Application Note No. 119

Analysis of difficult to retain components using OPTIC Multimode Inlet with OPTIC Cooler

Key words:
OPTIC-4
OPTIC-Cooler
Pyridine
N-Nitrosodimethylamine
8270
LVI
Dichloromethane (methylene chloride)

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Introduction

EPA 8270 method is used to determine the concentration of semi-volatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air and water samples. Pyridine and N-Nitrosodimethylamine are just two out of a number of 'difficult to retain' compounds in the long method list. It is known that N-Nitrosodimethylamine is difficult to separate from the solvent under the prescribed chromatographic conditions and Pyridine may perform poorly at the GC inlet port temperatures listed in the method. Lowering the inlet temperature reduces the amount of Pyridine degradation and improves the recovery of N-Nitrosodimethylamine. When using the prescribed splitless injection, the inlet port should be cooled down to sub-ambient temperatures in order to achieve a significant improvement. At the same time, one should use caution in lowering the inlet temperature as the performance of other analytes may be adversely affected. A better solution may be found if Large Volume Injections (LVI) is applied for the analysis. Besides significant sensitivity increase, the LVI method has an important inherent property – solvent cooling effect [1]. Presence of this cooling effect significantly improves the analytes recovery even if a moderate lowering of the inlet temperature is used. In this note we demonstrate reliable and sensitive detection of both Pyridine and N-Nitrosodimethylamine using LVI injection method in combination with our new Peltier inlet cooler.

[1] Hans G.J. Mol, Hans-Gerd Janssen, and Carel A. Cramers, J. High. Res. Chromatogr. 18 (1995) 19-27.

Sample

500-1000 ng/µl Megamix 8270 from Restek diluted with dichloromethane to 0.4 ng/µl. Injection volume is 25 µl.

Instrumentation

GC Inlet: OPTIC-4 Multimode Inlet
Inlet cooler: OPTIC Cooler
Liner: A100095, EPA 8270 packed liner
GC-MS: Shimadzu QP2010
GC column: GL Sciences InertCap 5 MS/Sil, 0.25 m x 30 m, film 0.25 µm

Conditions

OPTIC-4

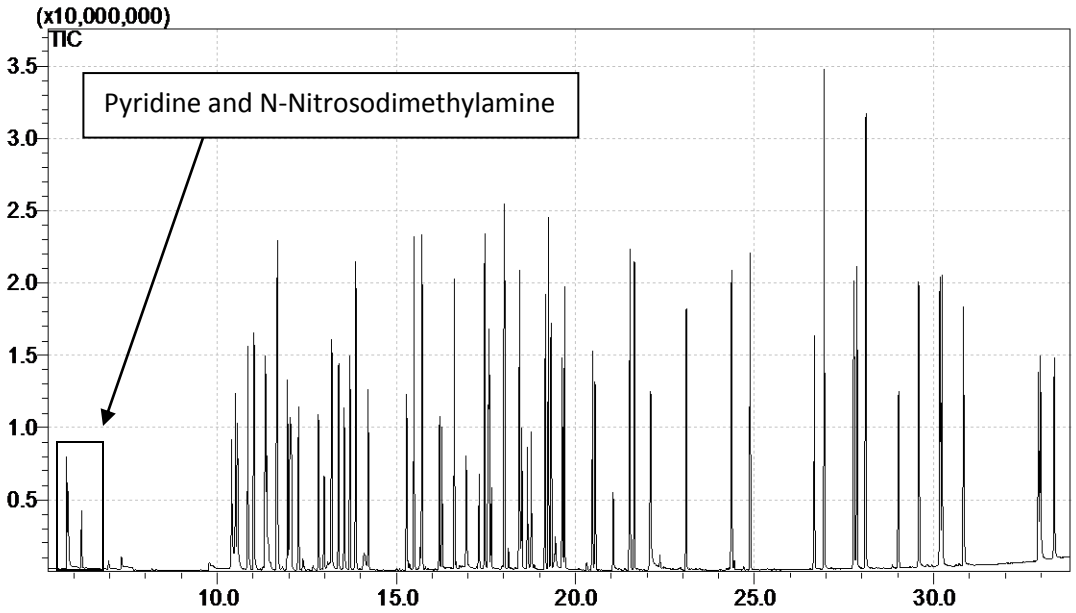
Method Type	Large Volume
Method Name	8270lvi
Equilibration Time	00:05 mm:ss
End Time	35:00 mm:ss
Initial Temperature	7 °C (and 30°C)
Ramp Rate	8 °C/s
Final Temperature	300 °C
Temperature Control	Keep Current Temperature
Septum Purge Flow	5 ml/min
Vent Mode	Fixed Time
Vent Time	01:40 mm:ss
Carrier Control Mode	Flow Control
Transfer Time	02:30 mm:ss
Sample Sweep Column Flow	1.0 ml/min
Transfer Column Flow	1.0 ml/min
Start Column Flow	1.0 ml/min
End Column Flow	1.0 ml/min
Vent Flow	30.0 ml/min
Split Flow	25.0 ml/min

GC/MS

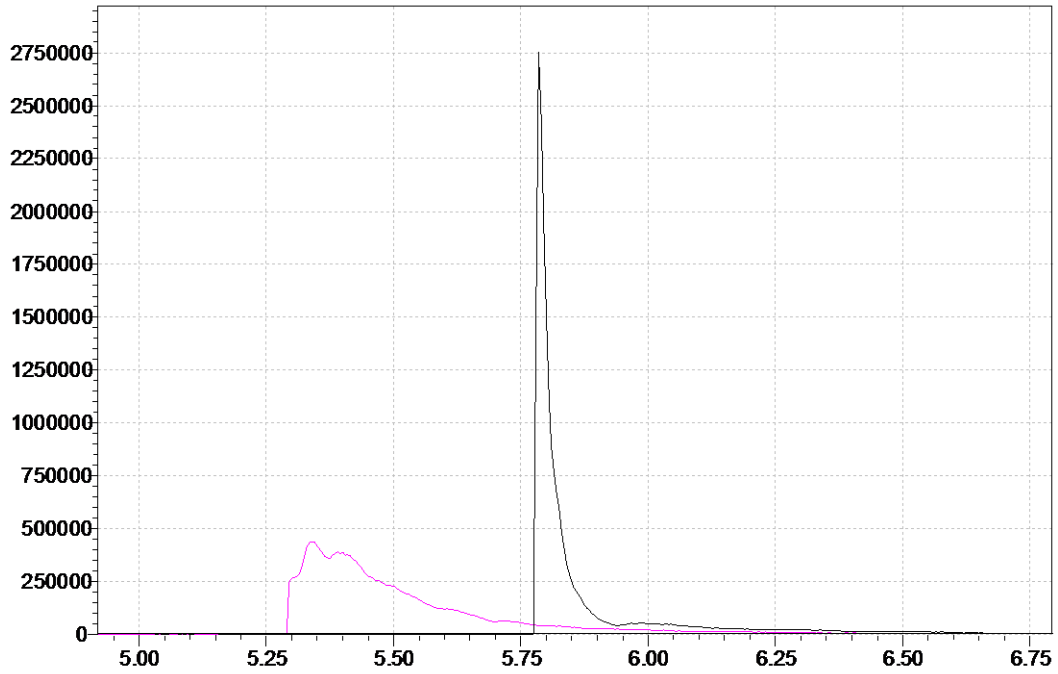
Column Oven Temp. :		30.0 °C
Oven Temp. Program		
Rate (°C/min)	Temperature(°C)	Hold Time(min)
-	30.0	4.00
10.00	330.0	2.00
Equilibrium Time		0.0 min
GCMS-QP2010		

Ion Source Temp	200.00 °C
Interface Temp	250.00 °C
Solvent Cut Time	4.00 min
Detector Gain Mode	Relative
Detector Gain	0.00 kV
Threshold	0
MS Table	
Start Time	4.00 min
End Time	35.00 min
ACQ Mode	Scan
Event Time	0.30sec
Scan Speed	1250
Start m/z	40.00
End m/z	400.00
Sample Inlet Unit	GC

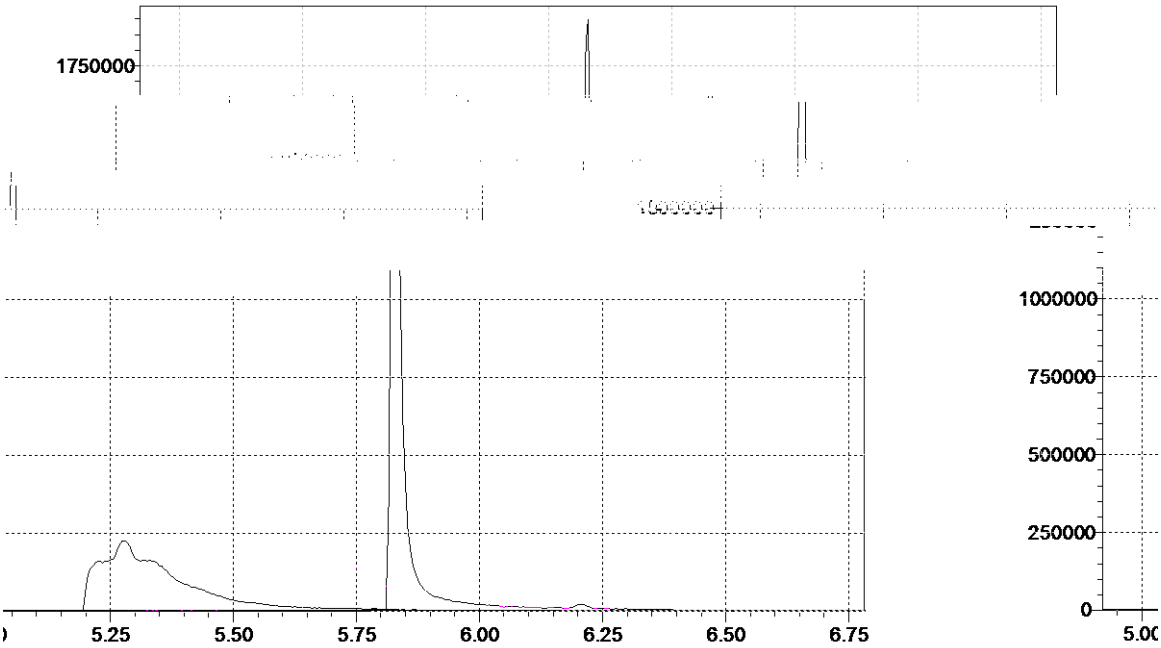
Results:



Chromatogram of 8270 Megamix in TIC mode



Component: Pyridine
Pink: 25 µl injection, the initial inlet temperature 30°C
Black: 25 µl injection, the initial inlet temperature 7°C



Component: N-Nitrosodimethylamine
Pink: 25 µl injection, the initial inlet temperature 30°C
Black: 25 µl injection, the initial inlet temperature 7°C

Conclusions:

Recovery of ‘difficult to retain’ components can significantly be improved when using an LVI injection method in combination with the OPTIC Peltier Cooler for the inlet.