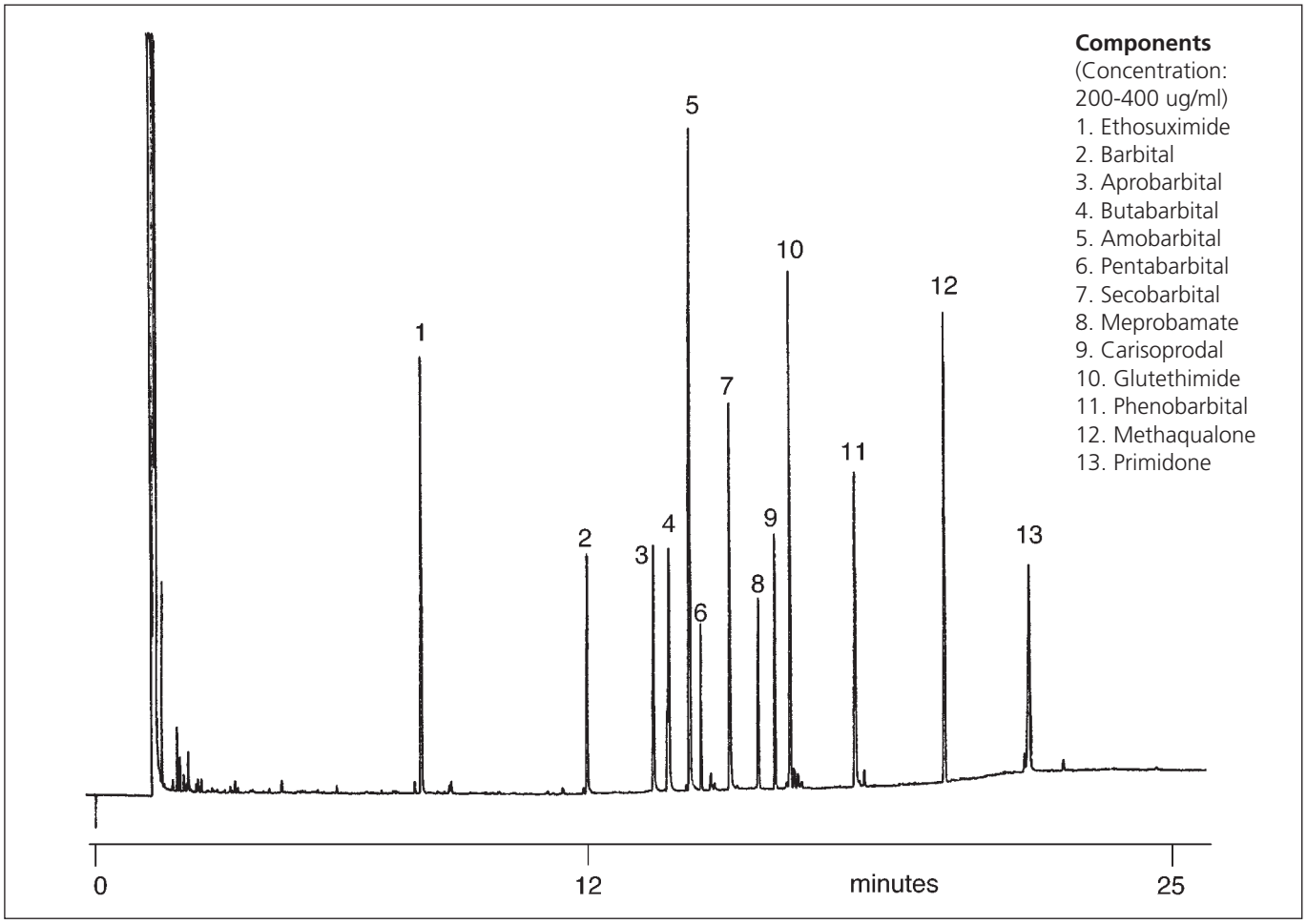
ANALYSIS OF ACID/NEUTRAL DRUGS ON BPX35

Column Part No.:	**054711**	Final Temp.:	300 °C, 5 min
Phase:	BPX35, 0.25 µm	Carrier Gas:	He, 150 kpa
Column:	25 m x 0.22 mm ID	Injection Mode:	Split, (20:1)
Initial Temp.:	100 °C, 1 min	Detector:	FID, 380 °C
Rate:	10 °C/min		

Note: BPX35 is a low bleed column with a maximum temperature of 360 °C. Very compatible with GC/MS systems..



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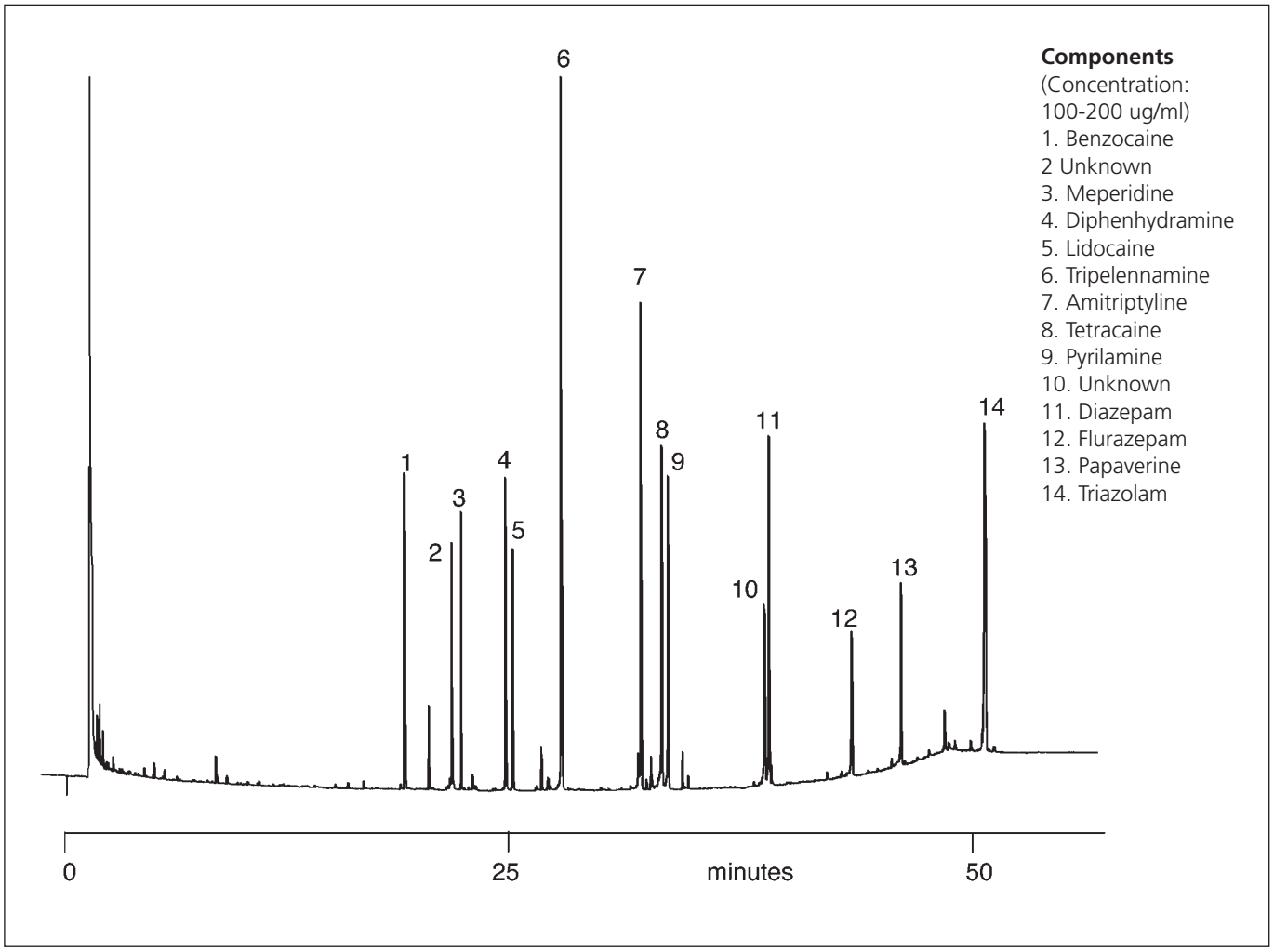
ANALYSIS OF BASIC DRUGS ON BPX35

Column Part No.:	**054711**	Final Temp.:	325 °C, 5 min
Phase:	BPX35, 0.25 µm	Carrier Gas:	Helium 150 kpa
Column:	25 m x 0.22 mm ID	Injection Mode:	Split, 0.5 µL (20:1)
Initial Temp.:	100 °C	Detector:	FID, 380 °C
Rate:	5 °C/min		



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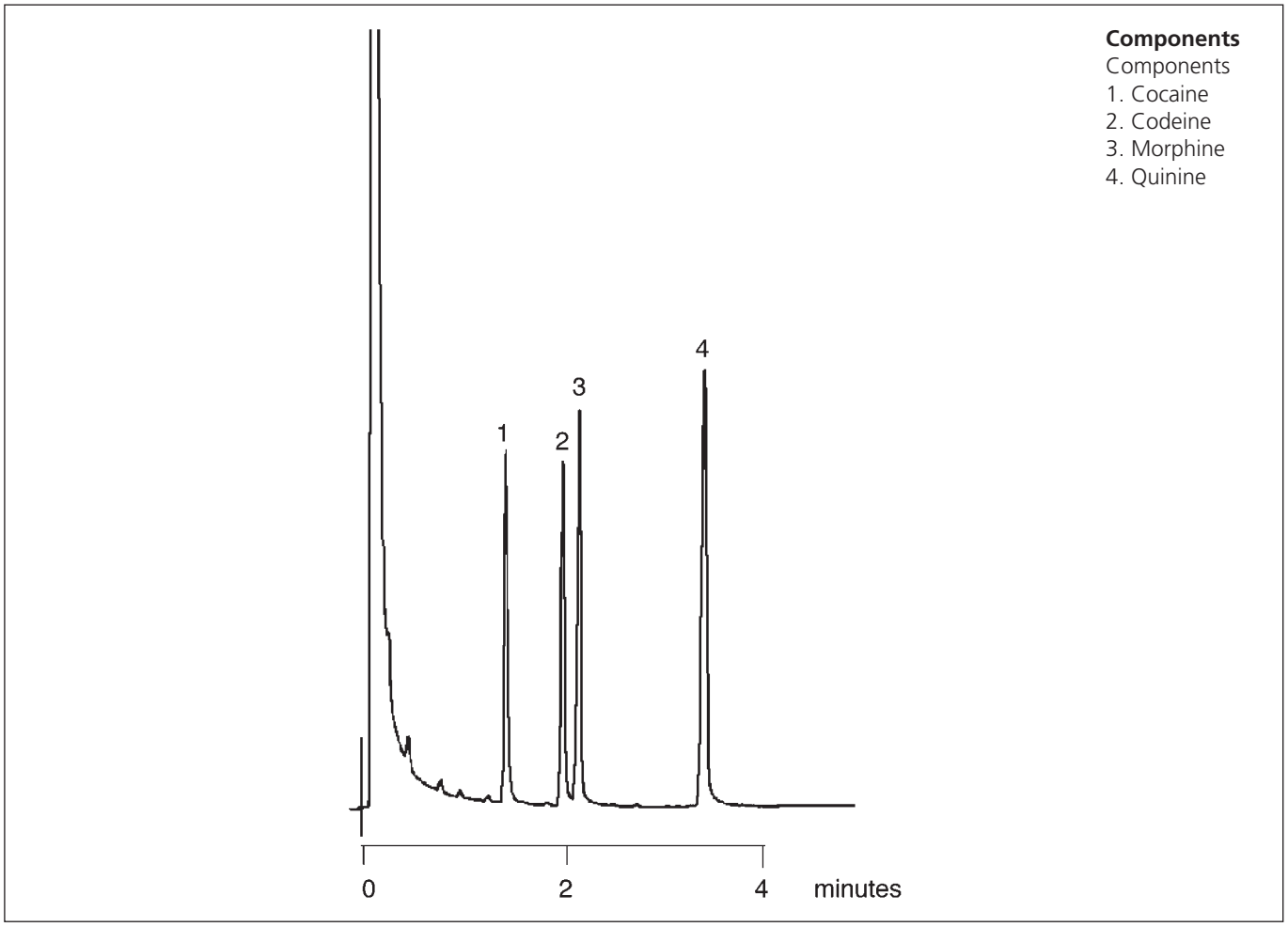
ANALYSIS OF ALKALOIDS ON BP5

Column Part No.:	**054198**	Final Temp:	300 °C, 0 min
Phase:	BP5, 1.0 µm film	Detector:	FID
Column:	25 m x 0.53 mm ID	Sensitivity:	128 x 10^{-12} AFS
Initial Temp.:	200 °C, 0 min	Injection Mode:	Split
Rate:	25 °C/min		

Note: A 0.53 mm ID column can be used to screen samples rapidly.



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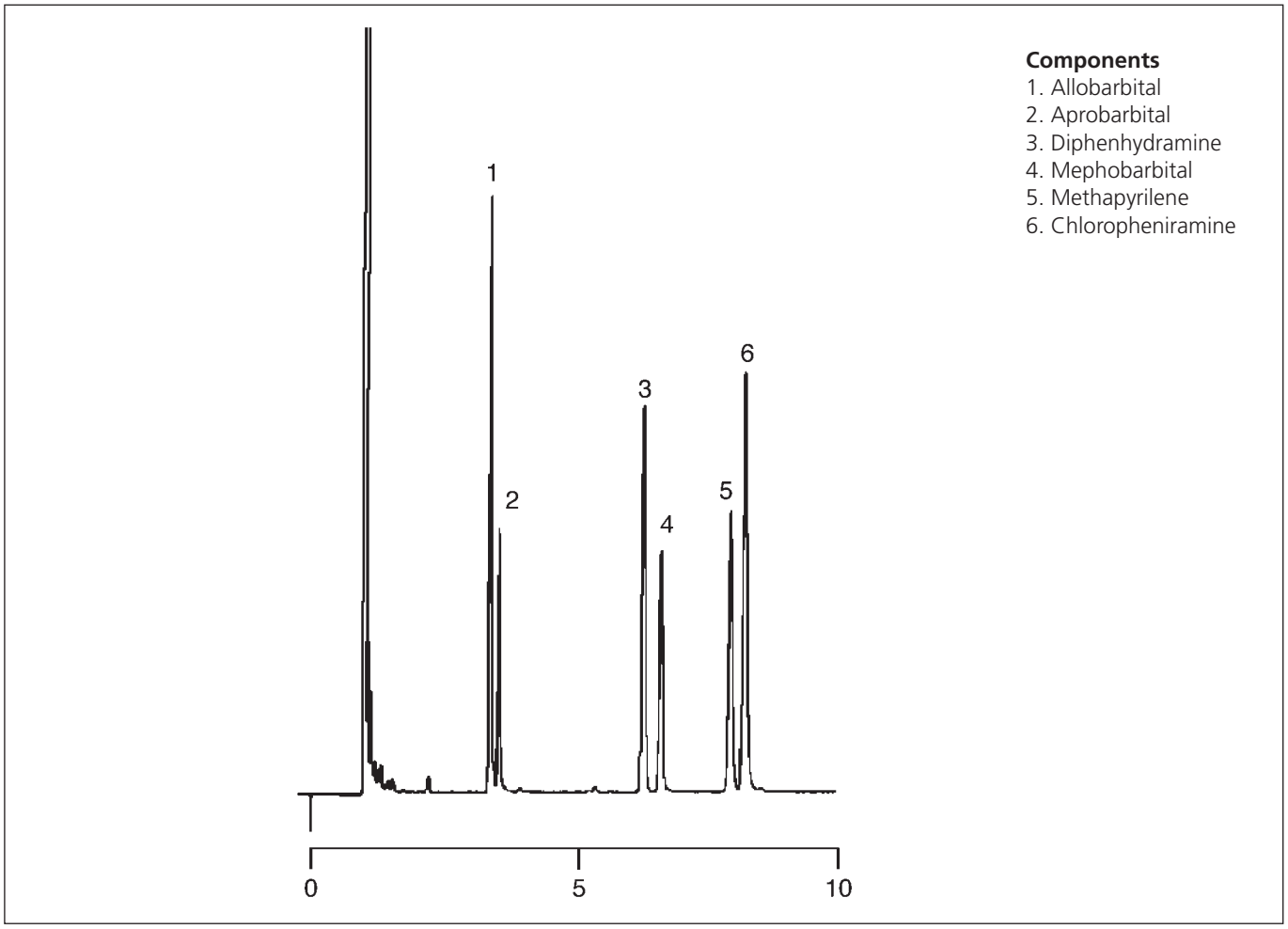
ANALYSIS OF SEDATIVES/HYPNOTICS ON BP1

Column Part No.:	**054087**	Final Temp.:	250 °C, 3 min
Phase:	BP1, 1.0 µm film	Detector:	FID
Column:	25 m x 0.53 mm ID	Sensitivity:	1024 x 10^{-12} AFS
Initial Temp.:	180 °C, 0 min	Injection Mode:	Split
Rate:	10 °C/min		



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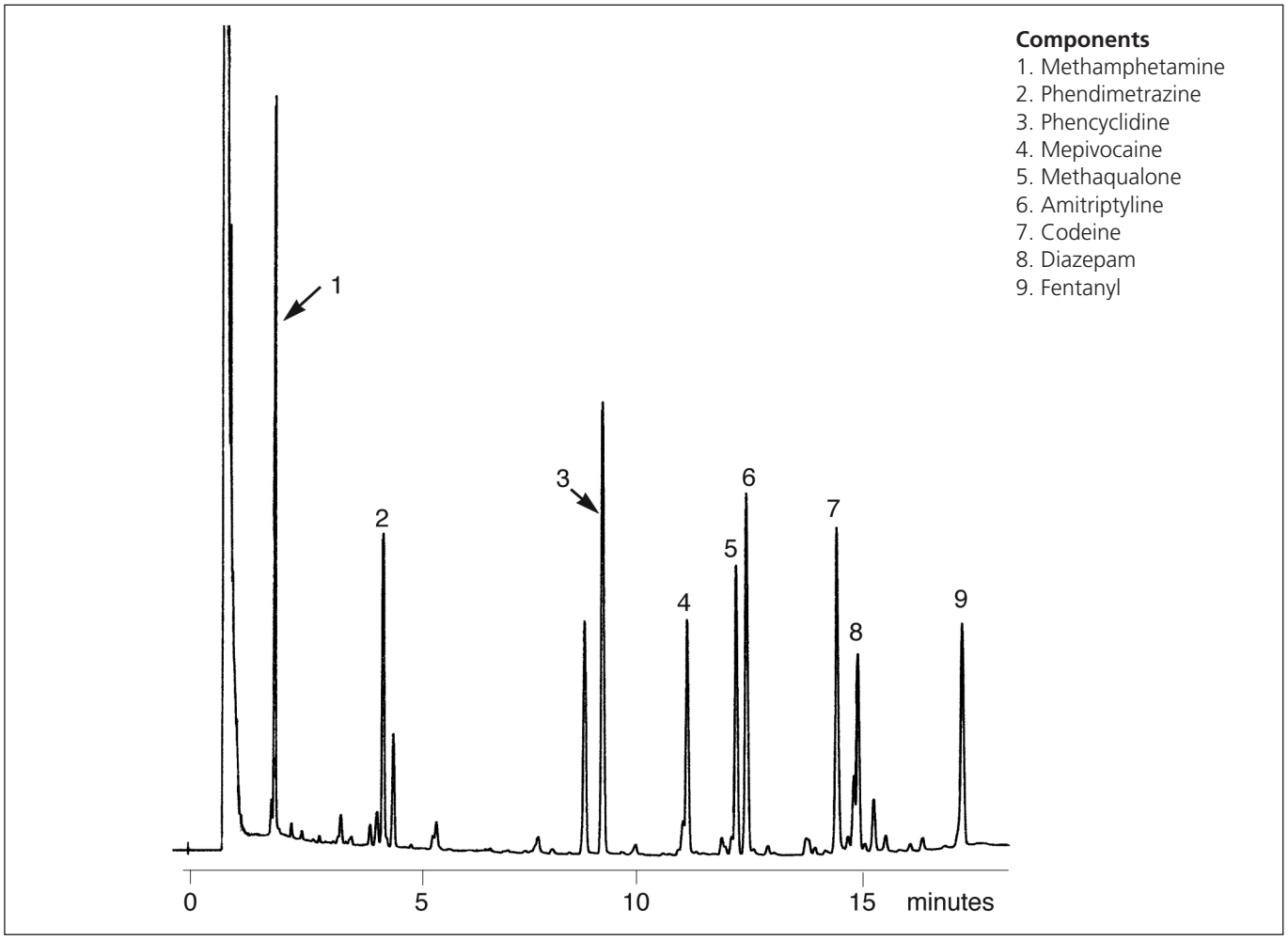
ANALYSIS OF BASIC DRUG SCREEN ON BPX5

(10-20 ng/component)

Column Part No.:	**054131**	Final Temp.:	310 °C
Phase:	BPX5, 1.0 µm	Detector:	FID
Column:	25 m x 0.53 mm I.D.	Injector:	Split, 240 °C
Initial Temp.:	120 °C	Carrier Gas:	H_2, 2 psi
Rate:	10 °C/min		

Note: The low bleed nature of the BPX5 allows trace analysis to be performed.



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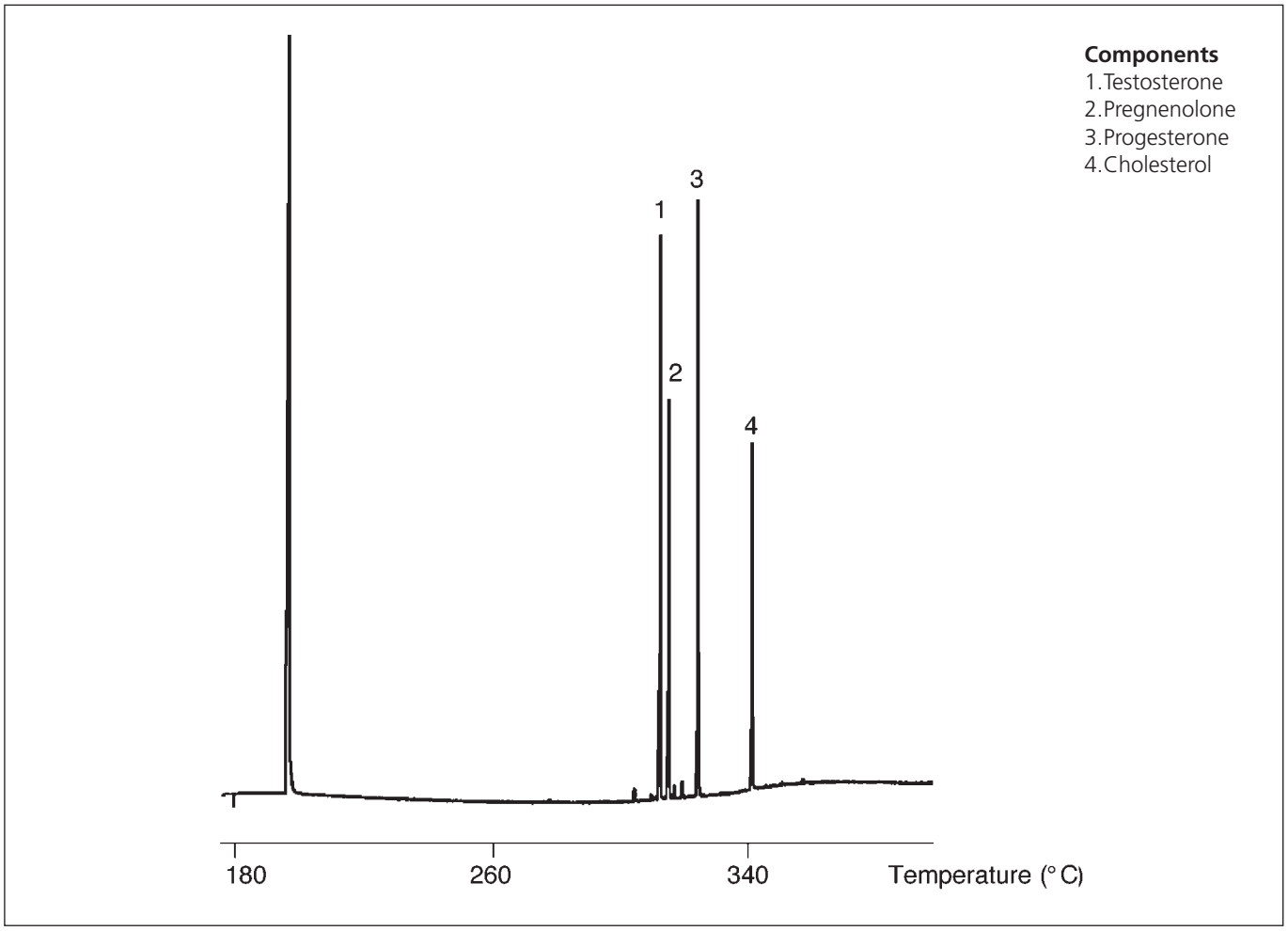
STEROID ANALYSIS (UNDERIVATISED) ON BPX5

Column Part No.:	**054113**	Final Temp.:	350 °C, 10 min
Phase:	BPX5, 0.25 µm	Detector:	FID
Column:	25 m x 0.22 mm ID	Sensitivity:	32 x 10^{-12} AFS
Intial Temp.:	180 °C	Injection Mode:	Split
Rate:	8 °C/min	Carrier Gas:	H_2, 10 psi



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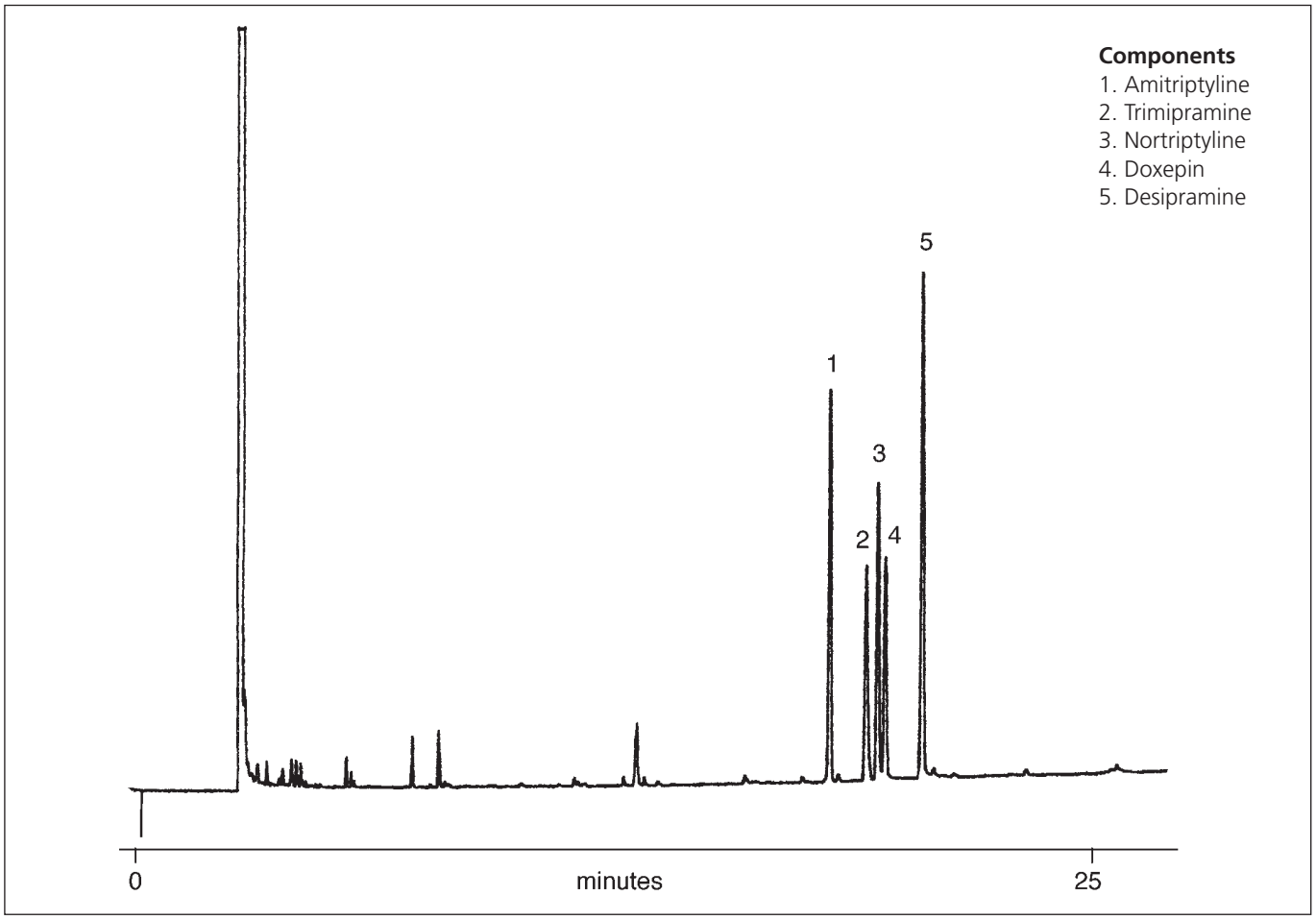
SGE Analytical Science

ANALYSIS OF TRICYCLIC ANTIDEPRESSANTS ON BPX35

Column Part No.:	**054711**	Final Temp:	280 °C
Phase:	BPX35, 0.25 µm	Carrier Gas:	Helium, 150 kpa
Column:	25 m x 0.22 mm ID	Injection Mode:	Split (20:1)
Initial Temp.:	210 °C, 1 min	Detector:	FID, 380 °C
Rate:	5 °C/min		

Note: BPX35 is a low bleed, chemically inert phase which allows trace analysis to occur.



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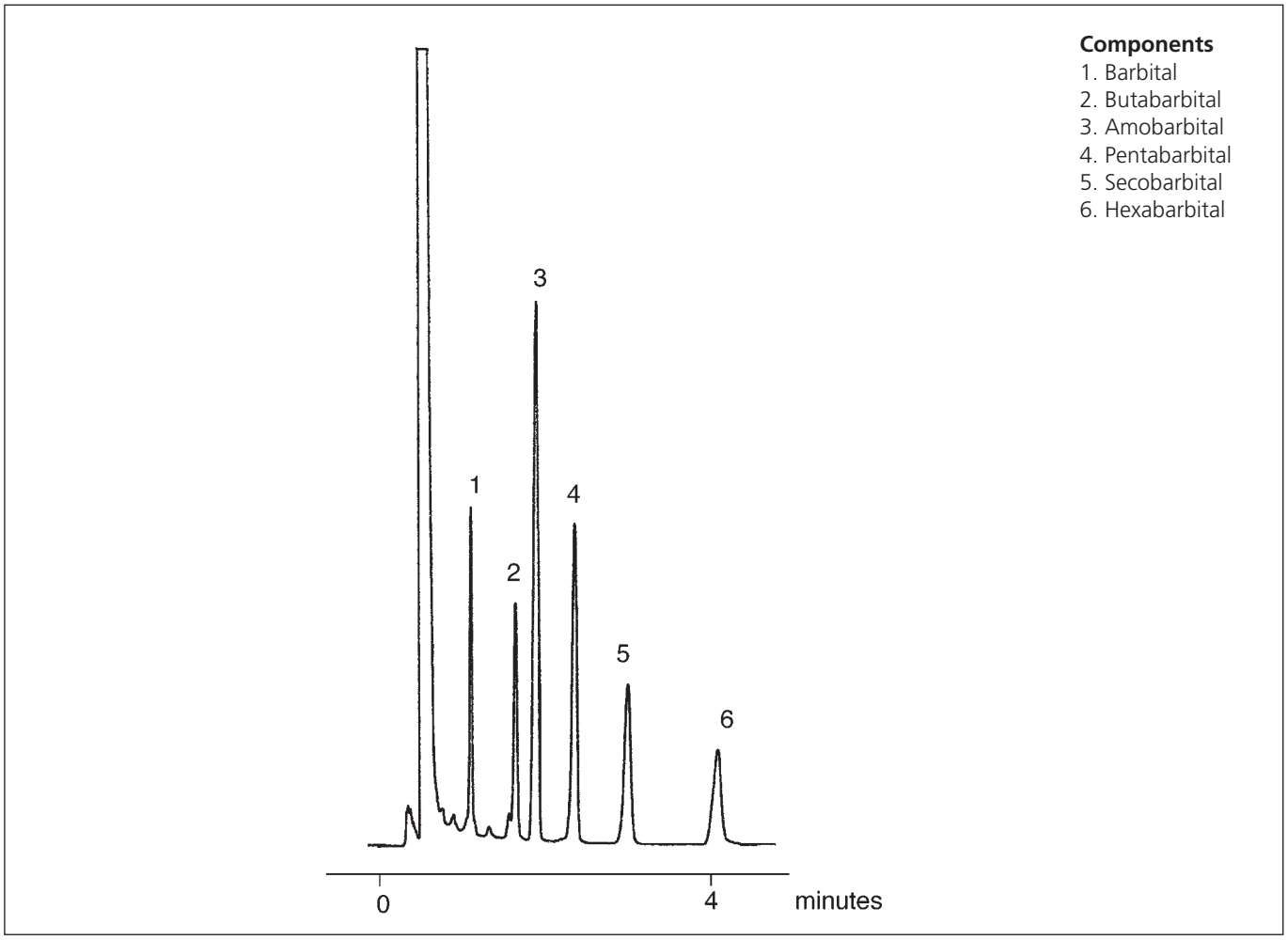
SGE Analytical Science

UNDERIVATISED BARBITURATES ON BP5

Column Part No.:	**054197**	Carrier Gas:	Hydrogen
Phase:	BP5, 1.0 µm	Carrier Flow:	10 mL/min
Column:	12 m x 0.53 mm I.D.	Injection Volume:	0.1 µL
Temp:	195 °C		



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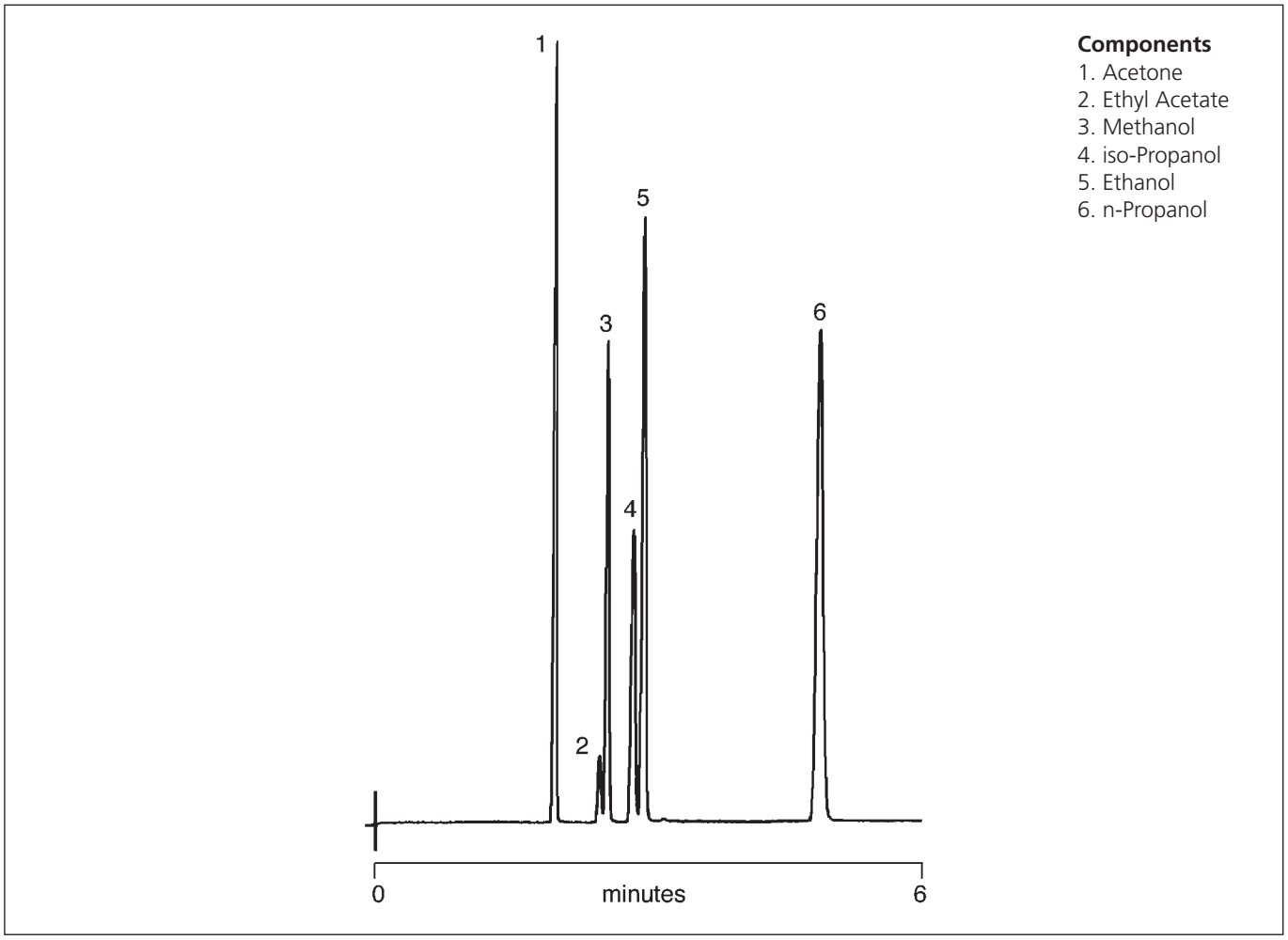
ANALYSIS OF BLOOD ALCOHOL ON BP20

Column Part No.:	**054442**	Detector:	FID
Phase:	BP20, 1.0 µm film	Sensitivity:	64 x 10^{-12} AFS
Column:	25 m x 0.32 mm ID	Injection Mode:	Split
Initial Temp:	Isothermal, 60 °C		

Note: The BP20 column allows the use of aqueous solutions.



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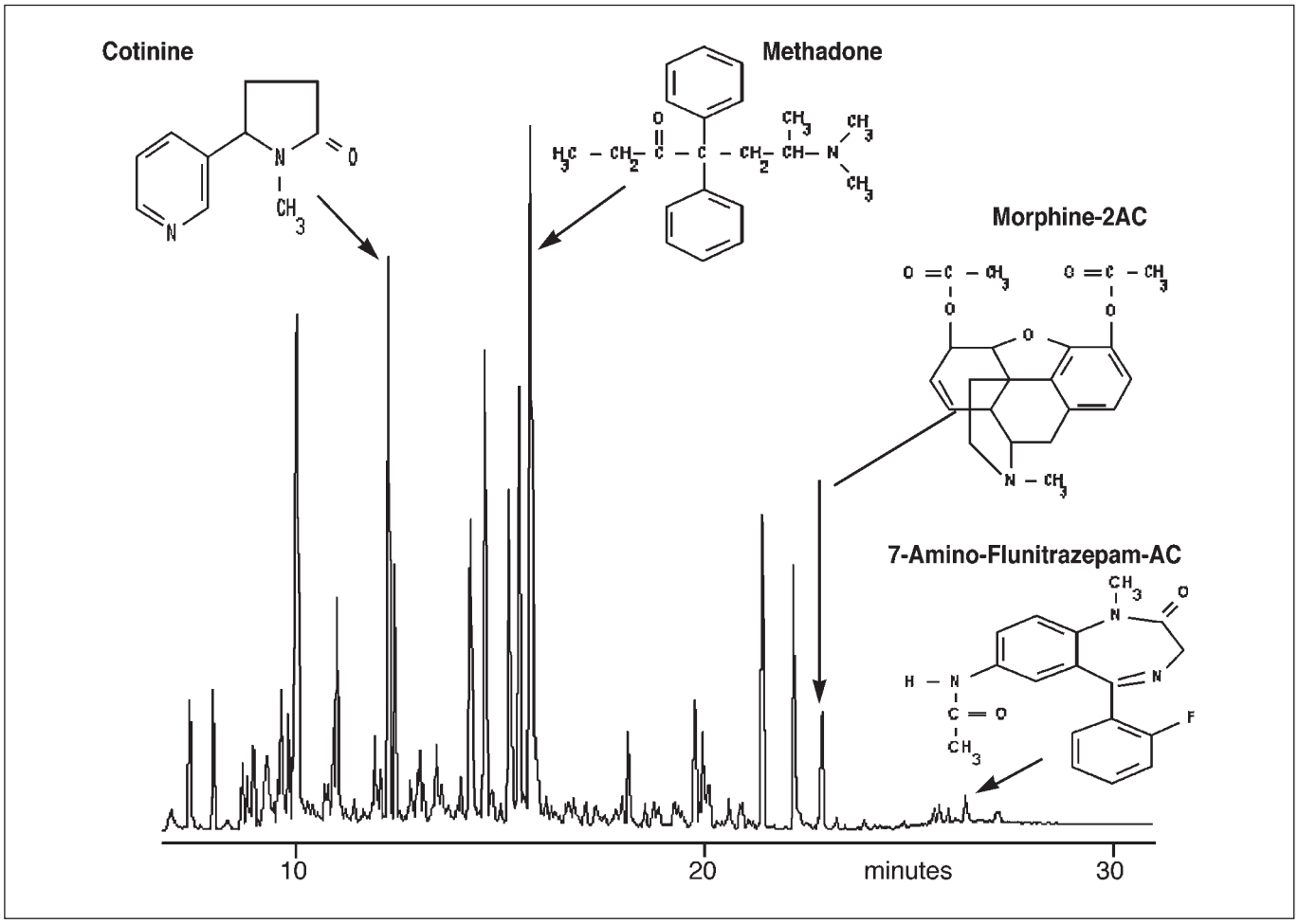
ANALYSIS OF DRUGS OF ABUSE ON BPX35

Column Part No.:	**054711**	Temp 2:	200 °C
Phase:	BPX35, 0.25 µm film	Rate 2:	7 °C/min
Column:	25 m x 0.22 mm ID	Temp 3:	295 °C
Initial Temp.:	80 °C	Rate 3:	20 °C/min
Rate 1:	15 °C/min	Final Temp.:	340 °C, 6 min



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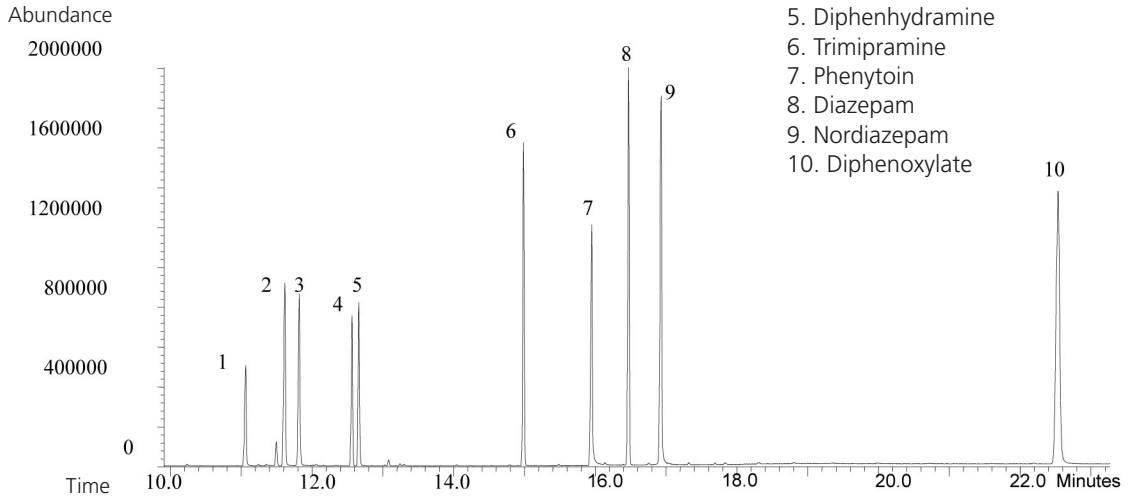
ANALYSIS OF HORSE RACING TEST MIX ON BPX5

Column Part No.:	**054101**	Constant Flow:	On
Phase:	BPX5, 0.25 µm film	Average Linear Velocity:	45 cm/sec at 75 °C
Column:	30 m x 0.25 mm ID	Injection Mode:	Splitless
Horse Racing standard*:	10 ppm in methanol	Purge on Time:	0.5 min
Initial Temp:	75 °C, 2 min	Purge on (Split)	
Rate 1:	15 °C/min to 300 °C	Vent Flow:	60 mL/min
Rate 2:	20 °C/min to 320 °C	Injection Volume:	1 µL
Final Temp:	320 °C, 8 min.	Injection Temperature:	250 °C
Detector Type:	Mass Spectrometer	Liner Type:	4 mm ID
Carrier Gas:	He, 14.5 psi		Double Taper Liner
Carrier Gas Flow:	1.5 mL/min	Liner Part Number:	092018



**Sample supplied by Racing Analytical Services Ltd.*

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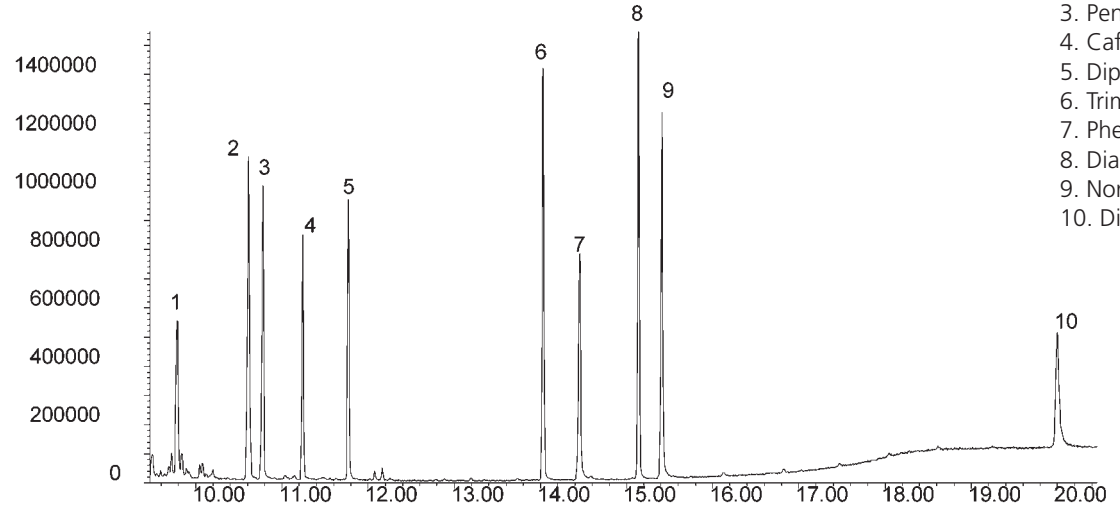
ANALYSIS OF HORSE RACING TEST MIX ON SOLGEL-1ms™

Column Part No.:	**054795**	Carrier Gas Flow:	1.7 mL/min
Phase:	SolGel-1ms™ 0.25 µm film	Constant Flow:	On
		Average Linear Velocity:	35 cm/sec at 75 °C
Column:	30 m x 0.25 mm ID	Injection Mode:	Splitless
Horse Racing standard*:	10 ppm in methanol	Purge on Time:	0.5 min
Initial Temp.:	75 °C, 2 min	Purge on (Split) Vent Flow:	60 mL/min
Rate 1:	15 °C/min to 300 °C	Injection Volume:	1 µL
Rate 2:	20 °C/min to 320 °C	Injection Temperature:	250 °C
Final Temp.:	320 °C, 8 min.	Liner Type:	4 mm ID Single Taper Liner
Detector Type:	Mass Spectrometer		
Carrier Gas:	He, 28.7 psi	Liner Part Number:	092017



Components
1. Metronidazole
2. Amylobarbitone
3. Pentobarbitone
4. Caffeine
5. Diphenhydramine
6. Trimipramine
7. Phenytoin
8. Diazepam
9. Nordiazepam
10. Diphenoxylate

**Sample supplied by Racing Analytical Services Ltd.*

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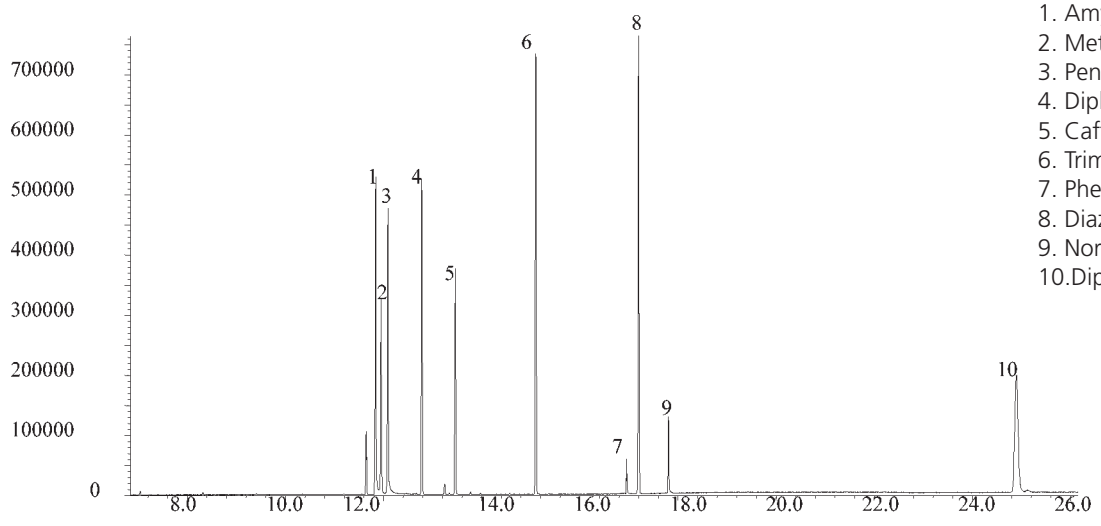
ANALYSIS OF HORSE RACING TEST MIX ON BPX35

Column Part No.:	**054701**	Constant Flow:	On
Phase:	BPX35 0.25 µm film	Average Linear Velocity:	45 cm/sec at 75 °C
Column:	30 m x 0.25 mm ID	Injection Mode:	Splitless
Horse Racing standard*:	10 ppm in methanol	Purge on Time:	0.5 min
Initial Temp.:	75 °C, 2 min	Purge on (Split) Vent Flow:	60 mL/min
Rate 1:	15 °C/min to 300 °C	Injection Volume:	1 µL
Rate 2:	20 °C/min to 320 °C	Injection Temperature:	250 °C
Final Temp.:	320°C, 8 min	Liner Type:	4 mm ID Double Taper Liner
Detector Type:	MSD		
Carrier Gas:	He, 14.5 psi	Liner Part Number:	092018
Carrier Gas Flow:	1.5 mL/min		



Components
1. Amylobarbitone
2. Metronidazole
3. Pentobarbitone
4. Diphenhydramine
5. Caffeine
6. Trimipramine
7. Phenytoin
8. Diazepam
9. Nordiazepam
10. Diphenoxylate

**Sample supplied by Racing Analytical Services Ltd.*

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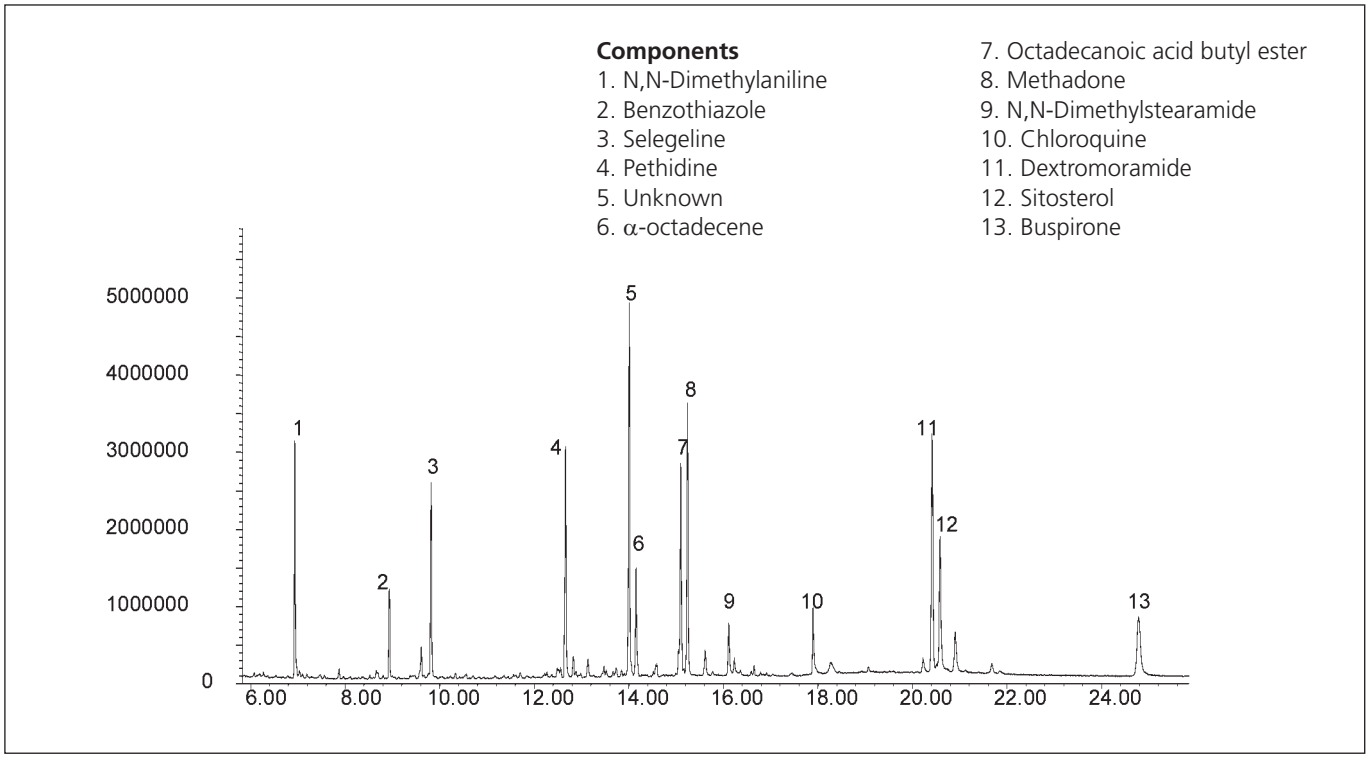
ANALYSIS OF VARIOUS DRUGS ON BPX50

Column Part No.:	**054751**	Carrier Gas Flow:	1.8 mL/min
Phase:	BPX50, 0.25µm film	Constant Flow:	On
Column:	30 m x 0.25 mm ID	Average Linear Velocity:	35 cm/sec at 40 °C
Sample:	5-10 ppm in methanol	Injection Mode:	Splitless
		Purge on Time:	0.5 min
Initial Temp:	150 °C, 0.5 min	Purge on (Split)Vent Flow:	60 mL/min
Rate 1:	10 °C/min to 180 °C	Injection Volume:	1 µL
Rate 2:	1.5 °C/min to 220 °C	Injection Temperature:	250 °C
Rate 2:	30 °C/min to 260 °C	Liner Type:	4 mm ID Single Taper Liner
Final Temp:	260 °C, 5 min		
Detector Type:	FID	Liner Part Number:	092017
Detector Temp.:	320 °C	Full Scan / SIM:	Full scan 45-450
Carrier Gas:	He, 25.7 psi		



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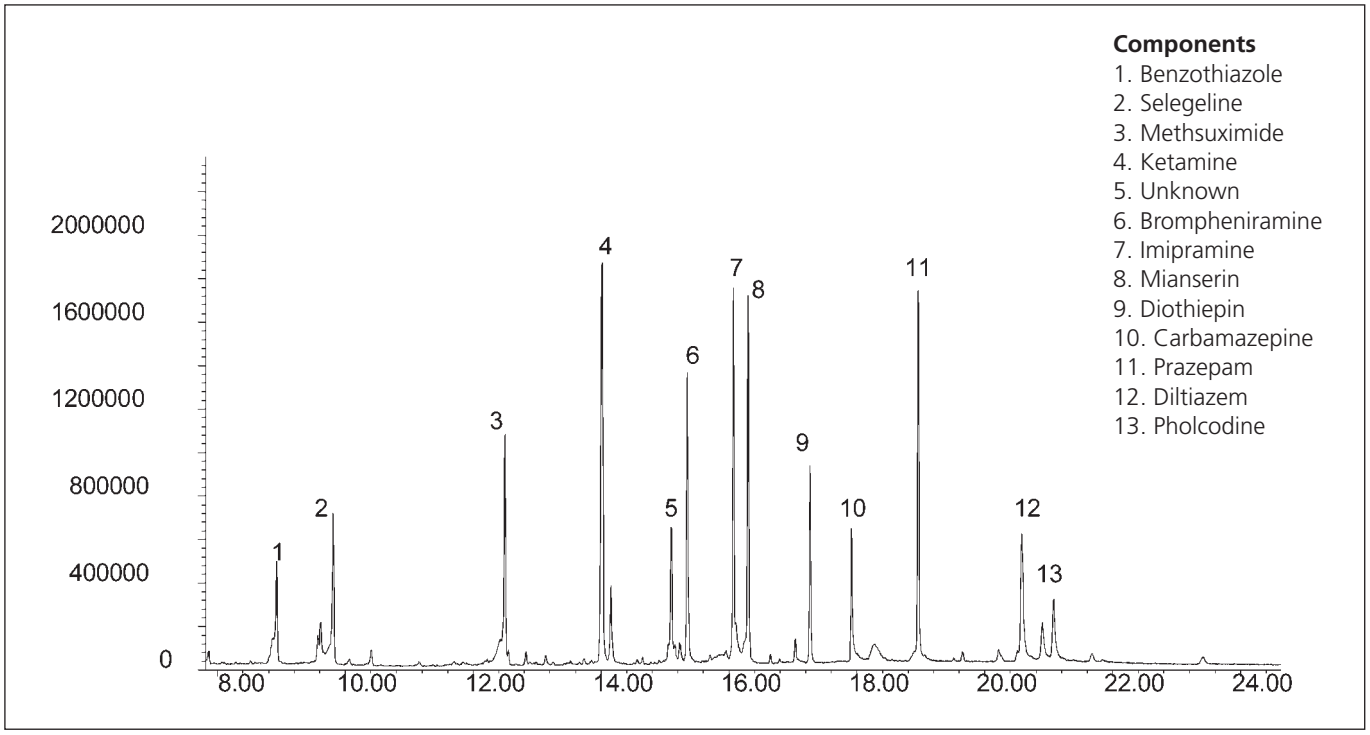
ANALYSIS OF A VARIETY OF ANTIDEPRESSANT AND ANTICONVULSANT DRUGS ON BPX50

Column Part No.:	**054751**	Carrier Gas Flow:	1.8 mL/min.
Phase:	BPX50, 0.25 µm film	Constant Flow:	On
Column:	30 m x 0.25 mm ID	Average Linear Velocity:	35 cm/sec at 40 °C
Sample:	5-10 ppm in methanol	Injection Mode:	Splitless
		Purge on Time:	0.5 min
Initial Temp:	150 °C, 0.5 min	Purge on (Split) Vent Flow:	60 mL/min
Rate 1:	10 °C/min to 180 °C	Injection Volume:	1 µL
Rate 2:	1.5 °C/min to 220 °C	Injection Temperature:	250 °C
Rate 2:	30 °C/min to 260 °C	Liner Type:	4 mm ID Single Taper Liner
Final Temp:	260 °C, 5 min		
Detector Type:	FID	Liner Part Number:	092017
Detector Temp.:	320 °C	Full Scan / SIM:	Full scan 45-450
Carrier Gas:	He, 25.7 psi		



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APPLICATION NOTE

FALSE POSITIVE DRUG TESTS FROM CONTAMINATED FEED

An On-line and Off-line Application of Micro-SPE (MEPS)

INTRODUCTION

Papaver somniferum (opium poppy) is a feed contaminant that can result in positive drug tests for racing horses.

A Micro-Extraction Packed Sorbent (MEPS) / GCMS method is described below for the regulatory testing of equine and human urine samples. The method allows for the separation of morphine and its metabolites from the metabolites of potential botanical markers that indicate the ingestion of poppy seeds or straw. MEPS is the miniaturization of conventional SPE from milliliter to microliter bed volumes that allows SPE to be used with very small samples. The manipulation of the small volumes is achieved with a precision gas tight syringe. With a typical void volume of 7 µL, the MEPS elution is compatible with GC and LC inlets making it ideal for integration into an automated sampling system for on-line SPE.

EXPERIMENTAL CONDITIONS

A 300 µL sample of diluted equine urine from an animal receiving contaminated feed was hydrolyzed with b-glucuronidase or acid, filtered and extracted on a C8/SCX MEPS cartridge conditioned with methanol (30 µL), and potassium phosphate buffer (0.2 M, pH 6, 30 µL) at a flow rate of 5 µL/sec. The exhausted fraction was ejected at the same rate and the sorbent washed with 100 µL phosphate buffer, 50 µL acetic acid (1% v/v) and 100 µL methanol. The sorbent was dried with air (3 x 80 µL at 50 µL/sec) and the sorbent eluted with 20 µL dichloromethane-isopropanol-ammonia (49:49:2). The organic phase was evaporated under nitrogen and derivatized with 10 µL of acetic anhydride-pyridine (1:2) at 80 °C for 30 minutes before evaporation and reconstitution in 5 µL of ethyl acetate.

RESULTS

The extract was analyzed by GCMS on a BPX5 column (Figure 1a and 1b).

CONCLUSIONS

This application of mixed mode C8/SCX MEPS for a complex biological fluid, allowed the microscale preparation of a small volume sample with comparable performance to conventional SPE techniques. Used off-line with derivatization and GCMS here, the sample was also suitable for on-line ESI-LCMSMS analysis by changing the elution solvent to methanol-ammonia (98:2) or methanol-trimethylamine (98:2).

In most cases, MEPS allows the same level of sample concentration as is possible with off-line conventional SPE while providing opportunities for truly hybrid multi-dimensional methods. MEPS methods may be readily adapted from established SPE methods including those based on mixed mode or complex chemistries.

Like SPE, MEPS is for use with liquid samples (either normal or reversed phase) and yields four fractions: the unretained, weakly bound, strongly bound and irreversibly bound. However, because MEPS is a double pass system (sample and solvent enter and exit from the bottom of the bed), the weakly bound fraction (commonly the interferences eliminated by washing) is less strongly bound. The irreversibly bound fraction affects MEPS and conventional SPE and is usually associated with sorbent wetting rather than sample purification and so the irreversible binding of matrix material from one sample does not preclude reuse of the device for a sample of the same type.

Also like conventional SPE, the number of times the device can be re-used is dependent on the sample matrix. For simple applications, MEPS devices have been used successfully for more than 50 cycles.

REFERENCES

(1) Wynne PM, The accidental contamination of animal feed by naturally occurring opiates with particular reference to morphine. An independent review and analysis of current knowledge. The British Equestrian Trade Association (London), 2005; pp1-143.

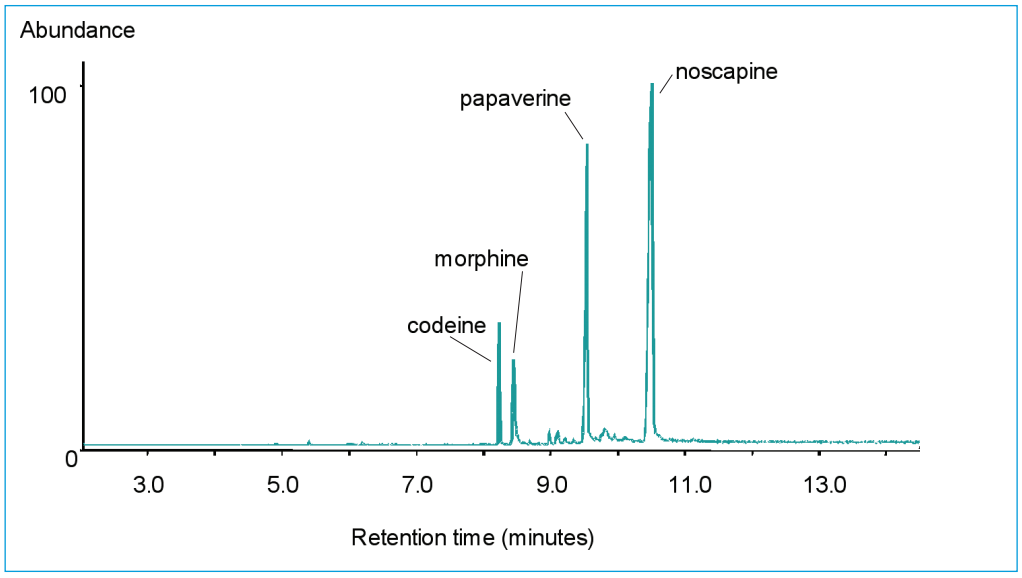

Figure 1a: The analysis of *Papaver somniferum* ssp. *setigerum* by GCMS following micro-SPE extraction of poppy residues and GCMS analysis of the recovered fraction.

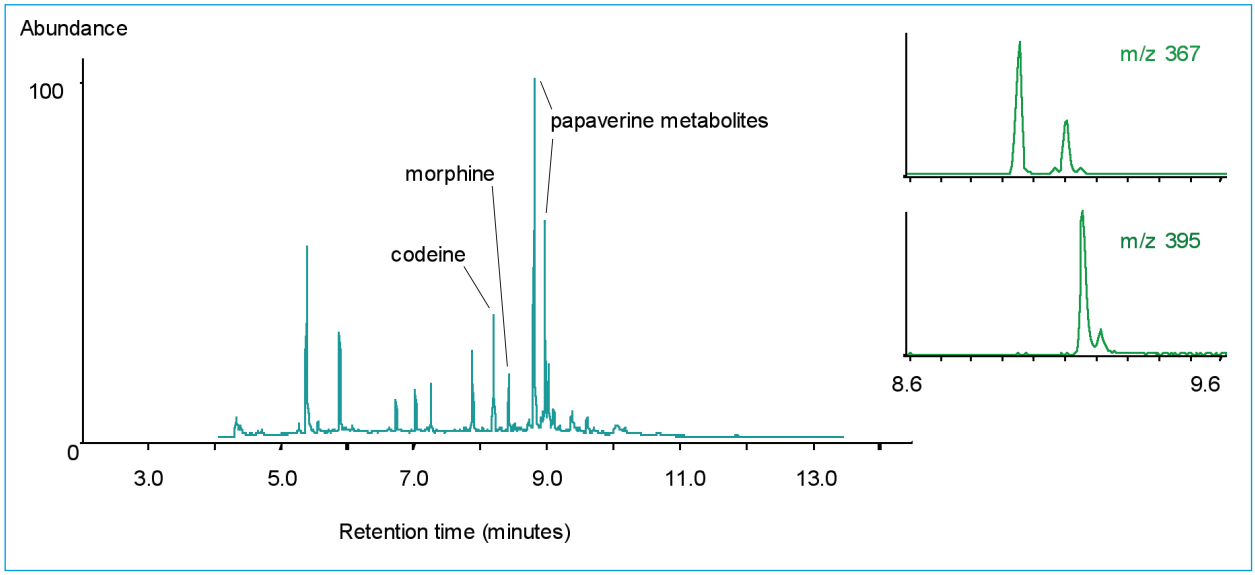

Figure 1b: The analysis of enzyme hydrolyzed horse urine by GCMS following micro-SPE extraction and micro-derivatization (peracetylation) of the recovered basic fraction. (Inset m/z 367 corresponds to O-desmethylpapaverine metabolites and m/z 395 is the O,O'-didesmethylmetabolites)