Application Note

Drug Screening

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Forensic Analysis of Drugs of Abuse With the Agilent 8890 GC

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Abstract

This Application Note presents a method for the accurate identification of commonly screened drugs. The workflow comprised an Agilent 8890 GC, an Agilent 5977A MSD, an Agilent 7693A automated liquid sampler, and an Agilent J&W DB-5ms Ultra Inert, high-efficiency column. This method enables superior resolution of common drugs, and is highly reproducible.

Introduction

Analytical confirmation of narcotic substances requires a high level of data confidence and an extremely low margin of error. The presence of both street and prescription drugs of abuse in forensic samples are often confirmed using gas chromatography (GC) techniques in tandem with single quadrupole mass spectrometers (MS), also known as GC/MS. Seized drug analyses often do not involve quantitation, but rely on high-quality mass spectra resulting in a library match. To best match peak spectra, chromatography must be optimized, and systems must be maintained routinely. Agilent has many publications that have demonstrated successful analysis conditions using standard MS dimension columns, both in drug confiscations[1] and forensics or clinical studies[2,3]. In this Application Note, a high-efficiency column was selected for evaluation on the 8890 GC. High-efficiency columns require excellent pressure control due to their smaller inner diameter and higher restriction. The sixth-generation EPC, available on the 8890 GC, demonstrates exceptional control and precision when supplying pressures needed for GC/MS flows of less than 2 mL/min. The 8890 GC can implement and log user-configurable maintenance counters, diagnostic events, and other system parameters, lending another layer of confidence to the collected data. The 8890 GC also incorporates features of previous generation Agilent GCs, such as retention time locking, inert-treated consumables, and a familiar software interface.

Experimental

The workflow featured an 8890 GC including a split/splitless inlet, together with a 5977 GC/MSD and a 7693A automated liquid sampler (ALS) system. Agilent MassHunter GC/MS software was used to acquire and process data. The 5 µg/mL standard used in testing was an Agilent GC/MS forensic toxicology checkout mixture (p/n 5190-0471). This 28-compound mixture contains amphetamines, opiates, benzodiazepines, and other commonly screened drugs. A high-efficiency, Ultra Inert (UI) column was used to screen the analytes, and a UI low pressure drop liner was used to minimize inlet discrimination[1]. In general, high-efficiency columns require much less sample to be injected because they are easily overloaded. The simplest way to implement this is to apply a higher split ratio to the method. Alternatively, further dilution of samples can be beneficial when separating on a high-efficiency column.

Table 1. Consumables used for drugs of abuse data acquisition.

Part	Description
ALS syringe	Agilent ALS syringe, 10 µL, Blue Line (p/n G4513-80204)
Vials, MS certified	5182-0716
Septa	Advanced Green (p/n 5183-4761)
Liner	Inlet liner, UI, low pressure drop (p/n 5190-2295)
Column	Agilent J&W DB-5ms UI, 20 m × 180 µm, 0.18 µm (p/n 121-5522UI)
Extractor lens	9 mm (p/n G3870-20449)

Table 2. Method conditions for drugs of abuse on the 8890 GC.

Parameter	Value
Syringe size	10 µL
Injection volume	1 µL
Inlet type	Split/splitless
Inlet mode	Split
Inlet temperature	250 °C
Split flow	30 mL/min
Split ratio	20:1
Gas saver time	Off
Septum purge mode	Standard
Septum purge	3 mL/min
Carrier gas	Helium
Column	J&W DB-5ms UI, 20 m × 180 µm, 0.18 µm
Column flow	1.5 mL/min
Oven equilibration	1 min
Oven program	95 °C, 20 °C/min to 300 °C, hold 3.5 minutes
GC run time	13.75 minutes
MSD transfer line	280 °C

Table 3. Analysis conditions for drugs of abuse on a 5977 GC/MSD (extractor).

5977 GC/MSD conditions	
Parameter	Value
Source	Extractor, 9 mm lens
High vacuum pump	Turbo
Mode	Scan
Range	*m/z* 40 to 500
Tune algorithm	Etune
Source temperature	250 °C
Quadrupole temperature	175 °C

Results and discussion

During the method optimization, overloading was most noticeable with the amphetamine compounds, so the split ratio was optimized around their peak shape. There was a trade-off in detection of lorazepam, oxazepam, and trazadone, as these compounds already produce a low response when compared to other drugs in the mix. Usually, confirmations are run at relatively high concentrations, resulting in higher split ratios than displayed with this midrange concentration sample. There was enough response with a 20:1 split to successfully integrate peaks, extract spectra, and generate a high-quality match for lorazepam, oxazepam, and trazadone under these conditions, but their lower response warrants an explanation.

Excellent resolution was attained with the high-efficiency column. All 28 peaks were easily identifiable, and when the resulting spectra were processed using a NIST library, every peak was properly identified as the top match, returning excellent match scores. Implementing background subtraction, especially at the higher temperature, significantly improved the match quality. Figure 1 shows a representative total ion chromatogram (TIC), and Table 4 presents a compound summary.

Reproducibility studies were executed, and the retention time relative standard deviation (RSD) for all compounds ranged from 0 to 0.08 %, excluding d-amphetamine and phentermine. Most compounds displayed a retention RSD of 0.03 % for 25 replicate injections.

These two compounds were removed from statistical analysis due to their limited interaction with the phases, as they elute immediately following the solvent tail. Larger split ratios improve the reproducibility of the early amphetamines, but sacrifice the response of oxazepam, lorazepam, and trazodone. Work has been done to improve response of these compounds[4] using traditional GC/MS columns with 250 µm diameters. These practices are helpful for high-efficiency columns as well.

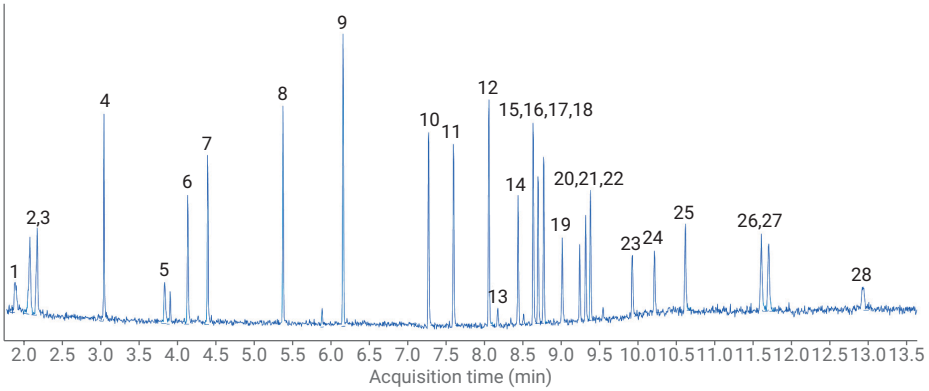

Figure 1. TIC of mixed drug injection (20:1 split of 5 µg/mL, full scan range *m/z* 40 to 500).

Table 4. Compound summary.

Index	Compound	RT
1	d-Amphetamine	1.85
2	Phentermine	2.05
3	Methamphetamine	2.14
4	Nicotine	3.03
5	MDA	3.83
6	MDMA	4.14
7	MDEA	4.40
8	Meperidine	5.40
9	Phencyclidine	6.19
10	Methadone	7.35
11	Cocaine	7.68
12	Proadifen	8.14
13	Oxazepam	8.26
14	Codeine	8.53

Index	Compound	RT
15	Lorazepam	8.61
16	Diazepam	8.73
17	Hydrocodone	8.79
18	THC	8.86
19	Oxycodone	9.10
20	Temazepam	9.33
21	Flunitrazepam	9.41
22	Heroin	9.47
23	Nitrazepam	10.02
24	Clonazepam	10.31
25	Alprazolam	10.72
26	Verapamil	11.71
27	Strychnine	11.81
28	Trazodone	13.04

Conclusion

This method produced excellent resolution between the analyzed drugs, with all 28 components accurately identified. Retention times were also reproducible over 25 injections, with RSDs of 0.03 % determined for most compounds. The 8890 GC split/splitless inlet offers precise thermal and pneumatic control to generate high-quality data using high-efficiency GC and GC/MS columns. Column overloading is the primary concern when high-efficiency columns are implemented, so they are an ideal option for:

- Applications where high split ratios are possible
- Injection volumes can be decreased
- Compounds of interest are impurities, or known to be low in concentration

Often, speed advantages are a result of migration to high-efficiency columns, but injection amount, calibration range, and desired resolution must be considered in the adoption. The Agilent Method Translator[5] is an ideal tool to assist in the evaluation and comparison of gas chromatography setpoints, and includes a calculation of column capacity. The 8890 GC is a next-generation mainframe offering smart technology features, while assuring compatibility with all Agilent consumables proven in a workflow.

References

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5. Agilent YouTube channel, Method Translator Education Series, https://www.youtube.com/watch?v=Q-359Q4qD-Q.

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