

Application Note No. 054

Analysis of Trace Level Silicone Oil in Vehicle Paint using Difficult Matrix Introduction (DMI) Coupled with Selective Exclusion

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Exclusion of major components allowing trace level detection

- No manual sample preparation
- May be automated using the Focus DTD

Instrumentation

- ATAS Optic 2-200 programmable injector
- HP5890 with FID

Sample analysed

Vehicle paint contaminated with both high and low levels of silicone oil.

Principles

- Inject 1 μ L of paint into the bottom of a DMI microvial
- Place microvial into a fritted liner and place in the Optic injector
- Vent the solvents at a low temperature
- Selectively desorb the silicone oil
- Transfer the components onto the head of the column in splitless mode
- At the end remove the microvial containing the involatile constituents and reuse the liner

Chromatogram

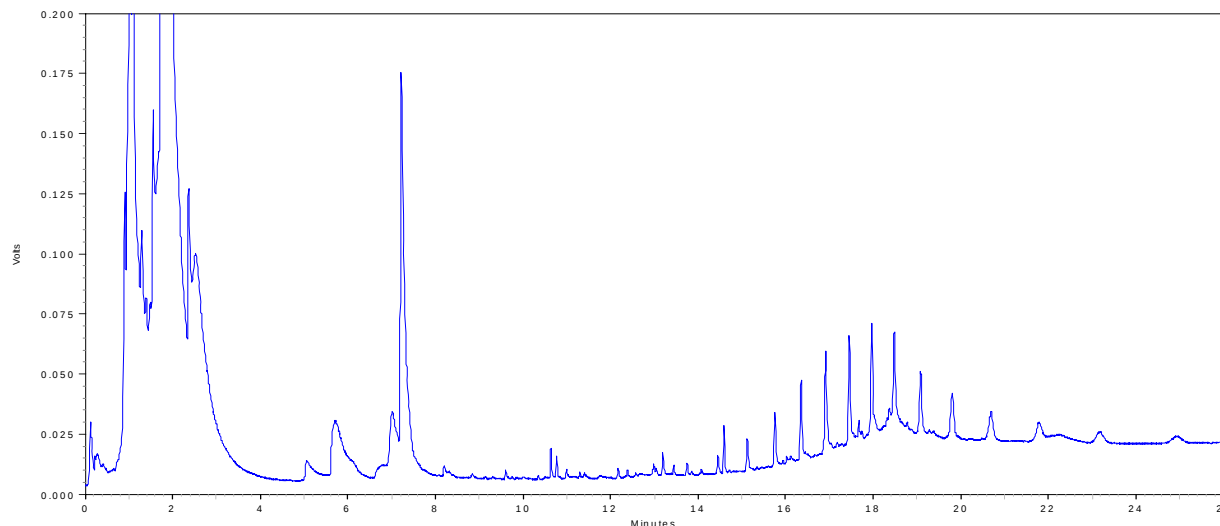


Figure: Paint spiked with low level silicone oil

We would like to thank Matthew Griffin from BMW for his kind permission to publish this information.

For more information please contact us at one of the addresses below.

Appendix

Optic Conditions:

- Liner: ATAS Fritted + 5 mm DMI
Microvial
- Mode: Expert
- Injection volume: 1 µL
- Gas Flows: Split: 100 ml/min
Vent: 120 ml/min
- Equilibration Time: 0:30 m:s
- Initial temperature: 100 °C
- Isothermal time: 12:30 m:s
- Ramp rate: 16 °C/s
- Final temperature: 325°C
- Vent time: 12:20 m:s
- Splitless time: 4:00 m:s
- Split open time: 16:30 m:s
- Purge pressure: 3 psi
- Purge time: 12:20 m:s
- Desorption pressure: 0 psi
- Desorption time: 2:00 m:s
- Initial pressure: 30 psi
- Final pressure: 60 psi

GC conditions:

- Column: DB5 30m x 0.32mm i.d. x 0.25 µm film
- Initial Temperature: 150 °C
- Initial Time: 2 mins
- Ramp Rate: 15 °C/min
- Final Temperature: 325 °C
- Final Time: 15 mins
- FID temperature: 350 °C

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