

# Agilent 5977B GC/MSD

Application Compendium



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# Agilent 5977B GC/MSD

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## Determination of Volatile Organic Compounds in Water by Purge and Trap Gas Chromatography/Mass Spectrometry

Using the Chinese Ministry of Environmental Protection method HJ 639-2012

**Author**  
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**Abstract**  
This application note highlights the determination of 57 volatile organic compounds (VOCs) in water using an Agilent Intuvo 9000 GC, an Agilent 5977B GC/MSD, and a Teledyne Tekmar Atomx XYZ purge and trap sample preparation system. Great performance was achieved with linearity across the expected range of concentrations and repeatability through eight injections. The limit of detection (LOD) and limit of quantitation (LOQ) were researched in both scan and selected ion monitoring (SIM) modes.

## Determination of Volatile Organic Compounds in Water by Purge and Trap GC/MS

This application note highlights the determination of 57 volatile organic compounds (VOCs) in water using an Agilent Intuvo 9000 GC, an Agilent 5977B GC/MSD, and a Teledyne Tekmar Atomx XYZ purge and trap sample preparation system. Great performance was achieved with linearity across the expected range of concentrations and repeatability through eight injections. The limit of detection (LOD) and limit of quantitation (LOQ) were researched in both scan and selected ion monitoring (SIM) modes.

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## Volatile Organic Compounds Analysis in Soils and Sediments Using the Agilent 8697 Headspace Sampler

**Author**  
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**Abstract**  
This application note describes volatile organic compounds analysis in soil and sediments using the Agilent 8697 headspace sampler, 8860 GC, and 5977B GC/MSD system. The system performance in terms of repeatability, limit of detection, limit of quantitation, and method recovery rate were evaluated with good results. The area repeatability was in the range of 1.0 to 4.3%; the LOD and LOQ in the quartz sand blank was from 0.51 to 1.21 µg/kg and from 1.7 to 4.1 µg/kg, respectively. The recovery rate for the soil samples at spiked concentrations of 50 and 125 µg/kg was 78.2 to 125.9% and 71.7 to 108.7%. The linearity across the tested concentration range is excellent, with the R<sup>2</sup> of all components better than 0.996. The test results met or exceeded the requirements of Chinese standard HJ 642-2013.

## Volatile Organic Compounds Analysis in Soils and Sediments Using the Agilent 8697 Headspace Sampler

This application note describes volatile organic compounds analysis in soil and sediments using the Agilent 8697 headspace sampler, 8860 GC, and 5977B GC/MSD system. The system performance in terms of repeatability, linearity, limit of detection, limit of quantitation, and method recovery rate were evaluated with good results. The area repeatability was in the range of 1.0 to 4.3%; the LOD and LOQ in the quartz sand blank was from 0.51 to 1.21 µg/kg and from 1.7 to 4.1 µg/kg, respectively. The recovery rate for the soil samples at spiked concentrations of 50 and 125 µg/kg was 78.2 to 125.9% and 71.7 to 108.7%. The linearity across the tested concentration range is excellent, with the R<sup>2</sup> of all components better than 0.996. The test results met or exceeded the requirements of Chinese standard HJ 642-2013.

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### Accelerated Determination of Microplastics in Environmental Samples Using Thermal Extraction Desorption-Gas Chromatography/Mass Spectrometry (TED-GC/MS)

**Authors**  
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Karl Theissen and Eke Klene Barne, QS&S, Inc.  
Tara Arnold, Agilent Technologies, Inc.

**Abstract**  
There is growing interest in quantifying microplastics in environmental samples. This application note presents a thermal extraction desorption-gas chromatography/mass spectrometry (TED-GC/MS) method that is well suited to automation and increased sample throughput. The method is also able to detect all particle sizes in the sample as long as the limit of detection (LOD) is reached and allows analysis of larger samples of 15 to 25 mg or more. Samples were decomposed by thermogravimetric analysis (TGA), and the gaseous decomposition products were trapped on a solid-phase sorbent, followed by thermal desorption-gas chromatography/mass spectrometry (TD-GC/MS) using an Agilent 5977B GC/MSD coupled to an Agilent 7890B GC. Target microplastic particle (MP) polymers were identified in environmental samples including surface water, finished compost, house dust, and drinking water. Quantification of MP polymers in environmental samples provided LODs of 0.06 to 2.2 µg, allowing the detection of MPs in trace amounts with sample weights of up to 1 g. Method repeatability was adequate for reliable quantification with RSDs of approximately 6 to 12%.

## Accelerated Determination of Microplastics in Environmental Samples using Thermal Extraction Desorption-Gas Chromatography/Mass Spectrometry (TED-GC/MS)

There is growing interest in quantifying microplastics in environmental samples. This application note presents a thermal extraction desorption-gas chromatography/mass spectrometry (TED-GC/MS) method that is well suited to automation and increased sample throughput. The method is also able to detect all particle sizes in the sample as long as the limit of detection (LOD) is reached and allows analysis of larger samples of 15 to 25 mg or more. Samples were decomposed by thermogravimetric analysis (TGA), and the gaseous decomposition products were trapped on a solid-phase sorbent, followed by thermal desorption-gas chromatography/mass spectrometry (TD-GC/MS) using an Agilent 5977B GC/MSD coupled to an Agilent 7890B GC. Target microplastic particle (MP) polymers were identified in environmental samples including surface water, finished compost, house dust, and drinking water. Quantification of MP polymers in environmental samples provided LODs of 0.06 to 2.2 µg, allowing the detection of MPs in trace amounts with sample weights of up to 1 g. Method repeatability was adequate for reliable quantification with RSDs of approximately 6 to 12%.

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Application Note  
Environmental

### Analysis of US EPA TO-15 for Ambient Air Monitoring Using Cryogen-Free Thermal Desorption and Gas Chromatography Coupled to a Single Quadrupole Mass Spectrometer (GC/MSD)

**Authors**  
Harsh Gulati and Helen Martin, Mueser International Ltd, Llanrwst, UK  
Tara Arnold and Cassie Eadie, Agilent Technologies, Inc., Wilmington, DE, USA

**Abstract**  
This application note describes the GC/MS analysis of humidified canister 'air toxics' samples at various relative humidities, using cryogen-free systems for thermal desorption preconcentration. Detection of 65 target compounds ranging in volatility from propene to naphthalene is demonstrated, with excellent peak shape and performance well within the criteria set out in US EPA method TO-15, including method detection limits as low as 4 pptv.

## Analysis of US EPA TO-15 for Ambient Air Monitoring Using Cryogen-Free Thermal Desorption and Gas Chromatography Coupled to a Single Quadrupole Mass Spectrometer (GC/MSD)

This application note describes the GC/MS analysis of humidified canister 'air toxics' samples at various relative humidities, using cryogen-free systems for thermal desorption preconcentration. Detection of 65 target compounds ranging in volatility from propene to naphthalene is demonstrated, with excellent peak shape and performance well within the criteria set out in US EPA method TO-15, including method detection limits as low as 4 pptv.

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# Agilent 5977B GC/MSD Application Notes

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## Quantification of Microplastics in Environmental Samples Using Pyrolysis and GC/MSD

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Koblenz-Landau, Germany  
Jiang Tianer  
Agilent Technologies, Inc.

**Abstract**  
There is growing interest in quantifying microplastics in environmental samples. This application note presents a robust pressurized liquid extraction (PLE) with pyrolysis-gas chromatography-mass spectrometry (pyr-GC/MS) method for quantification of microplastics like polyethylene (PE), polypropylene (PP), and polystyrene (PS) at low concentrations in environmental matrices using the Agilent 5977B GC/MSD, Agilent 7890B GC, and Agilent MassHunter workstation software. Limits of quantitation (LOQs) and reproducibility for real environmental samples were evaluated. The GC/MSD addressed the insufficient limits of detection that have challenged previous methods. PE, PP, and PS microplastics were quantified down to 0.005 mg/g. Excellent linearity ( $R^2 > 0.97$ ) for calibration samples from 0.005 to 1 mg/g was obtained. Relative standard deviations (RSDs) for both spiked and environmental samples were <10% or lower, demonstrating excellent system reproducibility and reliability.

## Quantification of microplastics in environmental samples using pyrolysis and GC/MSD

There is growing interest in quantifying microplastics in environmental samples. This application note presents a robust pressurized liquid extraction (PLE) with pyrolysis-gas chromatography-mass spectrometry (pyr-GC/MS) method for quantitation of microplastics like polyethylene (PE), polypropylene (PP), and polystyrene (PS) at low concentrations in environmental matrices using the Agilent 5977B GC/MSD, Agilent 7890B GC, and Agilent MassHunter workstation software. Linearity, limits of quantitation (LOQs), and reproducibility for real environmental samples were evaluated. The GC/MSD addressed the insufficient limits of detection that have challenged previous methods. PE, PP, and PS microplastics were quantified down to 0.005 mg/g. Excellent linearity ( $R^2 > 0.97$ ) for calibration samples from 0.005 to 1 mg/g was obtained. Relative standard deviations (RSDs) for both spiked and environmental samples were <10% or lower, demonstrating excellent system reproducibility and reliability.

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Environmental

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## Comparison of Fritted and Wool Liners for Analysis of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry

**Author**  
Angela Smith Henry PhD  
Agilent Technologies, Inc.

**Abstract**  
Gas chromatography/mass spectrometry (GC/MS) is commonly used in the analysis of semivolatile organic compounds in environmental matrices. Selecting the correct liner for an analysis, such as environmental matrices with nonvolatile compounds, can lead to less downtime of the GC/MS system for maintenance by providing longer lifetimes. Typically liners packed with glass wool or sintered frit liners are utilized for environmental analyses. This study shows that the Agilent Ultra Inert splitless low fritted liner is more resilient to a matrix challenge than splitless glass wool liners, as the sintered frit provided a significant barrier for matrix.


## Comparison of Fritted and Wool Liners for Analysis of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry

This study shows that the Agilent Ultra Inert splitless low fritted liner is more resilient to a matrix challenge than splitless glass wool liners.

Gas chromatography/mass spectrometry (GC/MS) is commonly used in the analysis of semivolatile organic compounds in environmental matrices. Selecting the correct liner for an analysis, such as environmental matrices with nonvolatile compounds, can lead to less downtime of the GC/MS system for maintenance by providing longer lifetimes. Typically, liners packed with glass wool or sintered frit liners are utilized for environmental analyses. This study shows that the Agilent Ultra Inert splitless low fritted liner is more resilient to a matrix challenge than splitless glass wool liners, as the sintered frit provided a significant barrier for matrix.

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Application Note  
Food Testing and  
Agriculture



### Determination of 2-MCPD and 3-MCPD Fatty Acid Esters in Infant Formula Using an Agilent 8890 GC System with an Agilent 5977B GC/MSD

**Authors**  
Yujuan Zhang, Xia Yang, and  
Shua Wang  
Agilent Technologies  
Shanghai Co. Ltd. Shanghai  
200131 P. R. China


**Abstract**  
This application note describes a reliable analytical method for determining the fatty acid esters of 3-monochloropropane-1,2-diol (3-MCPD) and 2-monochloropropane-1,3-diol (2-MCPD) in infant formula. Two different derivatization reagents, heptafluorobutyrylimidazole (HFBI) and phenylboronic acid (PBA), were evaluated for sample preparation. An Agilent 8890 GC system coupled with an Agilent 5977B GC/MSD was used for qualitative and quantitative analyses. Results demonstrated the benefits of the workflow solution for the analysis of monochloropropanediols in infant formula. Great peak shape and resolution were obtained. Satisfactory recoveries were achieved, ranging from 86.9 to 106.7%. Precision was also good, with the relative standard deviations less than 15%.

## Determination of 2- and 3-MCPD Fatty Acid Esters in Infant Formula Using GC/MSD

This application note describes a reliable analytical method for determining the fatty acid esters of 3-monochloropropane-1,2-diol (3-MCPD) and 2-monochloropropane-1,3-diol (2-MCPD) in infant formula. Two different derivatization reagents, heptafluorobutyrylimidazole (HFBI) and phenylboronic acid (PBA), were evaluated for sample preparation. An Agilent 8890 GC system coupled with an Agilent 5977B GC/MSD was used for qualitative and quantitative analyses. Results demonstrated the benefits of the workflow solution for the analysis of monochloropropanediols in infant formula. Great peak shape and resolution were obtained. Satisfactory recoveries were achieved, ranging from 86.9 to 106.7%. Precision was also good, with the relative standard deviations less than 15%.

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Application Note



### Rapid Rinse and Shoot: Screening Workflow for Pesticides in Fruit by GC/MSD in Under Six Minutes

**Authors**  
Anastasia A. Andreanova and  
Bruce D. Quimby  
Agilent Technologies, Inc.

**Abstract**  
The Agilent Intuvo 9000/5977B GC/MSD system enabled a fast screening workflow for residual pesticides present on the surface of fruits. This method used a GC oven with direct heating technology and MS3 spectral deconvolution. Residual pesticides were rinsed from the isolated commodity surface with acetone. The rinse was collected and injected into the GC/MSD system. The direct heating oven allowed a very high temperature program (up to 350 °C/min) to complete the GC/MSD analysis in 3.4 minutes. Spectral deconvolution coupled with the library search algorithm and time filtering using retention indices resulted in rapid and confident identification of residual pesticides present on the fruit. The NIST 172 spectral library, other commercially available libraries, and user-created libraries can be used for compound identification. MassHunter Unknowns Analysis software provides capabilities to create custom reports. The entire analysis from sample collection to reporting took under 6 minutes. The combination of the Intuvo Guard Chip and column decontamination yielded longer maintenance-free uptime. This approach is particularly useful for prioritizing samples for more in-depth analysis.

## Rapid Rinse & Shoot, Screening Workflow of Pesticides in Fruit by GC/MSD in 6 Minutes

Trace-level pesticide and environmental pollutants in the food supply continue to be a worldwide concern. These concerns are driving the demand for more rapid and reliable methods of analysis. The challenge is to find technologies that can search for hundreds of pesticides, PAHs, and other targets with simple sample preparation and a quick turnaround time. The Intuvo 9000/5977B GC/MSD system enables rapid screening for pesticides and other contaminants found on the surface of fruits and berries in 3.4 minutes.

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# Agilent 5977B GC/MSD Application Notes

Application Note  
Food Testing

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## Estimation of $\beta$ -Sitosterol in Milk Fat (Ghee) Samples

Agilent 8890/5977B Single Quadrupole GC/MS System



**Authors**  
Praveen Arora, Vishal Dhyani,  
and Dr. Suresh Yadao  
Agilent Technologies, Inc.

**Abstract**  
This application note demonstrates the use of the Agilent 8890 GC and the Agilent 5977B GC/MS single quadrupole mass spectrometer in the detection and quantification of  $\beta$ -sitosterol in milk fat samples to check for vegetable oil adulteration.  
The method provides the highest confidence for routine analysis of milk fat samples in the food industry, whether it is used in manufacturing, processing, commercial testing, or academia. Sample preparation for this method involved saponification, followed by extraction of unsaponifiable matter by liquid-liquid extraction (LLE) and derivatization of sitosterol to its trimethylsilyl derivative.

## Estimation of $\beta$ -sitosterol in Milk Fat (Ghee) by Agilent 8890 GC and 5977B MS

This application note demonstrates the use of the Agilent 8890 GC and the Agilent 5977B GC/MS single quadrupole mass spectrometer in the detection and quantification of  $\beta$ -sitosterol in milk fat samples to check for vegetable oil adulteration. The method provides the highest confidence for routine analysis of milk fat samples in the food industry, whether it is used in manufacturing, processing, commercial testing, or academia. Sample preparation for this method involved saponification, followed by extraction of unsaponifiable matter by liquid-liquid extraction (LLE) and derivatization of sitosterol to its trimethylsilyl derivative.

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Food Testing & Agriculture

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## 60-Second Screening of Foods Using the Agilent QuickProbe GC/MS System

Agilent QuickProbe GC/MS System

**Authors**  
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and David Peterson  
Agilent Technologies, Inc.

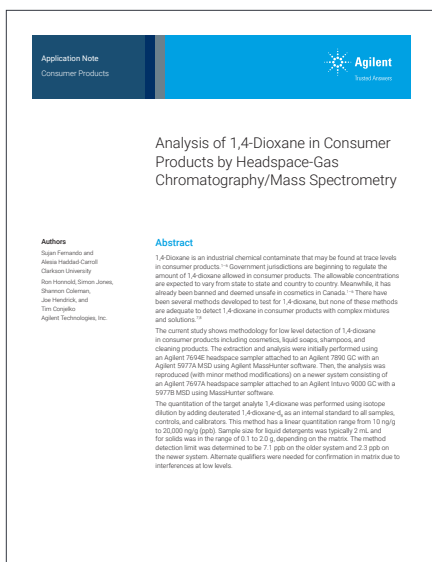
**Abstract**  
The Agilent QuickProbe, a direct insertion sampling device for GC/MS, was evaluated for the screening of nonextracted food samples. Foods analysis benefits from fast screening because it quickly identifies samples that are suspect and require further investigation.

## 60-Second Screening of Foods Using the Agilent QuickProbe GC/MS System

The Agilent QuickProbe, a direct insertion sampling device for GC/MS, was evaluated for the screening of nonextracted food samples. Foods analysis benefits from fast screening because it quickly identifies samples that are suspect and require further investigation.

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## Analysis of 1,4-Dioxane in Consumer Products by Headspace-GC/MS

1,4-Dioxane is an industrial chemical contaminant that may be found at trace levels in consumer products. 1–6 Government jurisdictions are beginning to regulate the amount of 1,4-dioxane allowed in consumer products. The allowable concentrations are expected to vary from state to state and country to country. Meanwhile, it has already been banned and deemed unsafe in cosmetics in Canada. 1–6 There have been several methods developed to test for 1,4-dioxane, but none of these methods are adequate to detect 1,4-dioxane in consumer products with complex mixtures and solutions. 7,8 The current study shows methodology for low level detection of 1,4-dioxane in consumer products including cosmetics, liquid soaps, shampoos, and cleaning products. The extraction and analysis were initially performed using an Agilent 7694E headspace sampler attached to an Agilent 7890 GC with an Agilent 5977A MSD using Agilent MassHunter software. Then, the analysis was reproduced (with minor method modifications) on a newer system consisting of an Agilent 7697A headspace sampler attached to an Agilent Intuvo 9000 GC with a 5977B MSD using MassHunter software. The quantitation of the target analyte 1,4-dioxane was performed using isotope dilution by adding deuterated 1,4-dioxane-d<sub>8</sub> as an internal standard to all samples, controls, and calibrators. This method has a linear quantitation range from 10 ng/g to 20,000 ng/g (ppb). Sample size for liquid detergents was typically 2 mL and for solids was in the range of 0.1 to 2.0 g, depending on the matrix. The method detection limit was determined to be 7.1 ppb on the older system and 2.3 ppb on the newer system. Alternate qualifiers were needed for confirmation in matrix due to interferences at low levels.

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Application Note  
Cannabis and Hemp  
Testing

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## Quantitation of Cannabinoids in Hemp Flower by Derivatization GC/MS

**Authors**  
Jennifer Sanderson and  
Jessica Westhead  
Agilent Technologies, Inc.

**Abstract**  
Total potency and total THC are two important calculations in the distinction of cannabis and hemp. Following U.S. Federal laws, hemp must be less than 0.3% total THC (by dry weight). In this application, offline derivatization of hemp sample extract was performed to determine total THC and quantitate an additional nine commonly analyzed cannabinoids by GC/MS. The derivatization allows for direct analysis and measurement of the thermally labile acids that are naturally occurring in hemp, which simplifies the determination of total THC.

## Quantitation of Cannabinoids in Hemp Flower by Derivatization GC/MS

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Cannabis & Hemp  
Testing

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## Terpenes Analysis in Cannabis Products by Liquid Injection using the Agilent Intuvo 9000/5977B GC/MS System

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Tary Heiser,<sup>1</sup> and  
Anthony Mecherson<sup>2</sup>  
<sup>1</sup>Agilent Technologies, Inc.  
<sup>2</sup>James Madison University  
School of Medicine

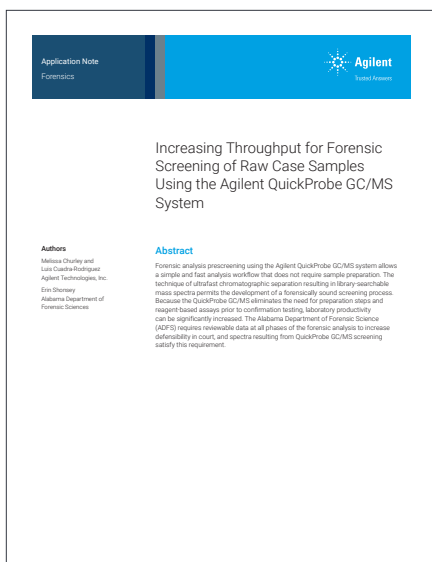
**Abstract**  
Terpenes are volatile and semivolatile chemicals that engender flavor and aroma organoleptic properties to cannabis and cannabinoid products. Cannabis growers and producers use terpene profiles to characterize specific strains of cannabis and hemp. To this end, a robust analytical method is necessary to chemically profile terpenes in cannabis and cannabinoid products prior to use in medicinal and recreational marijuana programs. Although regulatory agencies such as the California Bureau of Cannabis Control (BCC) do not regulate terpene content unless there is a specific label claim, terpenes are commonly analyzed in regulatory laboratories. The most common approach to terpene analysis in these laboratories is headspace gas chromatography (GC) with flame ionization detection (FID), mass spectrometry (MS), or both (FID/MS). Over the past several years, issues such as losses of sesquiterpenoids like  $\alpha$ -bisabolol have been observed in high-potency cannabis samples with headspace methodologies. This has led to a need for liquid injection terpene analysis. In this application note, we demonstrated a selective, sensitive, and robust method for the analysis of 40 chromatographically resolved terpenes common to Cannabis spp. using liquid injection GC/MS.

## Terpenes Analysis in Cannabis Products by Liquid Injection using the Agilent Intuvo 9000/5977B GC/MS System

In this application note, we demonstrated a selective, sensitive, and robust method for the analysis of 40 chromatographically resolved terpenes common to Cannabis spp. using liquid injection GC/MS.

This work developed and verified method parameters and outcomes for the liquid injection analysis of 40 chromatographically resolved terpenes in cannabis and in cannabinoid products using the Agilent Intuvo 9000/5977B GC/MS system. All data were matrix-matched and used an internal standard. This novel method used capillary flow technology to backflush matrix and other unwanted compounds before the next injection. CANNABIS

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## Increasing Throughput for Forensic Screening of Raw Case Samples Using the Agilent QuickProbe GC/MS System

Forensic analysis prescreening using the Agilent QuickProbe GC/MS system allows a simple and fast analysis workflow that does not require sample preparation. The technique of ultrafast chromatographic separation resulting in library-searchable mass spectra permits the development of a forensically sound screening process. Because the QuickProbe GC/MS eliminates the need for preparation steps and reagent-based assays prior to confirmation testing, laboratory productivity can be significantly increased. The Alabama Department of Forensic Science (ADFS) requires reviewable data at all phases of the forensic analysis to increase defensibility in court, and spectra resulting from QuickProbe GC/MS screening satisfy this requirement.

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Published in the USA, October 13, 2021  
5994-4252EN

