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Title: Using the JetClean Self-Cleaning Ion Source to Extend Maintenance Free Operation

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Introduction

Phthalate Analysis

Matrix and column bleed deposits over time degrade instrument response, necessitating routine source cleaning. The process requires removal and abrasive cleaning of the source, leading to lost productivity. The patented JetClean self-cleaning ion source (JetClean) was developed to maintain consistent MS response in difficult matrices for extended periods of time through the introduction of carefully controlled hydrogen into the MS source. This poster describes the application of JetClean to the analysis of phthalate esters (phthalates). Due to the phthalates' adverse health effects (plausible endocrine disruptors) their use is limited by international regulations.

Phthalates can exhibit several undesirable characteristics in GC/MS analysis:

- Non-linearity: some analytes have reduced response at the lower cal levels.
- Peak Tailing: compounds stick to the source with some ions tailing more than others.
- Sensitivity: Higher source temperatures are often used to improve linearity, but some compounds lose sensitivity.
- Dropping Response: Raw area response for replicate injections can exhibit significant loss of response with time.

These problems were investigated and addressed with a number of hardware and method changes, resulting in significantly improved results.

Experimental

Instrument Conditions and Test Mixture

GC	**Agilent 7890B**		
Ramp	°C/min	°C	Hold min
Initial		60	1.0
Ramp 1	20	220	1.0
Ramp 2	5	290	4.0
Runtime	40 min		

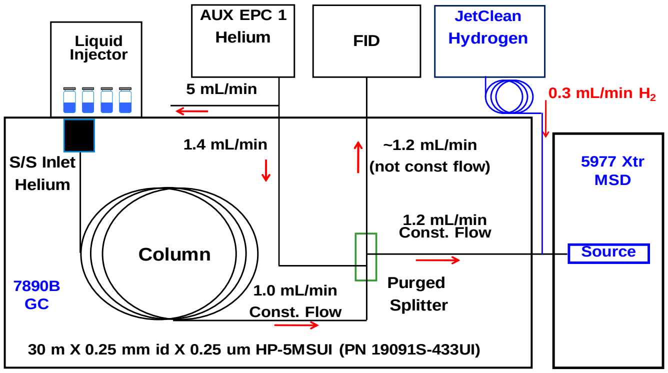
Inlet	Split/Splitless (or MMI)
Temp	290 °C
Mode	Hot Pulsed Splitless,
Init. Pressure	15.0 psi
Pulse Pressure	35 psi until 1.0 min
Purge Flow	50 mL/min at 1.0 min
Septum Purge	3 mL/min, switched
Injection volume	0.5 uL

Inlet Liner	Ultra Inert Single Taper w/ Wool (PN. 5190-2293)
Column	HP-5MSUI (19091S-433UI) 30m x 0.25 mm id x 0.25 um
Column Flow	1.0 mL/min, const flow

Splitter	2-way splitter
Pressure	4.2 psig (initial)
MSD Restictor	0.68 m x 0.12 mm id 1.2 mL/min const flow
FID Restictor	0.37 m x 0.12 mm id
Split ratio	~1:1 MSD:FID

MSD	**Agilent 5977 Xtr**
Solvent Delay	5.0 min
Acquisition Mode	SIM, TID ON
Tune	Atune
EMV	Gain 1
Quad Temp	150 'C
Source Temp	300 'C
Transfer Line	280 'C
Drawout Lens	9 mm
JetClean Flow	0.3 mL/min H_2
JetClean Mode	continuous

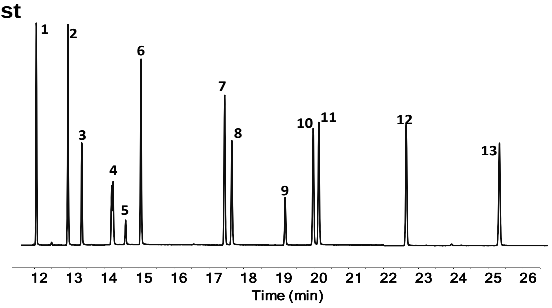
Hardware Configuration



The system has a CFT splitter between the MSD and FID. The FID signal makes a good reference for peak shape, linearity, and precision. Use of the FID helps distinguish between detector effects and inlet, liner, and column effects.

Phthalate Test Mixture

The phthalate test mixture was diluted in isooctane at various levels to make the calibration standards for linearity and parameter optimization.



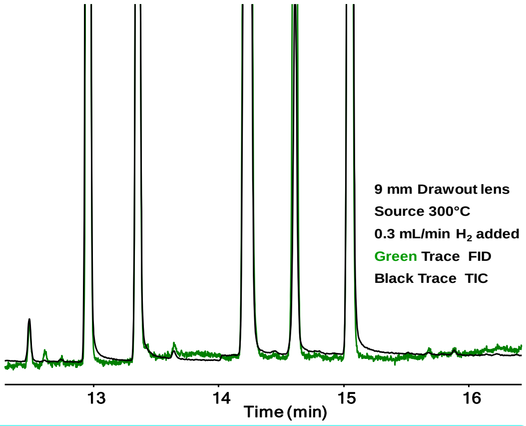
		Tgt Ion
1	Disobutyl	149
2	Dibutyl	149
3	Bis(2-methoxyethyl)	59
4	Bis(4-methylpentyl)	149
5	Bis(2-ethoxyethyl)	149
6	Dipentyl	149
7	Dihexyl	149
8	Benzyl butyl	149
9	Bis(2-butoxyethyl)	149
10	Dicyclohexyl	149
11	Bis(2-ethylhexyl)	149
12	Di n-octyl	149
13	Dinonyl	149

Results

Peak Shape Improvement

The first parameters investigated were source temperature and drawout lens diameter. A diameter of 9 mm instead of the typical 3 mm and a temperature of 300°C were found to be optimum. However, without hydrogen cleaning gas added, the peaks still showed tailing. The degree of tailing was not the same on all ions, EICs for multiple ions from the same peak had differing amounts of tailing.

The figure to the right shows that this differential tailing is greatly reduced with the continuous addition of 0.3 mL/min of H_2 to the source, resulting in TIC and FID peak shapes to be very comparable.



Linearity Improvement

The calibration parameters were studied, examining source temperature, H_2 addition, and tune type. In all cases, the 9 mm drawout lens was used. Calibrations were ISTD with 3 runs at each level.

Optimal results were found with the source at 300°C, 0.3 mL/min H_2 added, and using ATUNE.

Cal Range, pg	2.5 - 1250	2.5 - 1250	2.5 - 1250	2.5 - 1250	2.5 - 1250	2.5 - 1250	2.5 - 1250	2.5 - 1250
Stat	r^2	%RSD	r^2	%RSD	r^2	%RSD	r^2	%RSD
Source Temp °C	300	300	230	230	230	230	230	230
H_2, mL/min	0.3	0.3	0.3	0.3	0.3	0.3	no H_2	no H_2
Tune	Atune	Atune	Atune	Atune	Etune	Etune	Etune	Etune
Tetradecane	0.9999	3.0	0.9999	5.9	0.9999	4.2	0.9979	22.9
Dimethyl	0.9998	2.4	0.9999	6.2	0.9993	6.9	0.9993	15.0
Diethyl	0.9998	1.9	0.9999	6.9	0.9990	8.8	0.9987	17.5
Disobutyl	0.9998	4.4	0.9999	8.8	0.9987	9.4	0.9990	15.9
Dibutyl	0.9999	10.9	0.9998	14.7	0.9983	14.0	0.9982	21.2
Bis(2-methoxyethy	0.9998	21.1	0.9990	21.9	0.9980	26.3	0.9980	35.8
Bis(4-methylpentyl	0.9996	4.2	0.9999	9.5	0.9990	12.5	0.9991	20.0
Bis(2-ethoxyethyl)	0.9997	16.7	0.9994	20.8	0.9982	24.6	0.9983	32.0
Dipentyl	0.9998	3.8	0.9998	7.8	0.9978	15.7	0.9969	27.9
Dihexyl	0.9998	4.1	0.9999	6.8	0.9984	13.3	0.9988	20.0
Benzyl butyl	0.9997	5.9	0.9997	13.0	0.9982	19.7	0.9975	32.6
Bis(2-butoxyethyl)	0.9998	13.8	0.9997	20.3	0.9982	23.0	0.9990	30.3
Dicyclohexyl	0.9979	5.4	0.9989	23.6	0.9976	30.7	0.9971	42.3
Bis(2-ethylhexyl)	0.9997	3.8	0.9999	7.9	0.9981	13.7	0.9982	22.7
Di n-octyl	0.9997	2.9	0.9999	7.7	0.9984	14.1	0.9992	19.3
Dinonyl	0.9996	3.1	0.9998	9.8	0.9985	14.4	0.9993	20.0

< 0.999

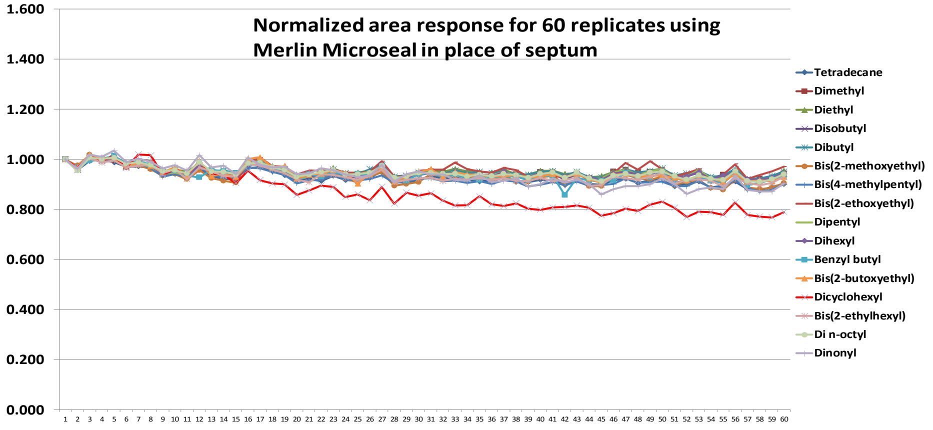
< 30%

< 20%

< 10%

Area Precision

One significant problem with phthalates is dropping response with replicate injections. This problem may be present even if no matrix is injected. Employing JetClean significantly increased precision, which was further improved by employing a Merlin Microseal instead of the standard spectrum.



Normalized area response of 60 consecutive injections of the phthalate mixture at 125pg level. All compounds show a remarkable replicate area precision. The Merlin Microseal contribution to the precision improvement is currently under investigation.

Summary and Conclusions

- The GC/MS analysis of phthalates can be improved in terms of peak shape, linearity, and repeatability by incorporating these changes to the analysis method:

- Run the source temperature at 300°C. Values lower than that may result in tailing and dropping response. Higher values result in problems with some of the more thermally labile phthalates.

- Reduce drawout lens interactions with the phthalates by changing to a larger diameter, like 9 mm.

- Using ATUNE.U instead of ETUNE.U improves linearity and peak shape for phthalates.

- Addition of continuous hydrogen to the source during analysis with JetClean improves peak shape, linearity, and replicate precision.

- Use of a Merlin Microseal further improved response stability.