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# Identify More Pesticides Faster Using the Agilent High-Efficiency Source

## The Agilent Pesticide DRS Screening GC/MSD Analyzer

Application Note

Food Testing & Agriculture


### Authors

Melissa Churley and Bruce Quimby

Agilent Technologies, Inc.



The Agilent Pesticide DRS Screening GC/MSD Analyzer, based on the Agilent 7890 GC and the Agilent 5977B GC/MSD, delivers fast screening and quantitation of large numbers of pesticides and endocrine disruptors in a single run. Deconvolution reporting software and a retention-time-locked database of pesticides and endocrine disruptors accelerates reporting and increases the number of targets screened. When configured with the 5977B GC/MSD and a high-efficiency source (HES), the analyzer identifies a greater number of pesticides while reducing analysis time.


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## Identify More Pesticides with the High-Efficiency Source

Routine analysis of residues in environmental and food samples requires confident identification and low-level detection along with fast reporting times. The GC/MSD pesticide analyzer addresses each of these needs through Deconvolution Reporting Software (DRS), which uses the NIST AMDIS program [1]. The Pesticides and Endocrine Disruptors database [2] accelerates reporting time, and increases the number of targets screened. In addition, proprietary capillary flow technology column backflush shortens cycle time, reduces chemical background, and optimizes uptime.

The 5977B GC/MSD and HES improve screening capability by increasing the number of ions created in the source and transferred into the quadrupole analyzer. More ions deliver more signal and, thus, better sensitivity. This increase in response translates into more targets found during the screening process with good library matches. Positive identification in food samples at 10 ng/g detection levels is now possible using the full-scan mode.

We demonstrated this in a DRS analysis of tomato extract that was spiked with over 200 pesticides at concentrations of 10 and 100 ng/g. This equates to injection of 10 and 100 pg of each pesticide, respectively. At the 10 ng/g level, 38 target compounds were identified using the HES as compared to none using the extractor source. Almost twice as many targets were identified at the 100 ng/g level (Table 1). Figure 1 shows an example AMDIS analysis for the target flusilazole, including raw and extracted spectra with library matching for the component.

## Conclusions

The Agilent Pesticide DRS Screening GC/MSD Analyzer delivers faster and more accurate screening of pesticides when configured with the Agilent 5977B GC/MSD and HES. When combined with Deconvolution Reporting Software, positive identification in full-scan mode for many targets in food at a concentration of 10 ng/g is possible.

Table 1. Number of AMDIS targets identified in tomato spiked at 10 and 100 ng/g using the extractor source (EXR) and HES (MMF = 80). The amount of pesticide injected was 10 and 100 pg, respectively. At the 10 ng/g level, 38 target compounds were identified using the HES as compared to zero using the extractor source. Almost twice as many targets were identified at the 100 ng/g level (Table 1). The NIST hit number breakdown (distribution) is given for categories 1st, 2nd, and ≥3 hit. Identified targets that were not spiked into tomato but had an AMDIS match score ≥80 and NIST hit no. ≤ 3 are also listed. Tuning conditions for each source are in parentheses.

| | EXR (atune) | | HES (autotune) | |
|---|---|---|---|---|
| | **10 ng/g** | **100 ng/g** | **10 ng/g** | **100 ng/g** |
| Number of targets with AMDIS score ≥ 80 | 0 | 91 | 38 | 164 |
| **Distribution of NIST hits** | | | | |
| 1st hit | 0 | 63 | 26 | 144 |
| 2nd hit | 0 | 12 | 7 | 14 |
| ≥ 3rd hit | 0 | 16 | 5 | 6 |
| Not spiked, ≤ 3rd hit | 2* | 4** | 2* | 8*** |

* Diethyl phthalate and benzophenone

** Benzilamide, benzophenone, quintozene metabolite (pentachlorophenyl methyl sulfide), indoxacarb, and dioxacarb decomposition product [phenol, 2-(1,3-dioxolan-2-yl)-]

*** Diethyl phthalate, benzophenone, fonofos, phenol, phthalic acid, di(oct-3-yl) ester, phthalimide, quintozene metabolite (pentachlorophenyl methyl sulfide), indoxacarb, and dioxacarb decomposition product [phenol, 2-(1,3-dioxolan-2-yl)-]

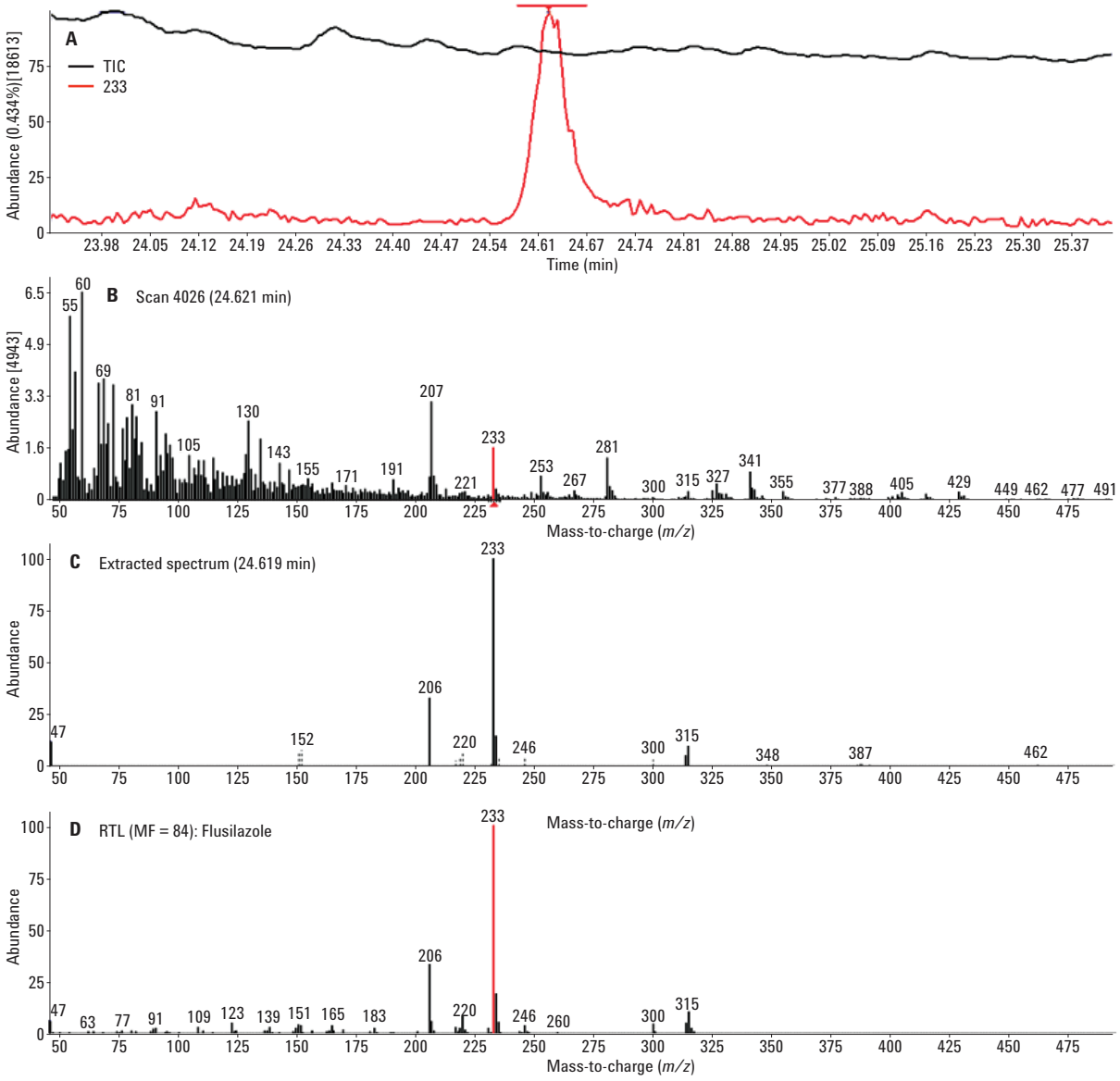


Figure 1. Analysis of 10 pg flusilazole in tomato using AMDIS. A) Overlay of extracted ion *m/z* 233 (red) and TIC (black); B) raw spectrum; C) extracted spectrum for the component; D) library spectrum, AMDIS match factor = 84. The reported NIST reverse match score is 73.

## Acknowledgement

The authors wish to thank Nathan Contino.

## References

1. Anon. NIST Standard Reference Database 1A, NIST/EPA/NIH Mass Spectral Library (NIST 14) and NIST Mass Spectral Search Program (Version 2.2), User's Guide. National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, MD, USA. http://www.nist.gov/srd/upload/NIST1aVer22Man.pdf
2. Wylie, P. L. *Screening for 926 Pesticides and Endocrine Disruptors by GC/MS with Deconvolution Reporting Software and a New Pesticide Library*; Application note, Agilent Technologies, Inc. Publication number 5989-5076EN, **2006**.

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# Improved Volatiles Analysis Using Static Headspace, the Agilent 5977B GC/MSD, and a High-efficiency Source

## Application Note

Environmental


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## Introduction

A preliminary assessment of environmental volatiles in water using static headspace analysis was performed using the Agilent 7697A Headspace Sampler, Agilent 7890B GC, and the Agilent 5977B GC/MSD system with high efficiency source (HES). The revolutionary design of the HES produces a higher ion current yield for many compounds (greater sensitivity), which allows flexible approaches to sample analysis such as lowering detection limits, reducing sample size, speeding up analysis, and so forth.

Volatile organic analysis (VOA) by static headspace is a widely applied technique, but it is also particularly challenging as the response factors for the many potential analytes vary widely. This application note presents a survey of select compounds of environmental interest as an indication of what may be achieved with the 5977B GC/MSD in this approach.

Analysis was performed in selected ion monitoring mode of a mixture of VOA compounds spiked into reverse osmosis (RO) water over a calibration range of 0.02–20 µg/L. Replicate injections were made at 0.04 µg/L to assess the method detection limits (MDL). A study of replicates of local tap water was used to demonstrate long-term stability for some naturally occurring compounds.

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# Materials and Methods

## Sample preparation

A 10 mL volume of water was added to each vial. Aliquots of Stock Standard (prepared in methanol) and Stock Internal Standard (prepared in methanol) were spiked into the solution and the vial sealed. Standards were prepared at 0.02, 0.05, 0.1, 0.2, 0.5, 1, 10, and 20 µg/L.

Table 1 presents a summary of the instrumental acquisition conditions.

*Table 1. Headspace Conditions and MS Conditions*

| Headspace parameters | Agilent 7697A Headspace Sampler |
|---|---|
| **Instrument settings** | |
| Loop size | 1 mL |
| Transfer line type | Fused Silica, deactivated (p/n160-2535-5) |
| Transfer line diameter | 0.53 mm |
| HSS-GC coupling | Transfer line interface (G3520A) |
| Carrier control | GC Instrument |
| Pressurization gas | Helium |
| Vial standby flow | 20 mL/min |
| **Temperature settings** | |
| Oven temperature | 75 °C |
| Loop temperature | 75 °C |
| Transfer line temperature | 110 °C |
| Transfer line interface (Aux1) | 115 °C |
| **Timing settings** | |
| Vial equilibration time | 12 minutes |
| Injection duration | 0.3 minutes |
| GC cycle time | 30 minutes |
| **Vial and loop settings** | |
| Vial size | 20 mL |
| Vial shaking | Level 7 |
| Fill pressure | 10 psi |
| Fill time | 0.2 minutes |
| Loop ramp rate | 20 psi/min |
| Loop final pressure | 7 psi |
| Loop equilibration time | 0.01 minutes |
| Post injection purge | 100 mL/min for 2 minutes |
| Leak check | Default, 0.2 mL/min |
| Mode | Single Extraction |

| GC parameters | Agilent 7890B GC |
|---|---|
| **Inlet** | |
| Inlet type | Split/Splitless Inlet (SSL) |
| Mode | Split |
| Inlet liner | Straight, 2 mm id, 250 µL (p/n 5181-8818) |
| Heater | 125 °C |
| Column flow | 1.5 mL/min constant flow |
| Total flow | 25 mL/min |
| Septum purge flow | 1.0 mL/min |
| Gas saver | OFF |
| Split ratio | 15:1 |
| Split flow | 22.5 mL/min |
| **Oven** | |
| Column | Agilent VF-624 MS |
| Column dimensions | 60 m × 0.25 mm, 1.4 µm |
| Equilibration time | 0.25 minutes |
| Temperature program | 32 °C (2 minutes),<br>12 °C/min to 220 °C (5 minutes) |
| **Mass Selective Detector parameters Agilent 5977B** | |
| Type | High Efficiency Source (HES EI) |
| Source temperature | 300 °C |
| Quad temperature | 150 °C |
| Transfer line temperature | 280 °C |
| Tune file | HES Auto Tune (HES_Atune.u) |
| Acquisition type | SIM (see Table 2) |
| Solvent delay | 3.95 minutes |
| Gain factor | 3 |

## Results and Discussion

Table 2 gives the results of an MDL study performed at 0.04 µg/L, with nine replicate analyses. Note that all MDLs are below 0.025 µg/L or 25 ppt with the exception of two compounds, which have MDLs below 30 ppt. The majority of compounds produce MDLs below 0.015 µg/L, including some compounds with relatively low response.

*Table 2. A Summary of Selected Compounds Acquired Stating Retention Times in Minutes, Target Quantitation Ion (SIM), and MDL Calculated for Nine Replicates at 0.04 µg/L*

| Name | RT | Quant ion | MDL | Name | RT | Quant ion | MDL |
|---|---|---|---|---|---|---|---|
| Vinyl chloride | 4.934 | 62 | 0.004 | 1,2-Dibromoethane | 13.427 | 106.9 | 0.006 |
| Bromomethane | 5.611 | 93.9 | 0.003 | Chlorobenzene | 13.969 | 112 | 0.015 |
| Chloroethane | 5.806 | 64 | 0.003 | Ethylbenzene | 14.03 | 91 | 0.014 |
| 1,1-Dichloroethene | 7.007 | 95.9 | 0.008 | 1,1,1,2-Tetrachloroethane | 14.049 | 130.9 | 0.005 |
| *trans*-1,2-Dichloroethene | 8.007 | 95.9 | 0.009 | *o*-Xylene | 14.664 | 91 | 0.018 |
| 1,1-Dichloroethane | 8.554 | 63 | 0.004 | Styrene | 14.683 | 104 | 0.015 |
| *cis*-1,2-Dichloroethene | 9.19 | 95.9 | 0.011 | Bromoform | 14.975 | 170.8 | 0.006 |
| 2,2-Dichloropropane | 9.208 | 77 | 0.013 | 1,1,2,2-Tetrachloroethane | 15.45 | 82.9 | 0.041 |
| Bromochloromethane | 9.47 | 127.8 | 0.004 | 1,2,3-Trichloropropane | 15.567 | 110 | 0.007 |
| 1,1,1-Trichloroethane | 9.769 | 96.9 | 0.005 | Bromobenzene | 15.573 | 155.9 | 0.017 |
| 1,1-Dichloro-1-propene | 9.921 | 75 | 0.012 | *n*-Propylbenzene | 15.63 | 91 | 0.017 |
| Carbon tetrachloride | 9.94 | 116.9 | 0.003 | 2-Chlorotoluene | 15.768 | 91 | 0.016 |
| Benzene * (blank issue) | 10.165 | 78 | 0.009 | 1,3,5-Trimethylbenzene | 15.84 | 105 | 0.018 |
| 1,2-Dichloroethane | 10.202 | 62 | 0.006 | 4-Chlorotoluene | 15.914 | 91 | 0.018 |
| Trichloroethene | 10.848 | 129.9 | 0.009 | *tert*-Butylbenzene | 16.225 | 134 | 0.017 |
| 1,2-Dichloropropane | 11.165 | 63 | 0.005 | *sec*-Butylbenzene | 16.499 | 105 | 0.016 |
| Dibromomethane | 11.275 | 173.8 | 0.006 | 4-Isopropyltoluene | 16.67 | 119 | 0.017 |
| Bromodichloromethane | 11.421 | 82.9 | 0.005 | 1,3-Dichlorobenzene | 16.719 | 145.9 | 0.020 |
| *cis*-1,3-Dichloropropene | 11.89 | 75 | 0.014 | 1,4-Dichlorobenzene | 16.841 | 145.9 | 0.023 |
| *trans*-1,3-Dichloropropene | 12.506 | 75 | 0.013 | *n*-Butylbenzene | 17.194 | 134 | 0.020 |
| 1,1,2-Trichloroethane | 12.762 | 96.9 | 0.011 | 1,2-Dichlorobenzene | 17.316 | 145.9 | 0.021 |
| Tetrachloroethene | 12.884 | 163.8 | 0.009 | 1,2-Dibromo-3-chloropropane | 18.334 | 154.9 | 0.010 |
| 1,3-Dichloropropane | 12.963 | 76 | 0.009 | 1,2,4-Trichlorobenzene | 19.493 | 179.9 | 0.028 |
| Dibromochloromethane | 13.238 | 126.8 | 0.004 | Hexachlorobutadiene | 19.651 | 224.8 | 0.006 |

*Blanks showed some low-level contamination for benzene.

Figure 1 shows an example of the linearity achieved over the concentration range 0.02 to 20 µg/L for several representative compounds. Figures 2 and 3 show the system stability for a few compounds of interest. It is noteworthy that these preliminary results do not benefit from the use of internal standard corrections (that is, external standard calibration, and so forth), which can be expected to greatly improve all aspects of the analysis.

## Conclusions

These preliminary results suggest a significant improvement in detection limits is possible in VOA applications through the HES of the Agilent 5977B GC/MSD. The signal improvement provided is not complicated by interferences, and results in clear enhancements in detection.

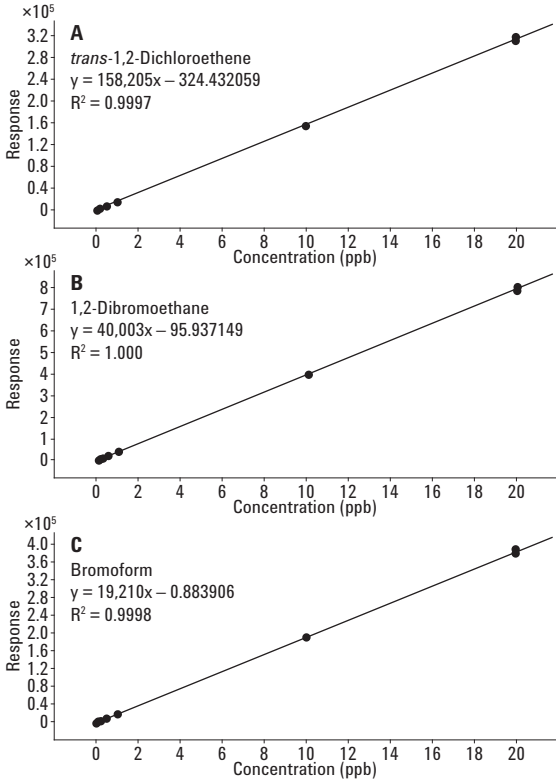


*Figure 1. Linearity to 20 µg/L (external standard) for trans-1,2-dichloroethene (A), 1,2-dibromoethane (B), and bromoform (C).*

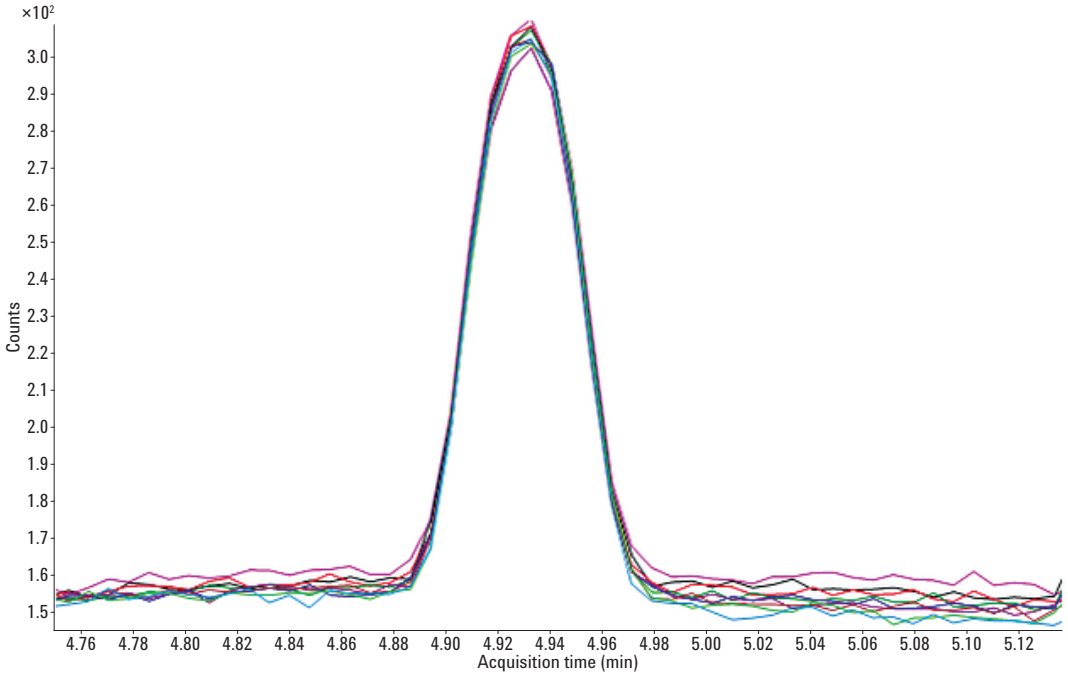


*Figure 2. Overlay of the EIC for nine replicate injections of vinyl chloride at 0.04 µg/L.*

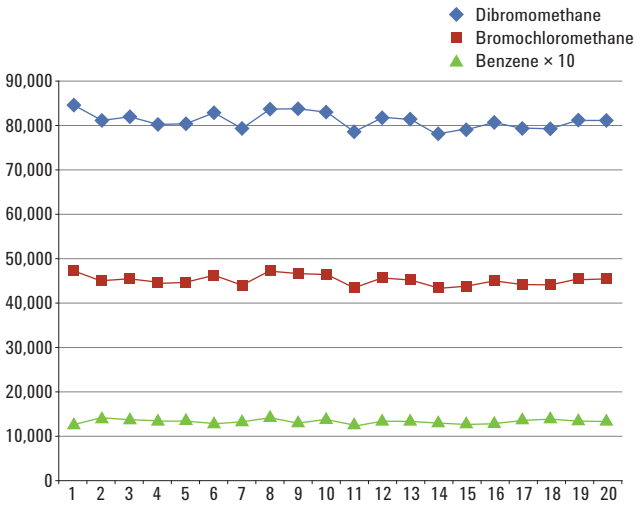


*Figure 3.* *Response as peak areas for replicate injections (n = 20) of selected compounds showing system stability in time: benzene ~0.01 ppb, dibromomethane 1.5 ppb, and bromochloromethane 0.8 ppb.*

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# The Agilent 5977B GC/MSD and High Efficiency Source (HES) Lowers Detection for Semivolatile Compounds

Application Note


## Authors

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## Abstract

The enhanced signal produced by the High Efficiency Source (HES) of the Agilent 5977B GC/MSD allows for flexibility in analytical approach. To assist setting expectations for strategies in semivolatile organic compound analysis, this application note suggests preliminary instrument detection limits for a range of analytes of interest. The results show that picogram or sub-picogram detection in scan mode is possible for a wide range of compounds.



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## Introduction

Semivolatile organic compounds (SVOCs) are a broad class of environmentally significant contaminants of global interest. These compounds are found on a variety of target analyte lists in GC/MS methods such as the USEPA 8270 and 525 methods, and comparable methods elsewhere. Although listed as targets and appropriate to selected ion monitoring (SIM) in GC/MS analysis, surveying samples by scanning GC/MS provides advantages such as full scan spectra for compound confirmation, tentatively identifying unexpected unknowns in samples that would escape SIM. In the past, scan sensitivity was borderline or insufficient when compared to SIM or the required detection limits. The High Efficiency Source (HES) of the Agilent 5977B GC/MSD represents a revolution in ion source design with greatly enhanced sensitivity that can be exploited to produce scan detection limits for SVOCs that were formerly only approached by SIM. This application note provides preliminary results for instrument detection limits (IDLs) for a few SVOCs across the classes of compounds typical to this analysis.

## Experimental

Many configurations and approaches are used for SVOCs, but common to all is a 5-phase column with a thickness appropriate to the loaded range. Because this was a survey of compounds and a preliminary investigation, an Agilent J&W DB-UI 8270D Ultra Inert GC column (0.25 mm × 30 m, 0.5 µm) was used, as is common in this analysis. This choice would favor higher amounts on-column, although the data suggest that a better approach would be based on a thinner film. Standards were prepared in dichloromethane, and 0.5 µL was injected using a 5-µL syringe in pressure-pulsed splitless mode into a double-taper liner. Replicate injections of a 5 ng/mL standard were used to determine an IDL for each compound. The Agilent 7890B GC with a 5977B HES was operated in scan mode from 50 to 550 u (sampling = 4), and a very low gain factor (0.1) to be pertinent to the desire for a working concentration range. This is essentially a standard configuration, operated in a conservative mode to survey possible IDLs.

## Results and Discussion

Estimated IDLs for the scan data were calculated using the external standard method, and were based on eight consecutive injections of 12 total injections. The average of five IDL determinations was reported as the IDL. As can be seen in Table 1, sub-picogram scan detection is common, with a few compounds showing picogram levels due primarily to lowered compound target ion response. Compound chromatography also played a role in some cases (for example, benzo[b]- and [k]fluoranthene, and so forth).

## Conclusions

Clearly, compound detection in scan mode is now able to discern amounts previously attained only in SIM mode. This advantage allows several analytical strategies to be explored and applied. The shoot less and get more approach means applying split injections with accelerated run times if high concentration levels wish to be maintained. Shooting less sample would also put less matrix in the liner, column, and so forth, and allow the analyst to get more runs before servicing is required. The prep less and save more approach means processing less sample. This would save time and costs not only in collection and transport, but in solvent use and disposal. These dramatically lowered scan IDLs also suggest that SIM IDLs will be enhanced, and so a combination of both strategies is possible to result in the most time and cost effective analysis possible.

*Table 1. Agilent 5977B Scan Mode Instrument Detection Limits for SVOCs*

| Compound | Scan IDL (pg) | Compound | Scan IDL (pg) |
|---|---|---|---|
| Dimethyl phthalate | 0.4 | *o*-Cresol | 2.5 |
| Diethyl phthalate | 1.1 | *p*-Cresol | 2.6 |
| Di-*n*-butyl phthalate | 0.9 | 2,4-Dimethylphenol | 0.5 |
| Butyl benzyl phthalate | 5.7 | 2,4-Dichlorophenol | 0.5 |
| *Bis*(2-ethylhexyl) phthalate | 0.5 | 4-Chloro-3-methylphenol | 1.6 |
| Di-*n*-octyl phthalate | 2.1 | 2,4,6-Trichlorophenol | 9.1 |
| 1,3-Dichlorobenzene | 0.3 | 2,4,5-Trichlorophenol | 3.4 |
| 1,4-Dichlorobenzene | 0.3 | Naphthalene | 0.2 |
| Benzyl alcohol | 3.1 | 2-Methylnaphthalene | 0.4 |
| 1,2-Dichlorobenzene | 0.3 | 2-Chloronaphthalene | 0.3 |
| 1,2,4-Trichlorobenzene | 0.3 | Acenapthylene | 0.4 |
| Azobenzene | 0.6 | Acenaphthene | 0.8 |
| Hexachlorobenzene | 2.1 | Dibenzofuran | 0.3 |
| *Bis*(2-chloroethyl) ether | 1.4 | Fluorene | 0.4 |
| *Bis*(2-chloro-1-methylethyl) ether | 0.6 | Phenanthrene | 0.2 |
| Aniline | 0.4 | Anthracene | 0.3 |
| N Nitroso-di-*n*-propylamine | 2.2 | Fluoranthene | 0.8 |
| Nitrobenzene | 0.4 | Pyrene | 0.8 |
| 4-Chloroaniline | 0.9 | Benz[a]anthracene | 0.4 |
| 2-Nitroaniline | 1.3 | Chrysene | 0.3 |
| 2,6-Dinitrotoluene | 1 | Benzo[b]fluoranthene | 0.7 |
| 3-Nitroaniline | 2.8 | Benzo[k]fluoranthene | 0.7 |
| 2,4-Dinitrotoluene | 1.4 | Benzo[a]pyrene | 0.9 |
| 4-Nitroaniline | 3.8 | Indeno[1,2,3-cd]pyrene | 0.7 |
| Diphenylamine | 0.6 | Dibenz[a,h]anthracene | 1.3 |
| Phenol | 0.60 | Benzo[g,h,i]perylene | 0.6 |
| 2-Chlorophenol | 0.5 | | |

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**Agilent Technologies**

# Screen More Drugs with the Agilent GC/MS Toxicology Analyzer with a High Efficiency Source

Application Note

Forensic Toxicology


## Authors

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Luis Cuadra-Rodriguez

Agilent Technologies, Inc.


## Introduction


Broad-range screening for drugs in biological samples requires full-spectrum identification confirmation for an unlimited number of targets, as well as spectral identification of nontargets. The Agilent GC/MS Toxicology Analyzer uses Deconvolution Reporting Software (DRS), the Forensic Toxicology Database Library and, when configured with the Agilent 5977B Mass Selective Detector, a high efficiency source (HES). In combination, these technologies screen a greater number of targets at low concentrations while reducing analysis time.


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## Screen at Lower Concentrations with the High Efficiency Source

Improved screening capability with the HES was demonstrated using human serum, as seen in Table 1. Negative serum, 2 mL, was extracted using the Agilent Bond Elut Certify general drug screen method M2721 [1]. The extract was reconstituted in 0.1 mL methanol and spiked with the GC/MS Toxicology Checkout Mixture (p/n 5190-0471) to yield 10 to 1,000 ng/mL (in vial) of underivatized standards.

Table 1. Lowest injected amount of drugs in spiked serum extract detected with AMDIS using a minimum match factor of 75.

| | HES (HES autotune) | | | Extractor (etune) | | |
|---|---|---|---|---|---|---|
| | Minimum amount injected (pg) AMDIS score > 75 | AMDIS Score | Equivalent concentration in serum (ng/mL)* | Minimum amount injected (pg) AMDIS score > 75 | AMDIS Score | Equivalent concentration in serum (ng/mL)* |
| Amphetamine | 500 | 94 | 25 | 500 | 75 | 25 |
| Nicotine | 50 | 92 | 2.5 | 50 | 81 | 2.5 |
| MDA | 500 | 77 | 25 | 500 | 76 | 25 |
| MDMA | 500 | 85 | 25 | 500 | 83 | 25 |
| MDEA | 10 | 76 | 0.5 | 500 | 97 | 25 |
| Meperidine | 10 | 85 | 0.5 | 50 | 85 | 2.5 |
| Phencyclidine | 50 | 83 | 2.5 | 500 | 90 | 25 |
| Methadone | 50 | 87 | 2.5 | 500 | 89 | 25 |
| Cocaine | 50 | 77 | 2.5 | 500 | 94 | 25 |
| SKF-525a | 50 | 77 | 2.5 | 100 | 81 | 5 |
| Codeine | 100 | 88 | 5 | 500 | 90 | 25 |
| Diazepam | 50 | 90 | 2.5 | 50 | 81 | 2.5 |
| Hydrocodone | 100 | 91 | 5 | 500 | 90 | 25 |
| Tetrahydrocannabinol | 50 | 75 | 2.5 | 100 | 78 | 5 |
| Oxycodone | 50 | 80 | 2.5 | 500 | 83 | 25 |
| Flunitrazepam | 500 | 88 | 25 | 500 | 75 | 25 |
| Diacetylmorphine | 100 | 79 | 5 | 1,000 | 83 | 25 |
| Fentanyl | 50 | 85 | 2.5 | 50 | 77 | 2.5 |
| Alprazolam | 100 | 76 | 5 | 1,000 | 85 | 50 |
| Verapamil | 50 | 84 | 2.5 | 500 | 90 | 25 |
| Strychnine | 500 | 86 | 25 | 500 | 77 | 25 |
| Trazodone** | > 1,000 | (71) | > 50 | > 1,000 | (68) | > 50 |

Drugs found at lower concentrations using the HES versus the extractor source are highlighted. Tuning conditions are in parentheses.

* Assumes 100% recovery from a 2 mL serum sample, reconstitution of extract in 0.1 mL and 1 µL injected.

** The amount of injected trazodone required to achieve a score of 75 exceeds 1,000 pg.

The benzodiazepines oxazepam, lorazepam, temazepam, nitrazepam, and clonazepam were not found at 1,000 pg.

The synthetic opioid fentanyl was added to the checkout mixture. Drug target compounds screened in serum, such as methadone, cocaine, hydrocodone, THC and others, can now be positively identified using full-scan mode at lower concentration (for example, 5 ng/mL for hydrocodone). The HES maximizes the number of ions that are created in the source and transferred into the quadrupole analyzer, which equates to more signal, and thus better sensitivity (Figure 1). This increase in response translates into more drug targets found during the screening process with good library matches.

## NIST Searchable Spectra with Isomer Differentiation

The forensic chemist is required to identify the drug of abuse with the highest degree of scientific certainty. To some extent, the issue of cocaine isomer determination has reinforced the requirement for methods of high specificity [2]. The 5977B GC/MSD with HES allows for the strict spectral integrity that is required to differentiate between cocaine isomers and obtain NIST searchable spectra for detected drugs (Figure 2).

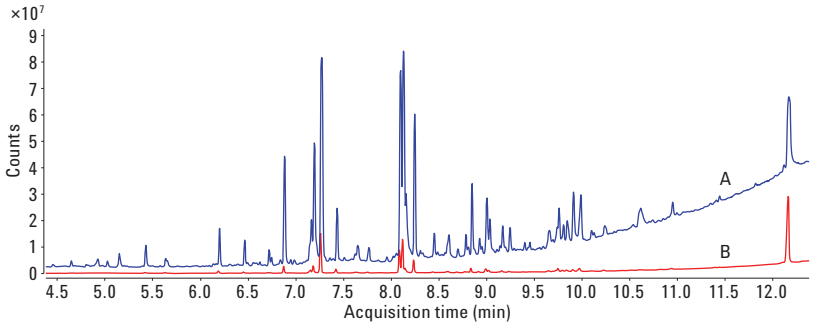


Figure 1. Overlay of TICs of 500 pg standard in serum using A) HES autotune, and B) extractor source etune.

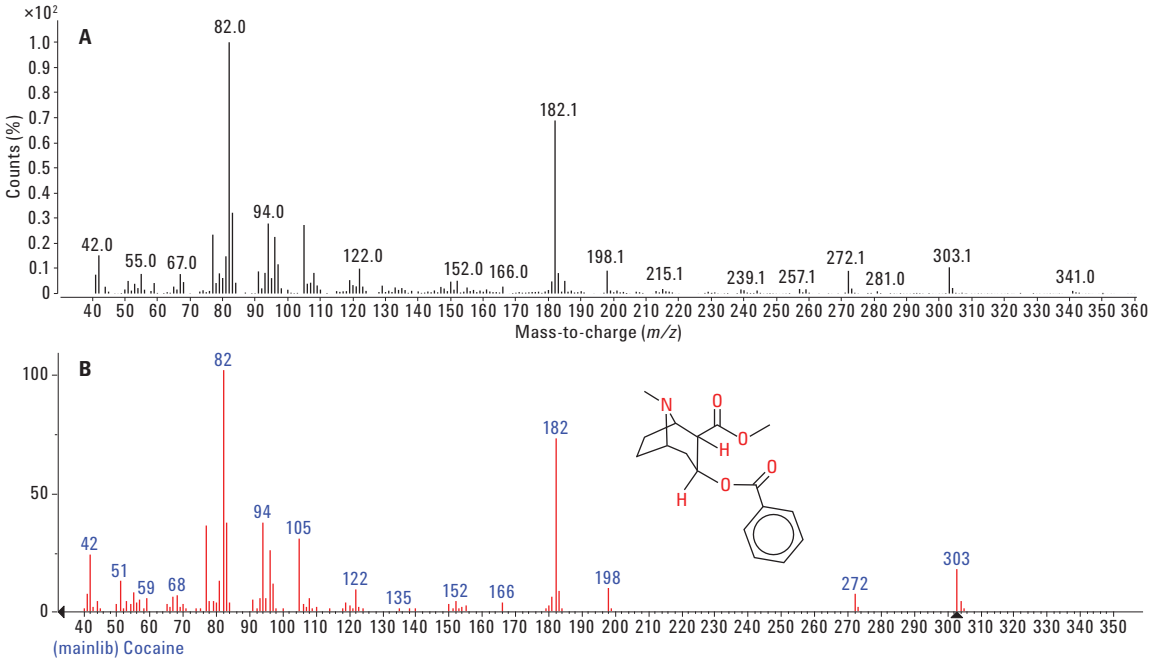


Figure 2. Mass spectrum of 100 pg cocaine spiked in serum (A) compared with NIST spectrum (B). Cocaine is the first hit in NIST. The match factor is 810 (good) [3], and is differentiated from pseudococaine, which has a match factor of 788 (fair). The equivalent concentration is 5 ng/mL based on complete recovery. Excellent NIST library matches (≥900) for cocaine as a first hit are returned at concentrations above this level.

## Conclusions

The high efficiency source of the Agilent 5977B GC/MSD greatly enhances the signal of drug targets. Resulting spectra are classical and NIST searchable. When combined with Deconvolution Reporting Software, detection levels during screen analysis approach those using SIM mode with derivatization.

## Acknowledgements

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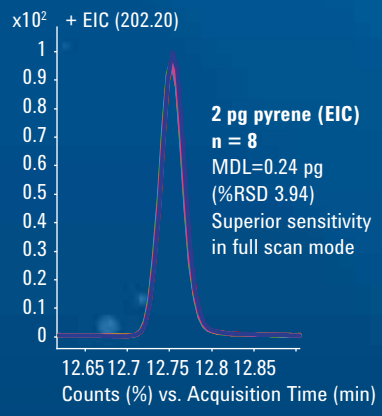


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