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Short communication

Direct sensitive simultaneous determination of fluorinated benzoic acids in oil reservoir waters by ultra high-performance liquid chromatography–tandem mass spectrometry

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ABSTRACT

A direct ultra-high performance reverse-phase HPLC (UHPLC) – electrospray MS/MS method was developed for the simultaneous determination of 16 fluorinated benzoic acids (FBAs) in oil reservoir waters. The separation was achieved within 5 min in a non-linear gradient mode using a 1-ml sample aliquot. The method detection limits were in the lower ng/ml range (between 0.05 and 50 ng/ml, depending on the compound) owing to the use of the travelling-wave collision cell technology. The method developed was more sensitive, faster (by avoiding sample preconcentration and purification steps) and more robust than the GC/MS methods currently used in oil industries. The accuracy of the method was verified by comparison with GC/MS results. It was applied to the determination of FBAs in water samples coming from reservoir tracing campaigns.

1. Introduction

Fluorinated benzoic acids (FBAs) are the most widely used tracers in campaigns aimed at the description of oil reservoirs [1,2]. As they are injected in low quantities, ultra-sensitive analytical techniques are required for their determination. The current reference analytical technique is gas chromatography with MS detection (GC/MS) which offers detection limits of 10–100 pg/ml [3,4]. However, the analysis time is long (60 min) and the procedure requiring a complex sample preparation (clean-up, derivatization) is tedious and error-prone (losses or contamination) [5].

The two most commonly used methods for benzoic acids are high-performance liquid chromatography (HPLC) and gas chromatography (GC) [6,7]. As FBAs are nonvolatile and polar, HPLC offers an advantage of the elimination of the need for derivatization and thus of a potentially faster direct analysis. However, the reported HPLC detection limits are three orders of magnitude higher (UV detection) [8–12] and one order of magnitude higher (MS–MS detection) [13] than those reported by GC–MS. Ways to improve them include the use of ultra high-performance liquid chromatography (UHPLC) [14–16] and the use of the latest generation mass spectrometers.

The objective of this work was to develop a method for the rapid determination of FBA traces in oil reservoir waters matching the sensitivity of the state-of-the-art GC methods [3,4] but avoiding the tedious sample preparation. A compromise needed to be found between peak capacity, chromatographic resolution, sensitivity, matrix effect and analysis time. This was achieved by the combination of UHPLC with multiple reaction monitoring (MRM) MS using travelling-wave collision cell technology (without and with scan-wave mode). This new type of mass analyzer uses a travelling voltage wave on which ions can surf maximizing analysis speed and sensitivity. The signal intensity is further improved in scan-wave mode because the ions are accumulated before being separated according to their mass-to-charge (m/z) ratio [17].

2. Experimental

2.1. Reagents and chemicals

19 FBA standards (purity > 97%) (Table 1) were purchased from Apollo Scientific (Denton, Manchester, UK). The chemicals: acetonitrile (CH_3CN, Fluka, LC/MS, 99.9%), acetic acid (CH_3COOH, glacial,

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Table 1
MS/MS optimized conditions for the Xevo TQ MS instrument.

Compound	Chemical formula	Nominal mass (g/mol)	MRM transition	Cone voltage (V)	Collision energy (eV)
2-FBA	$C_7H_5O_2F$	140	138.84 > 94.97	17	10
3-FBA			138.84 > 94.97	17	10
4-FBA	$C_7H_4O_2F_2$	158	138.84 > 94.97	17	10
2,3-dFBA			156.84 > 112.97	16	10
2,4-dFBA	$C_7H_3O_2F_3$	176	156.84 > 112.97	16	10
2,5-dFBA			156.84 > 112.97	16	10
2,6-dFBA	$C_8H_5O_2F_3$	190	156.85 > 112.97	16	10
3,4-dFBA			156.85 > 112.97	20	14
3,5-dFBA	$C_7H_2O_2F_4$	194	156.85 > 112.97	20	12
2,3,4-tFBA	$C_9H_4O_2F_6$	258	174.84 > 130.95	14	10
2,3,6-tFBA			174.84 > 130.95	14	10
2,4,5-tFBA			174.84 > 130.95	14	10
2,4,6-tFBA			174.84 > 130.95	14	10
3,4,5-tFBA			174.85 > 130.95	20	14
2-tFmBA			188.90 > 144.97	22	14
3-tFmBA			188.90 > 144.97	22	14
4-tFmBA			188.90 > 144.97	22	14
2,3,4,5-tetraFBA			193.00 > 148.95	14	8
3,5bis-tFmBA			257.00 > 212.90	26	18

Abbreviations: F, fluoro; B, benzoic; A, acid; d, di; t, tri; tFm, trifluoromethyl.

Riedel-de Haën, 100%), formic acid (HCOOH, Fluka, 98%, MS grade), sodium hydroxide (NaOH, Rectapur, Prolabo, 98% min), ammonium bicarbonate (NH_4HCO_3, ReagentPlus, 99%). were purchased from Sigma–Aldrich (Saint-Quentin, Fallaviers, France). Ultra-pure water (18.2 MΩ cm) obtained from a Millipore system (systems Elix 3 and Advantage, Millipore, Saint-Quentin, France) was used throughout.

2.2. Standards and samples

Standard solutions (300 μg/ml) were prepared by dissolving between 14.0 and 25.8 mg (accurately weighed) of a FBA in 40 ml water and 10 ml 3975 μg/ml NH_4HCO_3 solution. They were stored at 4 °C in the dark up to four months (the stability of their concentrations after four months of storage was confirmed within 10%). Working solutions were prepared by appropriate dilution of the stock solutions with water. Reservoir water samples spiked with a mixed standard solution at different concentrations (between 500 ng/ml and 100 pg/ml) were used for the method development. The analysed samples were oil reservoir waters from three tracing campaigns.

The samples were filtered through a GHP Acrodisc 13-mm syringe filter (0.2 μm GHP membrane, Pall Life Sciences, Interchim, France). The samples showed low to middle salinity (<10 g/L equivalent NaCl). In the case of higher salt content, samples should be diluted with 0.1% HCOOH prior to filtration in order to avoid the formation of a salt deposit on the cones and signal suppression [16].

2.3. Apparatus

An Acquity UPLC system (Waters Corp., Milford, MA) including a binary solvent pump, a cooled autosampler, an Acquity UPLC BEH C_{18} column, 50 mm × 2.1 mm (1.7 μm particles, Waters) with a matching Vanguard precolumn was used.

The detectors were: a diode-array UV detector (Acquity) used at 265 nm, a TQD (quadrupole–hexapole–quadrupole in T-wave mode) (Waters, Milford, MA) or XevoTQ (quadrupole-T–wave-quadrupole in scan wave mode) MS with an orthogonal Z-spray–electrospray interface (Waters). A hybrid mass spectrometer ESI-QTOF (QSTAR XL, Sciex, ON, Canada) was used in the method development.

2.4. Procedures

2.4.1. Chromatographic separation

The parameters studied included: mobile phase composition (H_2O/CH_3CN containing 0.1% of HCOOH or CH_3COOH), LC-gradients and columns (Acquity UPLC BEH C_{18} column: 2.1 mm × 50 mm, particle size 1.7 μm, and Acquity BEH HILIC column: 1 mm × 150 mm, 1.7 μm). Because of the target analytes and the detection in ESI_{neg} mode, it was decided not to work with an ion-pairing agent, such as trifluoroacetic acid (TFA) or heptafluorobutyric acid (HFBA).

Mobile phase was a mixture of water (A) and acetonitrile (B), both containing 0.1% HCOOH. The elution gradient (non-linear hyperbole) was: 0 min (5% B), 0.2 min (10% B), 1.8 min (28% B), 2.5 min (80% B), 3.2 min (80% B), and 4 min (5% B) for 1 min. The gradient steps after 2.5 min served to clean and re-equilibrate the column to guarantee the repeatability of the analysis. Total analysis time was 5 min, column equilibration included. The injected volume was 10 μL (or more if a sample was diluted). The flow rate was 0.85 ml/min, the column temperature was 45 °C and the autosampler temperature was 5 °C.

2.4.2. Mass spectrometric conditions

MS/MS data acquisition was performed with the electrospray source operating in negative mode (ESI_{neg}) under the MRM conditions listed in Table 1. The MS parameters were optimized for each instrument (see Supplementary Data file and detailed procedure described elsewhere [18]). The optimized values for the Xevo TQ MS instrument were: capillary voltage 2.50 kV; source temperature 150 °C; desolvatation temperature 400 °C; extractor voltage 3 V; RF lens 0.4 V. Nitrogen was used as both the nebulizing gas and the desolvatation gas. Cone gas and desolvatation gas flows were set at 20 L/h flow and 1000 L/h respectively. Argon was used as collision gas with a pressure of 2×10^{-3} mbar in the T-wave cell. Dwell times of 0.010 s/scan for 2,3,4,5-tetraFBA and 3,5bis-tFmBA and 0.017 or 0.025 s/scan for the other FBAs, were selected. The Masslynx software (Waters Corp., Milford, MA) was used to process data. Quantification was based on peak area.

3. Results and discussion

3.1. Choice of chromatographic conditions

Acetonitrile was selected owing to its lesser toxicity than methanol. Despite the FBAs pK_a values being below 4, it was

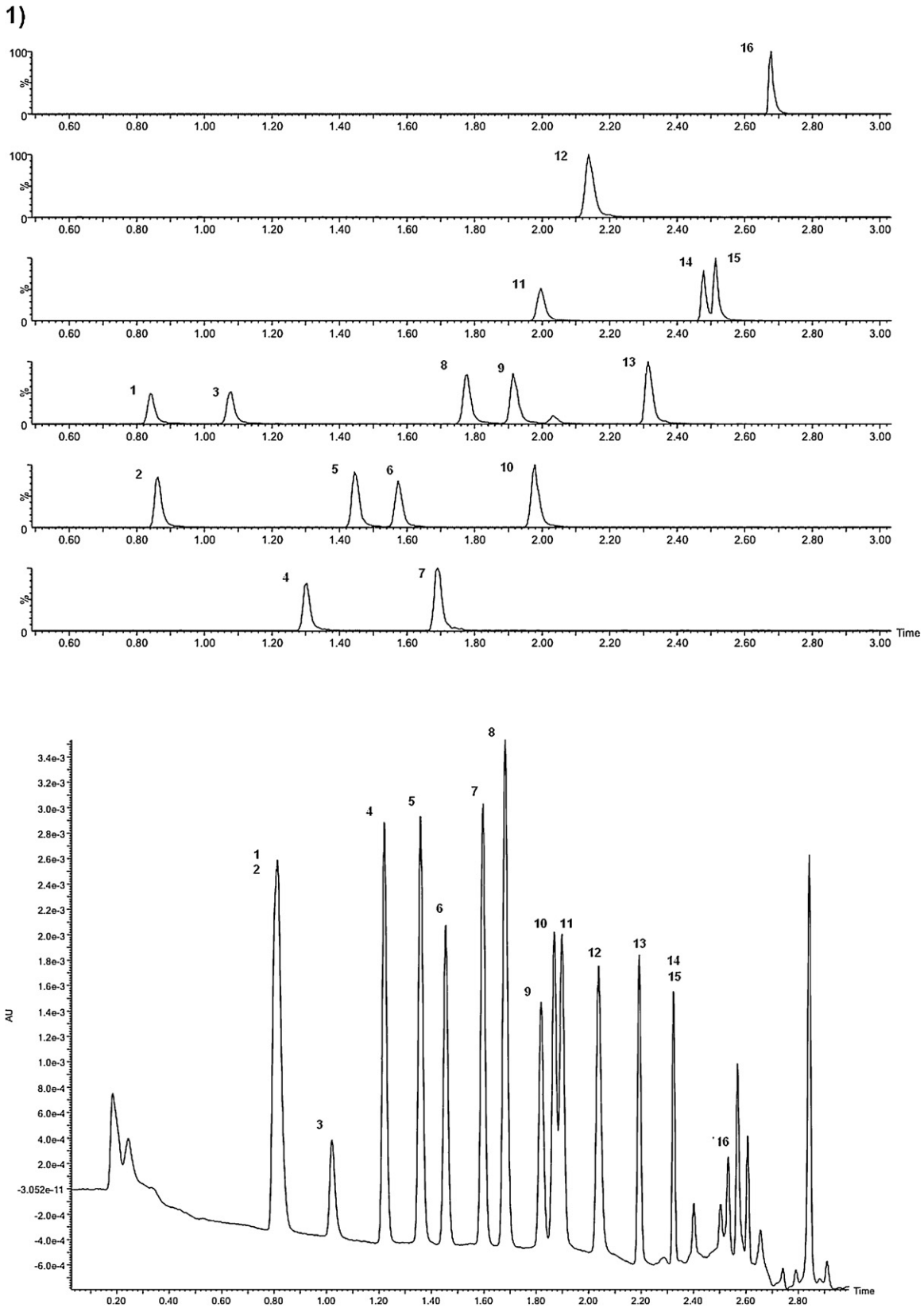

Fig. 1. Simultaneous detection of the 19 FBAs by UHPLC/UV/MS–MS. Spiked water sample with FBAs at about and 0.5 ppm and 85.0 ppb, respectively.

a low pH (1–2 units lower than pK_a set with HCOOH) that leads to higher S/N for most of the compounds in the ESI_{neg} mode (*cf.* SD file) [13,19]. Note that as the FBAs anionic forms are less retained during reversed phase chromatography, the ionization in less concentrated CH_3CN was less efficient. A post column addition of a base (ammonium bicarbonate) was tested but the benefit did not set off the complexity of the manifold.

Table 2
Method detection and quantification limits (MDL, MQL)[d]..

	Water			Reservoir water A	Reservoir water B	Reservoir water C
	MDL–MQL (ng/mL)[a]	MDL–MQL (pg/mL)[b]	MDL–MQL (pg/mL)[c]	MDL–MQL (ng/mL)[a]	MDL–MQL (ng/mL)[a]	MDL–MQL (ng/mL)[a]
2-FBA	1.7–5.6	90–297	–	4.6–15.2	6.9–22.8	6.4–21.1
3-FBA	0.5–1.7	150–495	86–284	1.3–4.3	1.1–3.6	1.7–5.6
4-FBA	1.0–3.3	500–1650	180–594	1.2–3.9	1.5–4.9	1.8–5.9
2,3-dFBA	0,7–2.3	20–66	50–165	1.4–4.6	2.0–6.6	0.8–2.6
2,4-dFBA	0.6–2.0	90–297	23–76	1.6–5.3	0.9–2.9	0.9–2.9
2,5-dFBA	0.6–2.0	500–1650	68–224	1.4–4.6	1.0–3.3	1.7–5.6
2,6-dFBA	0.8–2.6	30–99	–	8.4–27.7	12.0–39.6	7.3–24.1
3,4-dFBA	0.3–1.0	40–132	20–66	0.6–1.9	0.6–1.9	0.2–0.7
3,5-dFBA	0.6–2.0	35–115	22–73	0.6–1.9	1.1–3.6	0.2–0.7
2,3,4-tFBA	0.8–2.6	500–1650	310–1023	14.0–46.2	10.0–33.0	4.9–16.2
2,3,6-tFBA	1.7–5.6	3000–9900	960–3168	57.0–188.1	61.0–201.3	71.0–234.3
2,4,5-tFBA	0.8–2.6	1000–3300	650–2145	14.0–46.2	8.0–26.4	4.8–15.8
2,4,6-tFBA	1.7–5.6	300–990	–	57.0–188.1	76.0–72.7	21.0–69.3
3,4,5-tFBA	0.8–2.6	900–2970	290–957	1.4–4.6	1.1–3.6	1.4–4.6
2-tFmBA	0.5–1.7	100–330	72–238	0.3–0,9	0.8–2.6	1.0–3.3
3-tFmBA	0.2–0,7	30–99	39–129	0.4–1.3	0.3–0.9	0.3–0.9
4-tFmBA	0.3–1.0	100–330	31–102	0.8–2.6	0.3–0.9	0,2–0.7
2,3,4,5-tetraFBA	0.7–2.3	700–2310	240–792	8.5–28.0	2.8–9.2	1.8–5.9
3,5bis-tFmBA	0.06–0.20	3.30–10.90	0.40–1.30	0.13–0.43	0.04–0.13	0.03–0.10

"–" Compound not studied.
[a] TQD instrument, 15 μL injected.
[b] Xevo TQ instrument, 15 μL injected.
[c] Xevo TQ instrument, 50 μL injected.
[d] The method detection limit and quantification limit were defined and determined as the minimum detectable amount of analyte from waters spiked extract in MRM mode with a signal-to-noise ratio (S/N) of 3:1 and 10:1, respectively [22].

Table 3
Effect of the calibration on the accuracy of multitracer UHPLC/MS–MS analysis. Analyses of samples spiked at 6 and 30 ng/ml.

External calibration							Standard addition		
Matrices	H_2O		Reservoir water A		Reservoir water A		H_2O	Reservoir water A	Reservoir water A
	Target *X* (ng/mL)[a]	Found *X* (ng/mL)[b]	Target *Y* (ng/mL)	Found *Y* (ng/mL)	Target *Z* (ng/mL)	Found *Z* (ng/mL)	Found *X* (ng/mL)	Found *Y* (ng/mL)	Found *Z* (ng/mL)
2-FBA	6.2 ± 0.1	5.9	6.1 ± 0.1	5.9	32.3 ± 0.7	31.5	6.0	6.1	33.0
4-FBA	6.0 ± 0.1	6.0	5.9 ± 0.1	5.9	31.5 ± 0.7	31.5	6.2	6.0	31.6
2,4-dFBA	6.1 ± 0.1	6.2	6.0 ± 0.1	6.0	31.9 ± 0.7	32.1	5.9	6.1	32.4
2,5-dFBA	6.3 ± 0.1	6.2	6.2 ± 0.1	6.2	32.8 ± 0.7	33.9	6.1	6.3	33.8
2,6-dFBA	6.1 ± 0.1	6.0	6.0 ± 0.1	5.9	32.1 ± 0.7	32.4	5.9	5.8	31.3
3,4-dFBA	6.1 ± 0.1	6.1	6.0 ± 0.1	5.9	31.8 ± 0.7	32.6	6.0	6.1	32.3
2,3,4-tFBA	6.2 ± 0.1	6.2	–	–	32.6 ± 0.7	32.4	6.2	–	32.9
2,4,5-tFBA	6.1 ± 0.1	6.1	–	–	31.9 ± 0.6	33.0	6.0	–	32.5
3,4,5-tFBA	6.1 ± 0.1	6.2	6.0 ± 0.1	5.8	31.9 ± 0.6	32.0	6.2	6.0	32.3
2-tFmBA	6.1 ± 0.1	6.0	6.0 ± 0.1	6.0	31.9 ± 0.6	31.9	6.1	6.1	32.2
4-tFmBA	6.7 ± 0.1	6.3	6.6 ± 0.1	6.5	35.1 ± 0.7	34.7	6.7	6.7	35.8
2,3,4,5-tetraFBA	6.1 ± 0.1	6.0	–	–	32.7 ± 0.6	33.8	6.0	–	33.7
3,5bis-tFmBA	6.3 ± 0.1	6.2	6.0 ± 0.1	6.3	32.0 ± 0.6	30.8	6.1	6.2	32.2

"–"Compound not studied.
[a] Target concentration calculated with its uncertainty associated to the preparation of the standard solutions (weight, dilution).
[b] Average found concentration for each compound, determined with $n = 3$ (RSD < 8%).

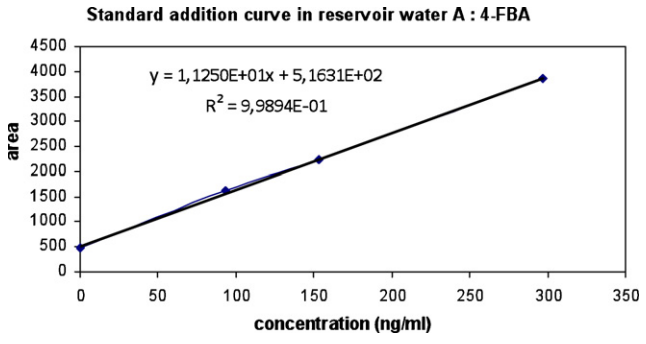

Fig. 2. Example of standard addition curve in reservoir water A for the 4-FBA.

The reversed-phase and HILIC columns were compared by using the MS parameters given in Section 2.4.2. The UPLC C_{18} column allowed an efficient chromatographic separation in a short time and a better sensitivity (with a 2-s peak width at half-height in comparison with 25 s for the HILIC column) (*cf.* SD file). Even if the separation of all the FBAs was possible with the HILIC column, the peaks were larger, the sensitivity was lower and the analysis time was longer. Therefore, the UPLC C_{18} column was chosen. In the optimized conditions given in Section 2.4.1, 16 FBAs could be detected and quantified simultaneously in only 5 min following a direct injection which makes this method suitable for high-throughput monitoring (Fig. 1). Note that the separated compounds are generally the most frequently used in tracing campaigns [20,21]. Indeed, 2,3-dFBA, 3,5-dFBA, 2,3,6-tFBA, 2,4,6-tFBA, are rarely used owing to their high costs.

Table 4
Multi-tracer analysis of real water samples coming from tracing campaigns. Comparison of UHPLC-MS/MS (Xevo TQ MS) values with the reference GC–MS values.

Field	Sample no.	Detected and quantified compound	Concentration (ng/ml)	
			This method[a]	GC–MS method
Real water samples				
A	1	4-FBA	4.6 ± 0.3	4.9
	2	4-FBA	14.0 ± 1.2	18.0
	3	4-FBA	10.0 ± 1.3	15.0
D	4	2-FBA	130.0 ± 15.5	160.0
	5	2-FBA	130.0 ± 13.8	150.0
E	6	2,5-dFBA	16.0 ± 1.5	11.0

[a] Uncertainty determined with $n = 3$ (two-sided 95% confidence intervals).

3.2. Optimisation of the MS/MS detection

Several ionization modes: electrospray ionization [22] in negative (ESI_{neg}) and positive mode (ESI_{pos}) and atmospheric pressure chemical ionization in negative mode ($APCI_{neg}$) were tested. The highest S/N was obtained in the ESI_{neg} mode.

Prior to MS/MS analyses, daughter ions of FBAs were identified by ESI-QqTOF MS with direct infusion of individual compounds (1.4 μg/ml at 20 μl min^{-1}). The choice of MRM transition was confirmed by triple quadrupole measurements (ESI-QqQ MS). The most abundant MRM between the precursor ion and the product ion corresponded to the loss of fragment COO ($m/z = 44$). The optimum transitions monitored were summarized in Table 1. The T-wave collision cell technology allowed the reduction of dwell times, thus increasing the number of transitions in the typical short transient signal from UHPLC without any significant effect on sensitivity. The FBA isomers could not be differentiated by MS–MS as their fragmentation patterns were similar. The same MS parameters were thus selected for the isomers.

3.3. Figures of merit

The performance of the method was evaluated in terms of the detection limits, linearity, repeatability, selectivity and robustness in waters having different physico-chemical characteristics (different salinity): MilliQ water and real-reservoir waters (A, B and C). The method detection limits (MDL) are compound and matrix dependant. They were typically at the ng/ml level for reservoir waters with the TQD instrument and a factor of 10 lower with the Xevo TQ MS instrument (Table 2). The sensitivity increased with a decrease of the compound polarity (and increase in the partitioning coefficients) (*cf.* SD file). The highest MDLs were observed for the more polar compounds eluted close to the void of the column and quasi-simultaneously with the salts of reservoir waters.

Satisfactory linearity ($r^2 > 0.995$) was observed for all the compounds regardless of the matrix. The studied and validated calibration linear ranges were depending on the compound: for instance, it went from 100 pg/ml to 500 ng/ml for 3,5-bis-tFmBA.

The retention times were reproducible for the high salinity reservoir water within 1% (<1 s) and peak areas within 10% (RSD, $n = 5$, spiked concentrations relevant to real water samples: 200 and 20 ng/ml). The highest RSD (5–8%) values were observed for the four least retained compounds eluted close to the void of the column.

The positive identification of the target analytes in samples was done by the combination of a specific MS/MS transition and the RT of the compound. These allowed the elimination of the risk of false positive results. The RT of the analytes analysed in an aqueous standard mixture and spiked in a real-world sample matrix agreed within ±2 s regardless of the compound and the matrix.

This analytical column also showed a good robustness having served for the analysis of 600 real reservoir water samples (ca. 4000 injections) over the period of one year. The retention times of all the compounds (injected as a mixture (20 ng/ml) in a synthetic brine, 10 g/L, eq. NaCl) were stable within 3%.

The main sources of uncertainty were identified to be the sampling and the medium (reservoir water) (*cf.* the Ishikawa diagram in the SD file).

3.4. Recovery experiments and method accuracy

The recovery of the method was established by analyzing different water samples spiked with the known amounts of the FBA standards. Two quantification methods were compared, external calibration (six-point calibration curve, from 2 to 48 ng/ml) and standard addition (four-point calibration curve – example for samples at 30 ng/ml, final concentrations values: 0, 94, 150, and 300 ng/ml, see Fig. 2). The results for H_2O and reservoir water A presented in Table 3 show good recoveries regardless of the compound and the matrix. Consequently, for low-salinity reservoir waters (<10 g/L eq. NaCl), external calibration was suitable. For higher salinity reservoir waters (reservoir waters B and C), the standard addition method was required to correct for the matrix effect (ca. 20%).

The accuracy of the method was verified by comparison of the results obtained with the values obtained independently by GC–MS and used as reference. The principles of the GC–MS method are described elsewhere [4]. The GC–MS method was validated by a round-robin exercise involving several commercial analytical laboratories offering this type of analyses to oil companies. MDL and MQL depend on the compound and the matrix and are typically in the range 0.15–0.50 ng/ml and 0.45–1.50 ng/ml, respectively.

Table 4 shows a satisfactory agreement between the UHPLC–MS/MS and GC–MS data (analytical methods completely independent). Note that the number of tracers was limited to those broken through the reservoir at the given sampling site.

4. Conclusion

UHPLC/MS–MS is an attractive alternative to GC–MS allowing a rapid simultaneous determination of FBAs in waters at the ng/ml level. With a minimum sample manipulation (only a 0.2 μm filtration), it competes favorably with GC–MS in terms of accuracy, repeatability, sensitivity, selectivity, robustness and analysis throughput. The problems which occurred during sample preparation for GC–MS, such as losses of some compounds during extraction steps in some high salinity reservoir waters or cross contamination during SPE extraction were solved by direct injection of reservoir waters. The UV detection is also interesting for on site monitoring to optimize the sampling procedure and before any MS/MS confirmation and quantification.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.chroma.2011.06.028.

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