

## **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<sup>1</sup>. 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.

<sup>1</sup> Hans G.J. Mol, Hans-Gerd Janssen, and Carel A. Cramers, J. High. Res. Chromatogr. 18 (1995) 19-27.

**Sample**

500-1000 ng/ $\mu$ l Megamix 8270 from Restek diluted with dichloromethane to 0.4 ng/ $\mu$ l. Injection volume is 25  $\mu$ l.

**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  $\mu$ m

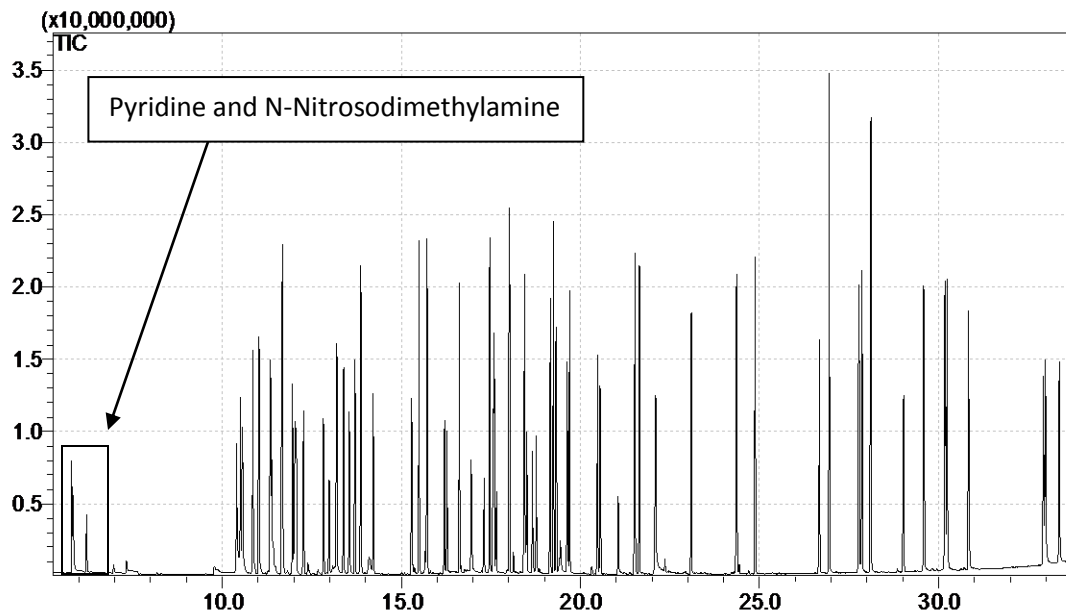
**Conditions****OPTIC-4**

|                          |                          |
|--------------------------|--------------------------|
| 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              |

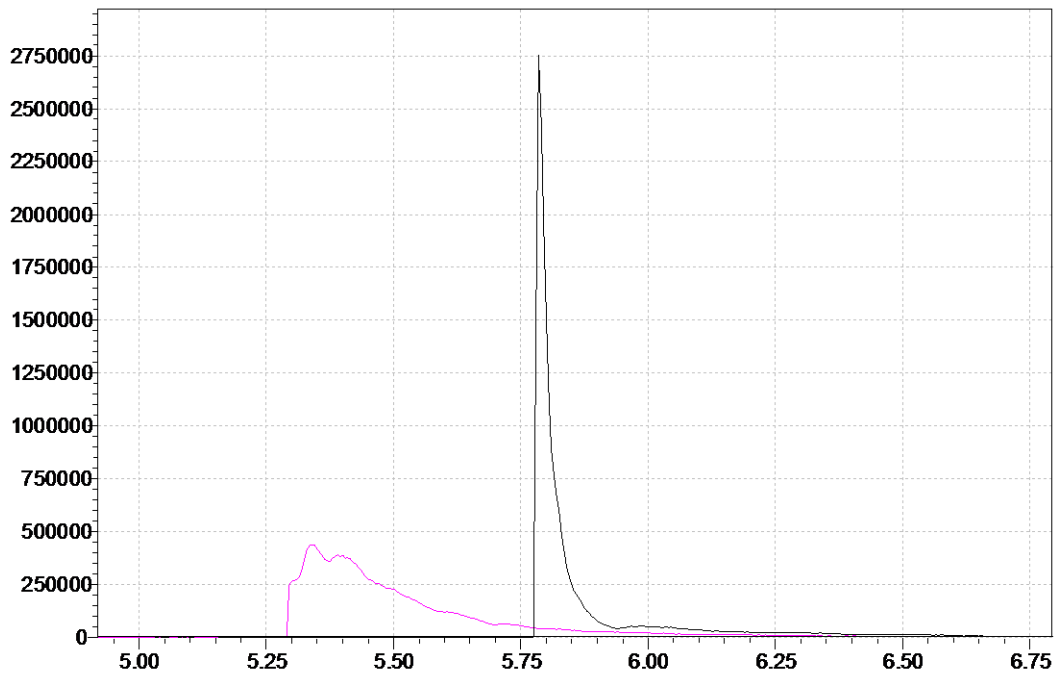
**GC/MS**

|                     |                 |                |
|---------------------|-----------------|----------------|
| 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         |                |
| <b>GCMS-QP2010</b>  |                 |                |

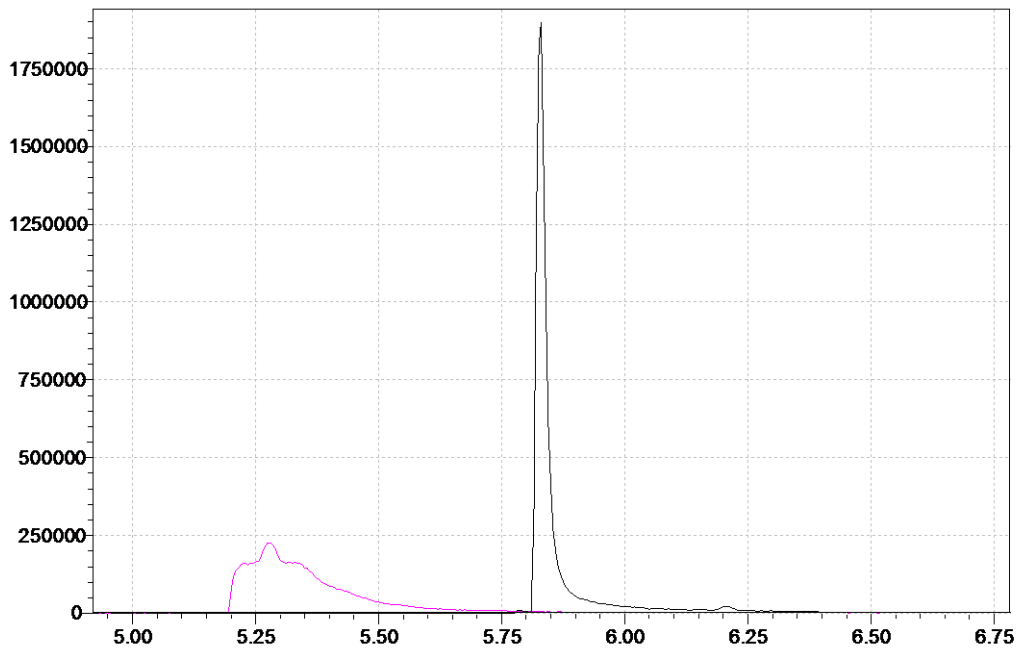
|                    |           |
|--------------------|-----------|
| Ion Source Temp    | 200.00 °C |
| Interface Temp     | 250.00 °C |
| Solvent Cut Time   | 4.00 min  |
| Detector Gain Mode | Relative  |
| Detector Gain      | 0.00 kV   |
| Threshold          | 0         |
| <b>MS Table</b>    |           |
| 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.