ENVIRONMENTAL ANALYSIS

ANALYTICAL EVALUATION OF THE AGILENT IDP-15 OIL-FREE SCROLL PUMP AND EDWARDS ROTARY PUMP ON THE AGILENT 7000 GC/MS-MS

Solutions for Your Analytical Business
Markets and Applications Programs

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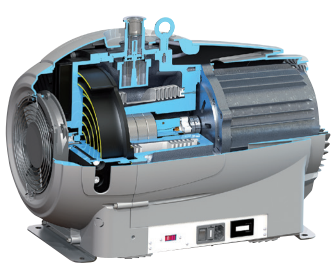
ABSTRACT

The Agilent IDP-15 Oil free scroll pump was evaluated and compared for analytical performance against the conventional Edwards 5 Pump in two independent studies. Both studies used Agilent 7000 Triple Quadrupole Mass Spectrometers (GC/MS-MS). Parameters assessed included vacuum readings and tune reports. The studies concentrated on two different sets of compounds and examined the chromatograms, calibration curves, qualifier ratios, repeatability and S/N produced when using the two different configurations.



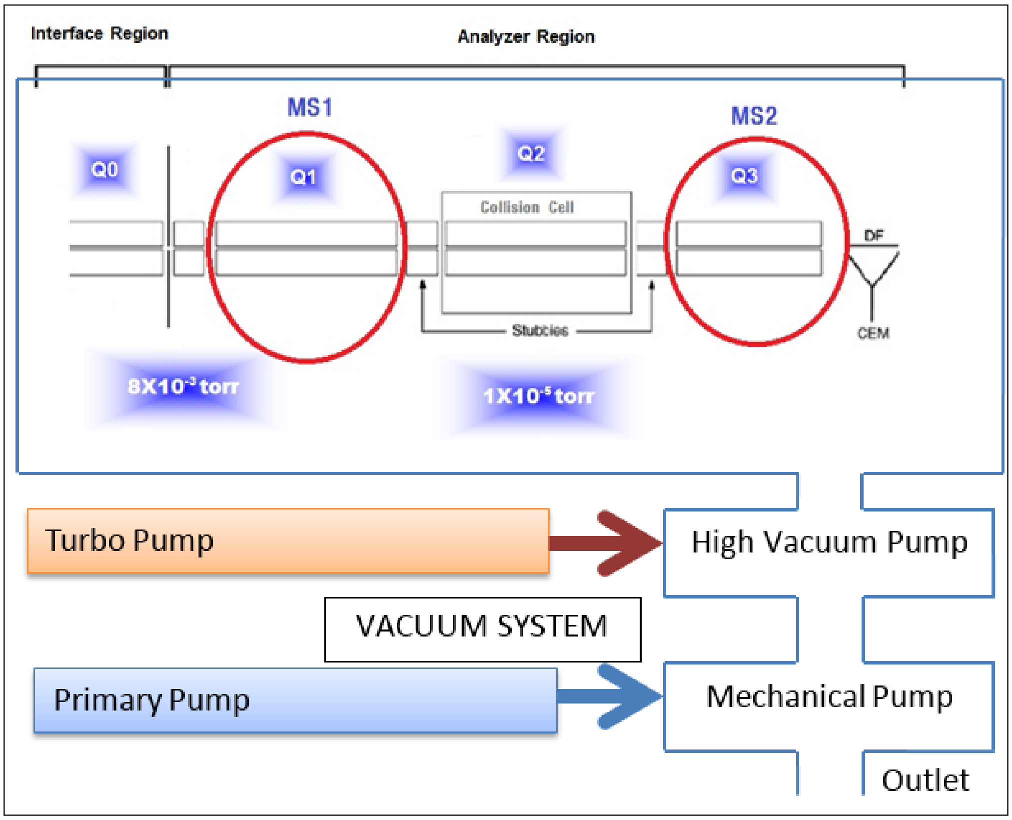
INTRODUCTION

We found that the IDP pump is more environmentally friendly and quieter than the rotary pump.

What's more it does not require oil refills and filter changes, whilst eliminating fume emissions and waste oil every 12 months, contributing to a lower cost of ownership. It also contributes to a quieter and more pleasant working environment, without the need for additional quiet covers or any other noise reduction devices. The purpose of this work was to assess whether the performance of the mass spectrometer is maintained or even improved with the IDP pump.

Triple quadrupole mass spectrometry is used for the structural determination of molecular ions or fragments. The ion of interest is selected by the first analyser (MS-1), collided with inert gas atoms in a collision cell, and the fragments produced separated by the second analyzer (MS-2). The mass spectrometer works under stringent vacuum conditions. This enables the analyte ions to move freely through the ion optics of the MS system. The presence of nitrogen and oxygen molecules would impair their movement, and could potentially cause undesired fragmentation.

A vacuum of 10^{-5} Torr ensures ions cover a mean free path ≈ 7m, which is considerably larger than any reasonable dimension of a quadrupole analyzer (0.5 m). Higher pump capacity is required on triple quadrupole systems due to the presence of additional gas molecules from the collision cell.



Schematic diagram of the vacuum system on an Agilent 7000 Series GC/MS-MS

The Agilent IDP-15 oil-free scroll vacuum pump is ideally suited for use in analytical instruments, such as GC/MS-MS. It is exceptionally quiet (<50 dBA) and oil free, unlike the conventional Edwards 5 rotary pump, which is noisier and requires oil exhaust replacement. The hermetic, isolated design of the Agilent IDP-15 pump provides rapid pump-down and a clean gas path through the pump, eliminating any risk of oil or grease contamination.

ANALYTICAL TECHNIQUE

STUDY 1: Performed by dtoLABS

Instrumentation

- Agilent 7890B GC and 7000C triple quadrupole MS system
- Injection mode: Pulsed Splitless
- Injection volume: 1 µL

Analytes

- Compounds: MCPA-thioethyl (MCPA-t), chlorothalonil, tebuconazole and deltamethrin
- Internal Standard – fenitrothion

STUDY 2: Performed by Research Institute for Chromatography (RIC)

Instrumentation

- Agilent 7890 GC and 7000C triple quadrupole MS system
- Injection volume: 1 µL and 2 µL

Analytes

- 16 EPA PAH compounds

RESULTS AND DISCUSSION

STUDY 1: Tuning & Vacuum

The MS detector must be tuned to ensure correct m/z alignment. We tuned it using PFTBA (perfluorotributylamine), which we introduced into the ion source by means of a valve. This compound produces a characteristic spectrum and some of these ions are used to set up the MS. The tune reports for the GC/MS-MS setup with the conventional Edwards 5 pump and with the IDP-15 scroll pump are shown in Table 1. Vacuum readings were similar on both systems. They are also well within the recommended limits set for water (< 20%) and air (oxygen < 2.5% and nitrogen < 10 %). Both systems passed system verification.

Parameter	Edwards 5 Pump	IDP-15 Scroll Pump
Rough Vacuum, mTorr	1.50E+2	1.48E+2
High Vacuum, Torr	8.36E-5	8.36E-5
Turbo Speed, %	100	100
Turbo Power, W	35.751	33.846
Water, % (<20%)	0.70	0.47
Oxygen, % (<2.5%)	0.15	0.12
Nitrogen, % (<10%)	0.56	0.46

Table 1. Comparison of tunes for both configurations.

We observed vacuum measurements at injection and during acquisition. We saw a spike in pressure during the initial few seconds of the run as a result of the pulsed splitless injection technique. However we was no differences between the vacuum gauge measurements of the two pumps.

Calibrations, Mass Spectra & Qualifier Ratios

We performed calibrations using both configurations, with calibration standards at 0.1, 1, 10, 50 and 100 ppb. Figure 1 compares the TIC chromatograms for both system setups. We made the calibrations on three nonconsecutive days, with eight replicate injections per standard. Table 2 provides summary of the calibration correlation coefficients, R2.

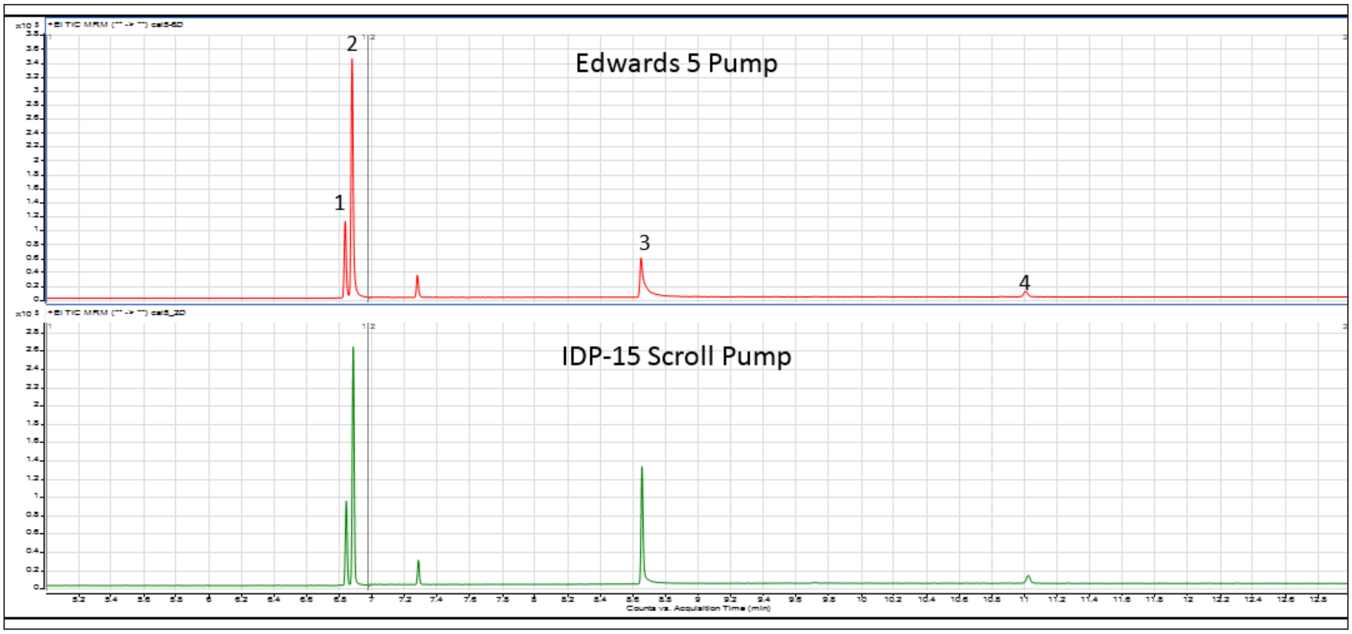


Figure 1. TIC Chromatograms using the Edwards 5 Pump and the IDP-15 Scroll Pump. Compounds are: [1] MCPA-t, [2] chlorothalonil, [3] tebuconazole and [4] deltamethrin.

	Edwards 5 Pump			IDP-15 Scroll Pump		
Compound	**Day 1**	**Day 2**	**Day 3**	**Day 1**	**Day 2**	**Day 3**
MCPA-t	0.999	0.998	0.996	0.999	0.999	0.998
Chlorothalonil	0.997	0.996	0.997	0.999	0.998	0.997
Tebuconazole	0.994	0.985	0.996	0.999	0.996	0.997
Deltamethrin	0.993	0.987	0.986	0.999	0.994	0.995

Table 2. Summary of correlation coefficients (R^2) obtained using both configurations. Calibration type 'weighted 1/x'.

Using the 10 ppb standard, we calculated the qualifier ratios of 2 MRM transitions for each compound. Figure 2 shows screenshots for the MRM transition of 243.9 - 154.9 for MCPA-t using both pumps. The ratios for all 4 compounds, along with the percentage differences between pumps, are tabulated in Table 3. The differences were all within the range of 0 - 10%.

Edwards 5 Pump

IDP-15 Scroll Pump IDP-15 Scroll Pump

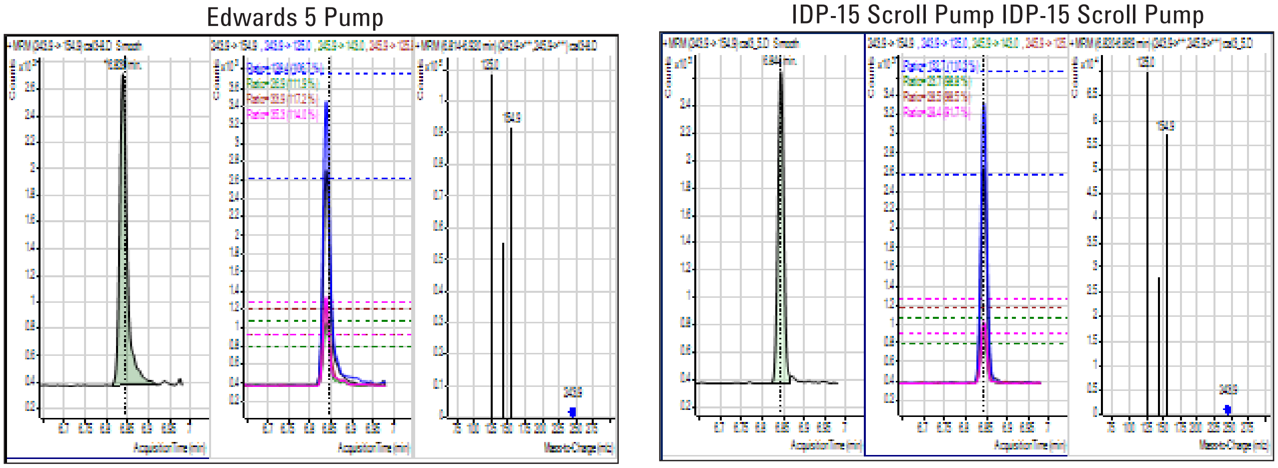


Figure 2. MRM transition of 243.9 – 154.9 for MCPA-t at 10ppb.

Compound	MRM Transition	Edwards 5 Pump	IDP-15 Scroll Pump	% Difference
MCPA-t	243.9 – 125.0	130	126	3
	245.9 – 143.0	25	25	0
Chlorothalonil	265.7 – 231.0	90	92	-1.5
	265.7 – 132.9	171	184	-10
Tebuconazole	249.9 – 153.0	26	31	-4
	125.0 – 89.0	220	220	0
Deltamethrin	252.7 – 92.9	204	191	10
	252.7 – 173.7	101	99	2

Table 3. Qualifier ratios and percentage differences for four compounds (two MRM transitions per compound).

Repeatability and signal-to-noise ratios

We calculated interday repeatability by analyzing eight replicate injections of the 10 ppb standard, on three nonconsecutive days. Table 4 summarizes the results, reported as absolute areas. We observed no significant differences in RSD percentage for the four compounds. Table 5 shows the ratio of signal to noise for each of the four compounds. in all four cases, the ratio was better when using the IDP-15, which should equate to greater sensitivity.

	Edwards 5 Pump			IDP-15 Scroll Pump		
Compound	Absolute Area	SD	% RSD	Absolute Area	SD	% RSD
MCPA-t	2803	125	4	2560	205	8
Chlorothalonil	5897	366	6	4394	306	7
Tebuconazole	2099	163	8	2567	227	9
Deltamethrin	356	48	13	358	50	14

Table 4. Interday repeatability results showing absolute areas, SD and % RSD for both pumps.

Compound	Standard	S/N - Edwards 5 Pump	S/N – IDP-15 Scroll Pump
MCPA-t	1 ppb	70	85
Chlorothalonil	1 ppb	138	174
Tebuconazole	1 ppb	59	71
Deltamethrin	50 ppb	105	126

Table 5. Signal-to-noise ratios using both configurations.

Sample analysis

To determine the RSD of the method, we used both configurations to analyze a spiked solution containing known concentrations of MCPA-t, chlorothalonil, and deltamethrin. Table 6 shows the results (expressed as concentrations) of analyzes we performed on three nonconsecutive days. We observed peak tailing for chlorothalonil on the Edwards 5 pump, which probably explains the low result. Overall, the results are good, with all RSDs being less than 15%. However, there was a noticeable difference between the recovery figures, with the Agilent IDP-15 pump demonstrating better accuracy for all compounds.

Compound	Expected Concn, ppb	Edwards 5 Pump			IDP-15 Scroll Pump		
		Result, ppb	% RSD	% Recovery	Result, ppb	% RSD	% Recovery
MCPA-t	50	43	5	86	53	8	106
Chlorothalonil	50	39	2	78	55	9	110
Deltamethrin	100	93	10	93	98	1	98

Table 6. Spiked sample results for MCPA-t, chlorothalonil and deltamethrin, analysed on three non-consecutive days.

STUDY 2

Tuning & Vacuum

Before installing the IDP-15 pump, we fitted the GC/MS instrument with a new capillary column and liner, then performed an MS autotune. We then vented system and opened the vacuum chamber door on the MS (Q1 only) to expose the ion source to atmospheric pressure. This action simulates that performed during source removal/cleaning. We then replaced the Edward 5 pump with the Agilent IDP-15 pump, using the same vacuum hose line. Finally, we initiated pump-down was and performed our tests. Afterward, we vented the instrument, reinstalled the Edwards 5 pump, and repeated the tests.

The installation and start-up of the IDP-15 pump was achieved without any problems. Immediately after initiating pump-down, the pump produced a high pitched noise, which disappeared after a few seconds. The vacuum chamber of the MS (quadrupole 1) firmly closed with the foreline vacuum alone and no real pushing was required. In contrast, the vacuum chamber was less firmly closed with the Edwards 5 pump and the door typically required some pushing. Vacuum readings taken at 15 and 45 minutes using both pumps were similar and autotunes performed after 90 minutes were also comparable.

An EPA PAH test mixture was analyzed in scan mode using both pumps at two different flow rates: 1mL/min (normal) and 2 mL/min (high). Figure 3 and 4 show the resulting total ion chromatograms. We observed no significant differences between the results obtained with either pump. We then compared mass spectra in scan mode. Figure 5 compares the mass spectra of pyrene on both systems at 1 mL/min and 2 mL/min. No appreciable differences were observed at either flow rate. From these results, we concluded that similar analytical performance can be achieved with both pumps.

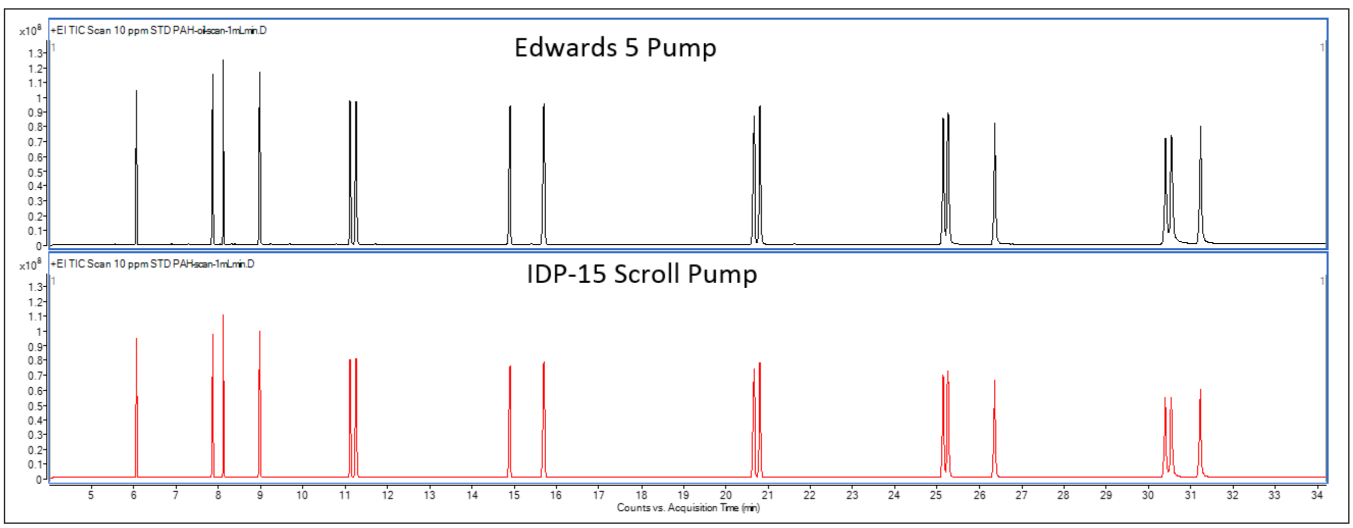


Figure 3. Analysis of 16 EPA PAH compounds at 1mL/min in Scan Mode.

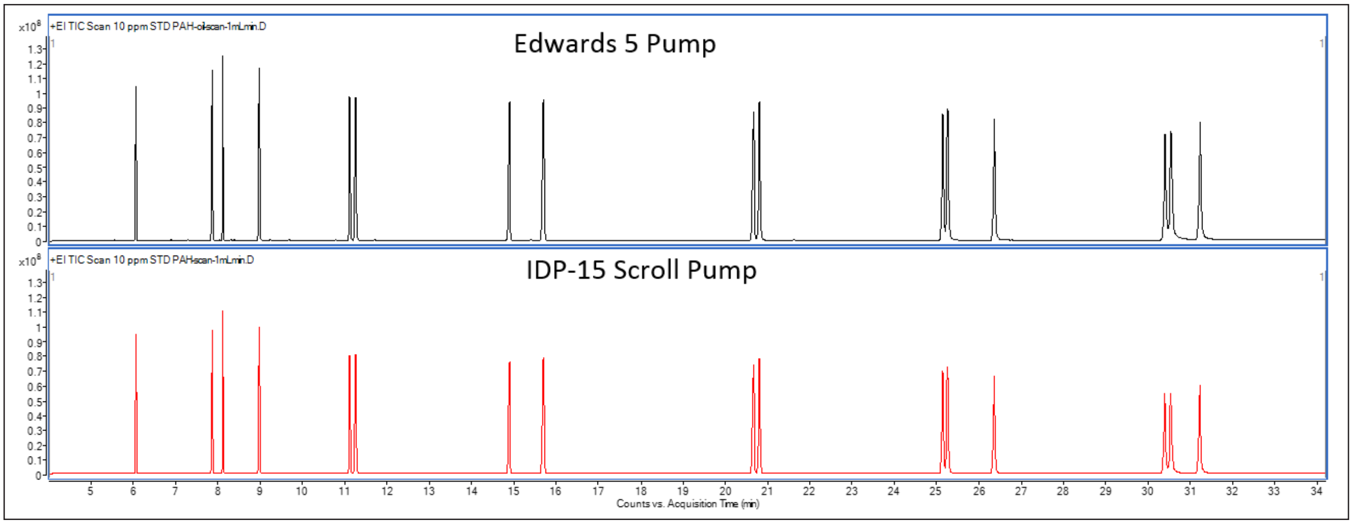


Figure 4. Analysis of 16 EPA PAH compounds at 2mL/min in Scan Mode.

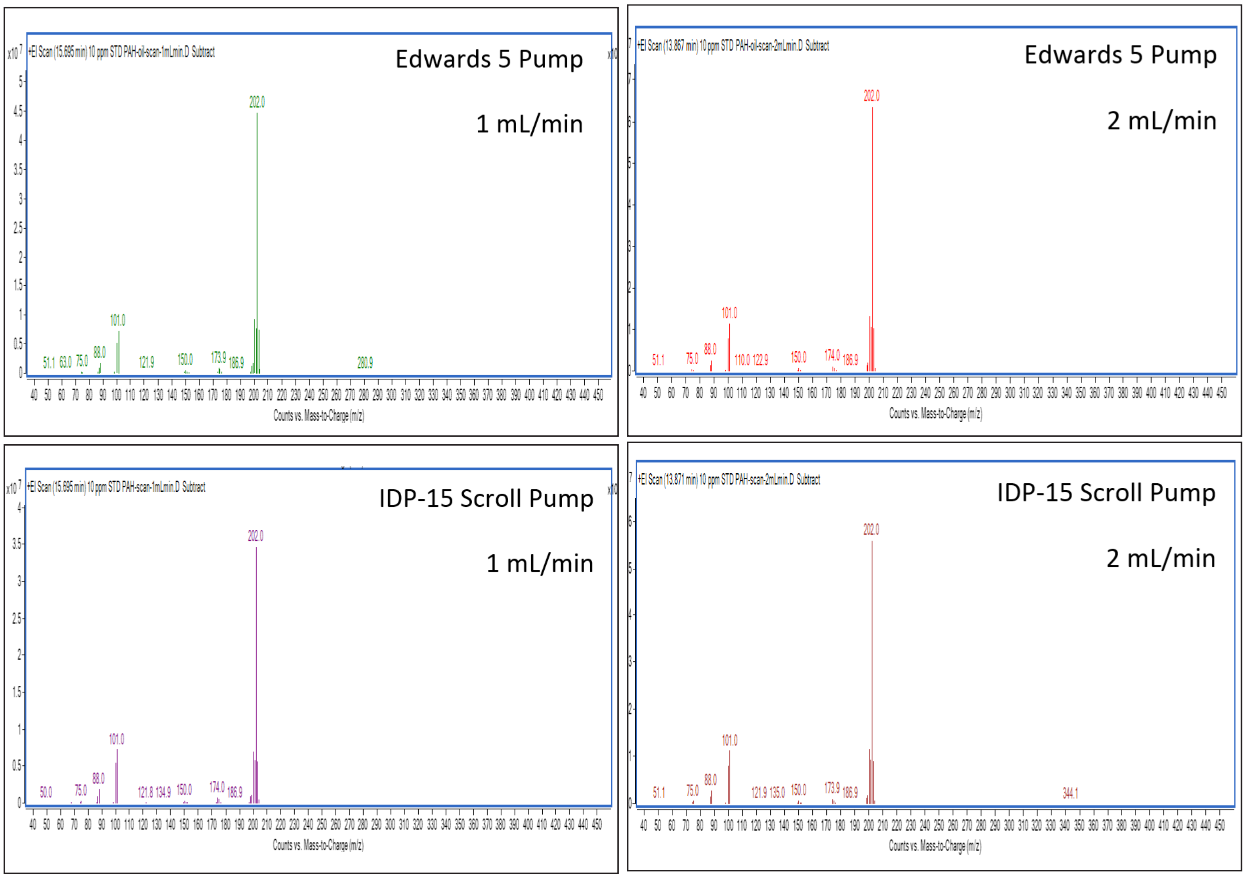


Figure 5. Comparison of pyrene mass spectra using both configurations at 1 mL/min and 2 mL/min.

CONCLUSIONS

The analytical performance of the IDP-15 Oil-Free pump was assessed against the conventional Edwards 5 oil pump through two independent studies. Vacuum readings using both pumps were comparable, as were tune parameters.

Analytical performance was also found to be similar and in Study 1, the IDP-15 pump configuration demonstrated better signal-to-noise ratios, as well as improved accuracy on the spiked sample. These preliminary studies suggest that similar, if not possibly better, analytical performance can be achieved using the IDP-15 pump. Further work would need to be undertaken to confirm this, but these initial results are very promising. In addition, this pump is more environmentally friendly and quieter.

Agilent IDP-15 Oil Free Pump (Part NumberX3815-64010) is available as oil-free upgrade solution in the aftermarket. The pump package includes an isolation valve, a fundamental requirement to save the turbo falls of violent air in case of a system shutdown. Tests are ongoing to qualify the pump on other Agilent tools and become a standard option of the new system