibration checks. There are several graphic options available for printing chromatograms and target compounds, as well as unknown peaks (nontarget analytes). Qualitative interrogation of full scan data is another very important reason to run Method 8270 in the mixed mode.

Results

All users of USEPA Method 8270 know that passing the DFTPP tune is critical to the validity of the sample data. The Bruker Scion SQ has built-in tune-to-target ratio in mass calibration for DFTPP, as shown in Figure 2.

A tune report is easily generated using built-in report formats by simply selecting the data file and DFTPP peak, and then generating a pass/fail report as shown in Figure 3.

System Performance Check Compounds (SPCCs) are used to check the integrity of the liner/column and the inertness/cleanliness of the ion source region. Pentachlorophenol is a good test for active sites, as it tends to fail peak Gaussian test probes when the system becomes dirty. SPCCs can be injected and then immediately checked for degradation, Gaussian peak shape, or resolution in one easy reporting tool that uses Microsoft Access to generate the required report. An example report for pentachlorophenol peak shape on the Scion SQ is shown in Figure 4.



Figure 1: Clean extract (left), sludge extract (right)

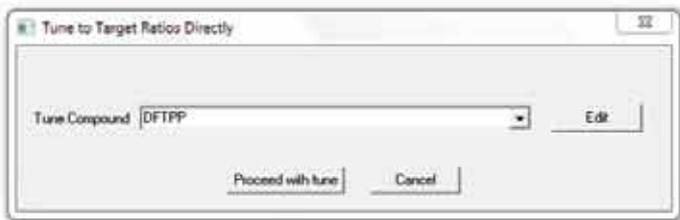


Figure 2: Tune-to-target for EPA tuning compounds are selectable in the software, and ratios can be easily edited if further optimization is required.

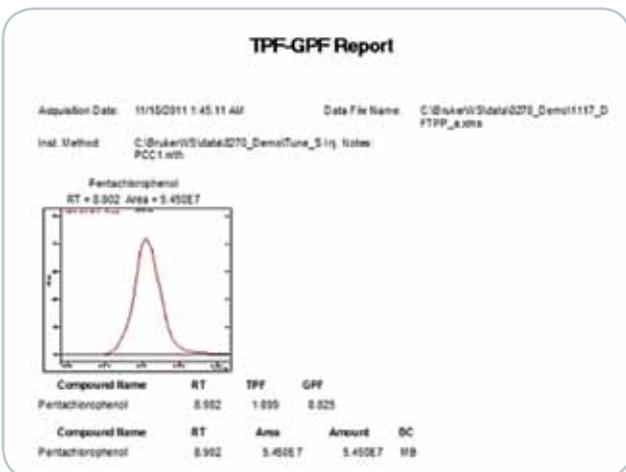
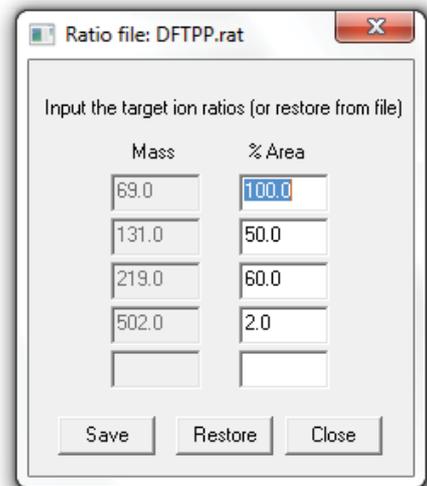


Figure 3: Example tune report for DFTPP

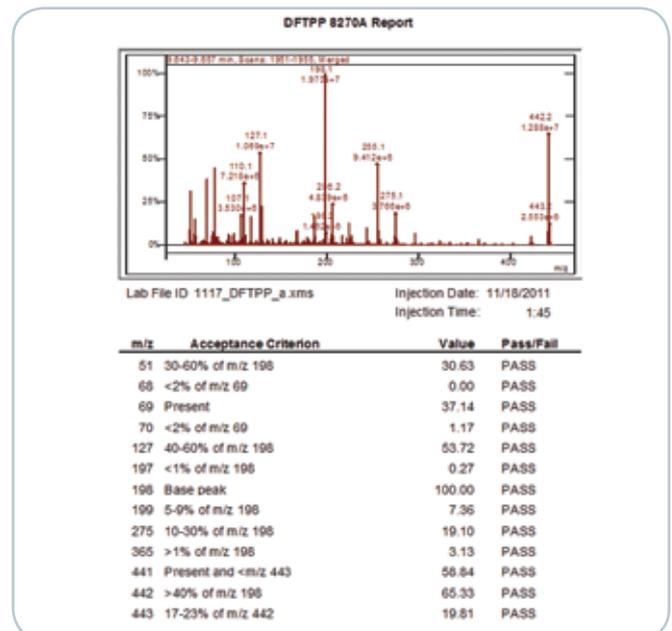


Figure 4: Peak tailing factor and Peak Gaussian report generated for the SPCC sample in Scion MSWS 8 Custom

Access reports

The other SPCCs are listed below in Table 2 as average RRF compared to the method 8270 criteria.

Compound	Method 8270 Criteria (Min RRF)	RRF on Scion SQ Cal Range
N-nitroso-di-n-propylamine	0.05	0.109
Hexachlorocyclopentadiene	0.05	0.138
2,4-Dinitrophenol	0.05	0.149
4-Nitrophenol	0.05	1.03

Table 2: SPCC Results

After the system checks out a calibration series is analysed. In this case, the laboratory required a much lower concentration range than suggested by the method, from 0.075 ppm to 30 ppm (Method uses 5 ppm to 200 ppm). These concentrations clearly challenge the analytical system, especially for compounds that tend to be absorbed by active sites at low concentration. Figure 5 is an example section of a chromatogram in full scan mode for the calibration standard at 0.075 ppm. Internal standards are at a concentration of 40 ppm as recommended by the method. Note that the trichlorophenols are easily detected in scan mode.

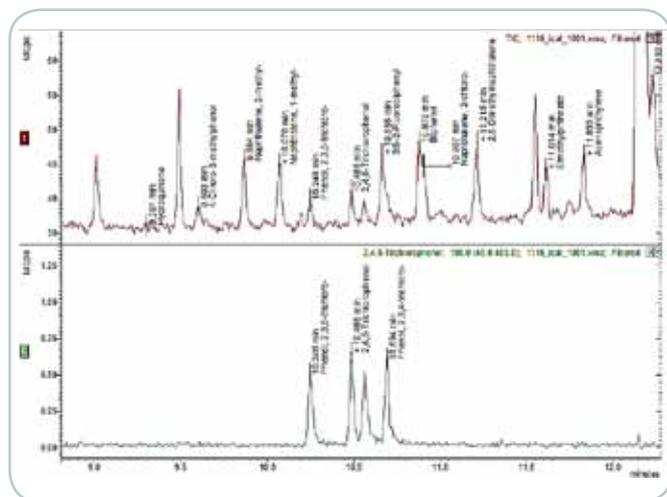


Figure 5: Full Scan of 0.075 ppm calibration standard (Top) and extracted ion chromatogram (bottom) for the trichlorophenols

Calibration curves for most analytes were linear over the calibration range, even for difficult compounds like pyridine as shown below. As seen here, the Scion SQ is a very inert system as there is no loss of the pyridine at the low end of the calibration curve.

Overall Calibration Statistics:

Average % RSD = 11.62 %

Average Correlation Coefficient = 0.9989

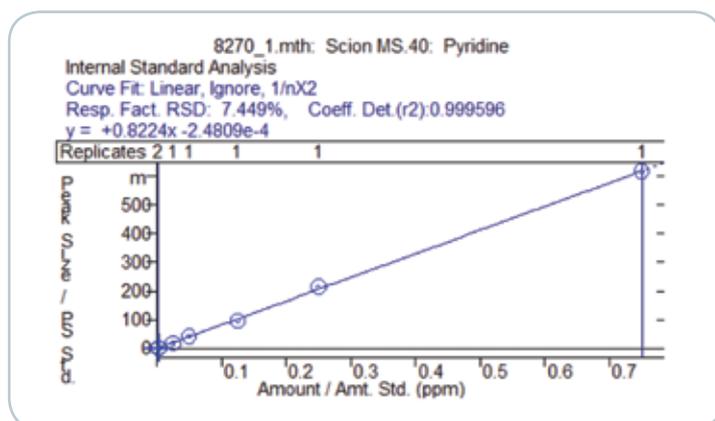


Figure 6: Pyridine calibration curve from 0.075 ppm to 30 ppm in full scan of the Scion SQ.

In order to gain even more sensitivity for key target compounds in Method 8270, SIM mode is often used. Modern GC/MS systems can be set up in a mixed scan mode, in which SIM ions are monitored at the same time during a full scan acquisition. The Scion SQ has a unique software feature known as Compound Based Scanning (CBS), in which SIM ions for compounds are stored in either a factory supplied library or user created library. The scan information, compound retention times, and individual dwell times are all stored and are easily selected and loaded directly into a data acquisition method.

CBS automatically optimizes scan times based upon desired number of data points to be acquired for a chromatographic peak, typically 10 points, so that accurate and precise quantitation is possible. The user does not have to manage complex time segments, because CBS automatically optimizes SIM scans throughout the chromatographic run based upon the retention time and retention time window. See Figure 7.

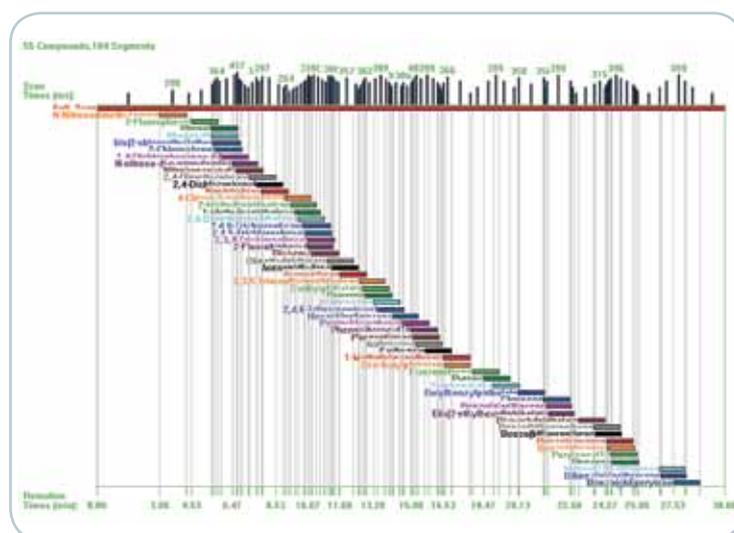


Figure 7: Mixed mode data acquisition for Method 8270. CBS automatically optimizes scanning ranges for target SIM ions. The full scan segment (red and on top) occurs throughout the run. The total scan time is represented by a bar graph at the top.

Conclusion

The Scion SQ GC/MS and MSWS software has been shown to be a complete solution for USEPA Method 8270. The robust ion source and sensitivity of the instrument allow for lower reporting limits in challenging sample extracts.

Compound based scanning is a novel approach allowing easy set-up and management of complex mixed mode methods. Compound scan information is loaded directly into the method by choosing them from a factory or user created library. It automatically optimizes scan times for SIM ions, and links the mass spectrometer acquisition table directly with data handling parameters, saving time and making it easier to add or delete compounds.

Standard EPA templates for Method 8270 and several other EPA methods are available in EnviroPro™, a Microsoft Access database reporting package.

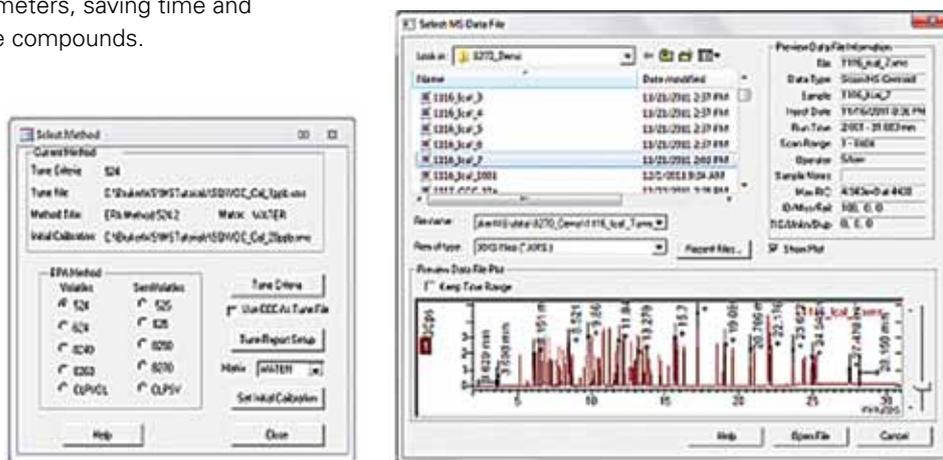


Figure 10: The Scion EnviroPro™ software package for environmental methods in Microsoft Access 2010.

Instrumentation & Software
Scion SQ
451-GC
436-GC
8400 A/S

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