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Data Article

Data comparing the separation and elution of vitamin D metabolites on an ultra performance supercritical fluid chromatography tandem-mass spectrometer (UPSFC-MS/MS) compared to liquid chromatography (LC) and data presenting approaches to UPSFC method optimization

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ABSTRACT

The data presented is related to the research article "Analysis of multiple vitamin D metabolites by ultra performance supercritical fluid chromatography-tandem mass spectrometry (UPSFC-MS/MS)" (Jenkinson et al., 2018) [1]. This article will include data obtained from method development, optimization and analysis of multiple vitamin D metabolites on an ultra performance supercritical fluid chromatography tandem-mass spectrometry (UPSFC-MS/MS). This includes chromatograms from column screening to confirm the most suitable column for analyte separation. Additionally, further chromatograms and figures compare separation and analyte signal strength during the optimization of other UPSFC parameters. Mass spectra will demonstrate the optimization of MS conditions for the UPSFC-MS/MS method. Chromatogram data from UHPLC vitamin D analysis is also presented in order to compare the separation and elution of vitamin D metabolites using

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UPSFC and UHPLC. This data will highlight the outputs that aid in method development and identifying the separation technique suited for vitamin D quantitation.

Specifications Table

Subject area	*Analytical Chemistry*
More specific subject area	*Vitamin D and supercritical fluid chromatography*
Type of data	*Chromatograms* *Mass spectra* *Figures*
How data was acquired	*Method development for the separation and analysis of vitamin D metabolites was performed on Waters ACQUITY UPC^2 and Waters ACQUITY UPLC coupled to a Waters Xevo TQ-MS mass spectrometer.*
Data format	*Raw and analyzed*
Experimental factors	*Working standards of vitamin D were prepared in methanol for UPSFC-MS/MS analysis and methanol/water (50/50%) for UPLC-MS/MS analysis.* *Derivatization of vitamin D metabolites was performed using 4-Phenyl-1,2,4-triazole-3,5-dione (PTAD) and 4-[2-(3,4-Dihydro-6,7-dimethoxy-4-methyl-3-oxo-2-quinoxalinyl)ethyl]-3H-1,2,4-triazole-3,5(4H)-dione (DMEQ-TAD).*
Experimental features	*Comparison of vitamin D metabolite elution and separation between UPSFC and UPLC.* *Optimization of UPSFC parameters for separation and detection of vitamin D.*
Data source location	*Birmingham, United Kingdom.*
Data accessibility	*Data is with this article*
Related research article	*C. Jenkinson, A Taylor, K. Storbeck, M. Hewison. Analysis of multiple vitamin D metabolites by ultra performance supercritical fluid chromatography-tandem mass spectrometry (UPSFC-MS/MS). Journal of Chromatography B. 2017.*, 1087–1088 (2018), pp.43–48. doi: 10.1016/j.jchromb.2018.04.025

Value of the data

- The direct comparison between optimized UPSFC and UPLC methods could provide an insight into which separation technique is best suited for routine analysis of vitamin D and other similar small molecules.
- The data presented from the UPSFC-MS/MS method development and optimization provides a benchmark for future method development approaches using this platform.
- The analytical methods presented incorporate the analysis of multiple active and inactive vitamin D forms across the metabolic pathway. This data will be valuable for clinical assessments in vitamin D health and disease.

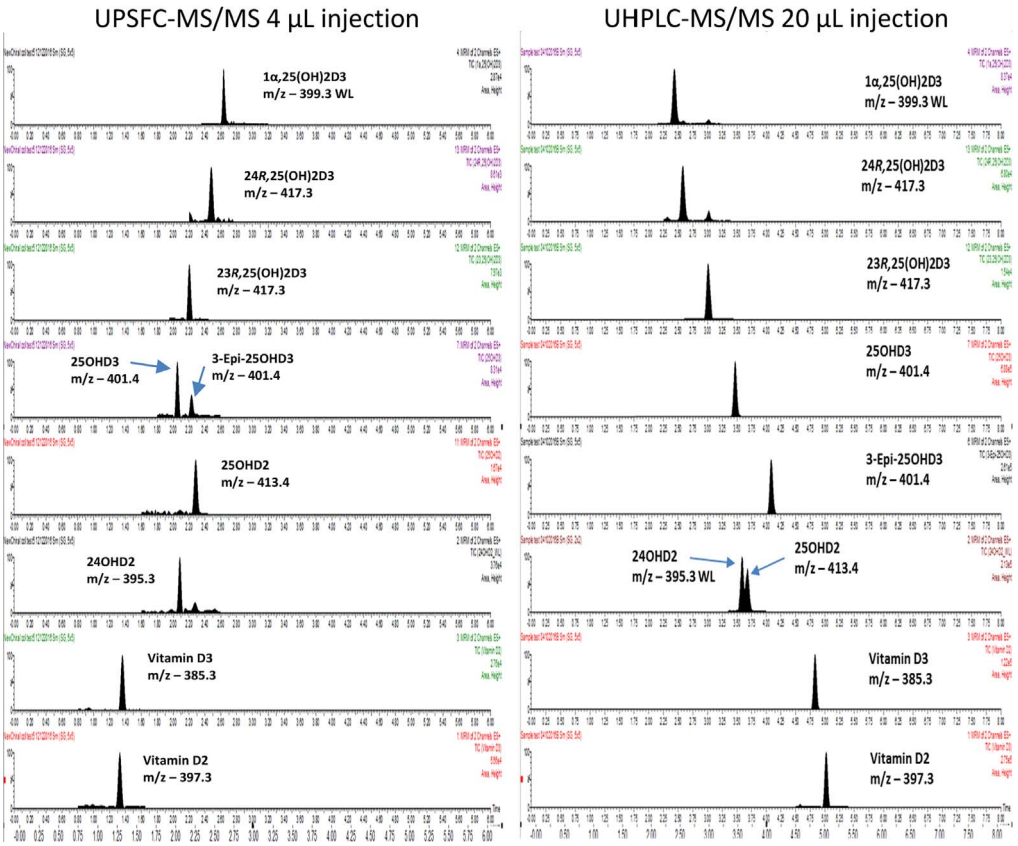

Fig. 1. Chromatogram of vitamin D analytes on UPSFC and UPLC, separated using a Lux Cellulose-2 chiral column.

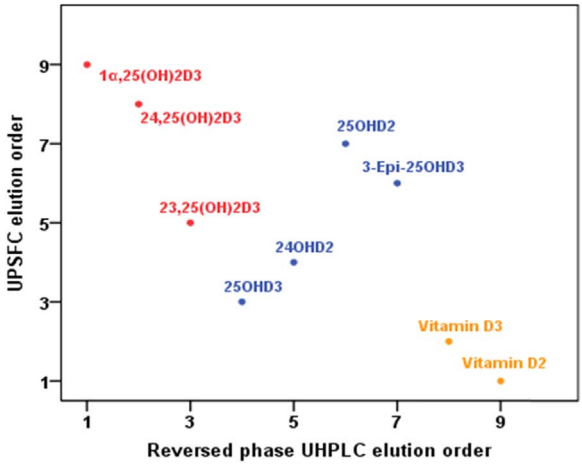

Fig. 2. Comparison in elution order of vitamin D analytes on UPSFC and UPLC.

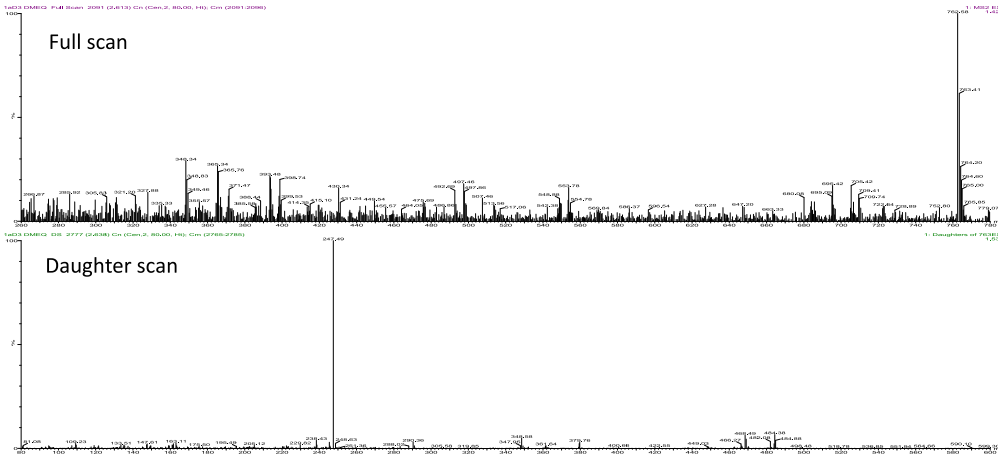

Fig. 3. Mass spectrum of full scan and daughter scans of 1α,25(OH)$_2$D3 following derivatization with DMEQ-TAD.

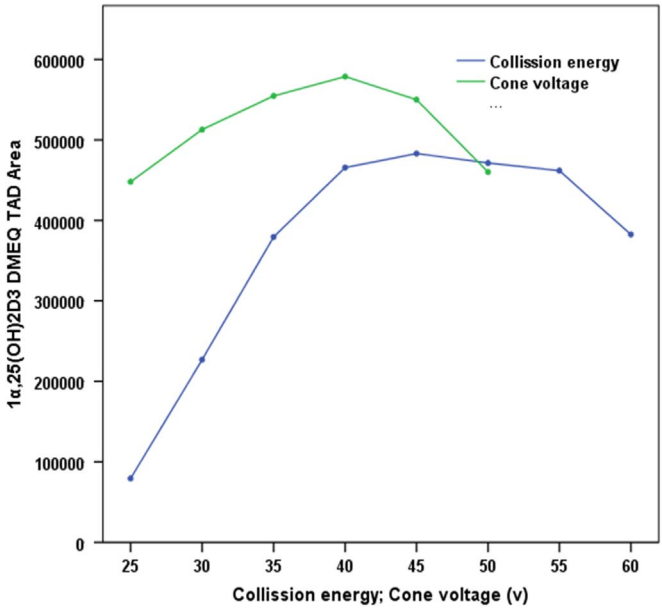

Fig. 4. Signal intensity (area) of 1α,25(OH)$_2$D3-DMEQ TAD at a range of collision energy (25–60) and cone voltage (25–50) values.

1. Data

1.1. Elution order and chromatography comparison between UPSFC-MS/MS and UPLC-MS/MS

The chromatograms in Section 1.1 compare the chromatography of UPSFC-MS/MS and UPLC-MS/MS for measuring multiple vitamin D metabolites; vitamin D3, vitamin D2, 25-hydroxyvitamin D3 (25OHD3), 25OHD2, 24OHD2, 3-epi-25OHD3, 1α,25-dihydroxyvitamin D3 (1α,25(OH)$_2$D3), 23,25

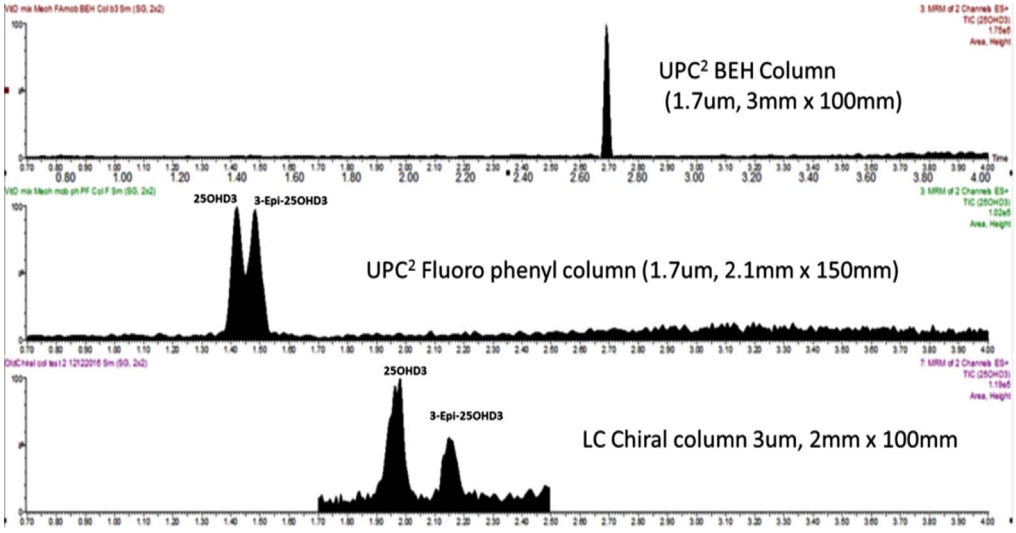

Fig. 5. Column screening on UPSFC to separate 25OHD3 and 3-epi-25OHD3. Both analytes have a mass to charge (m/z) of 401.6.

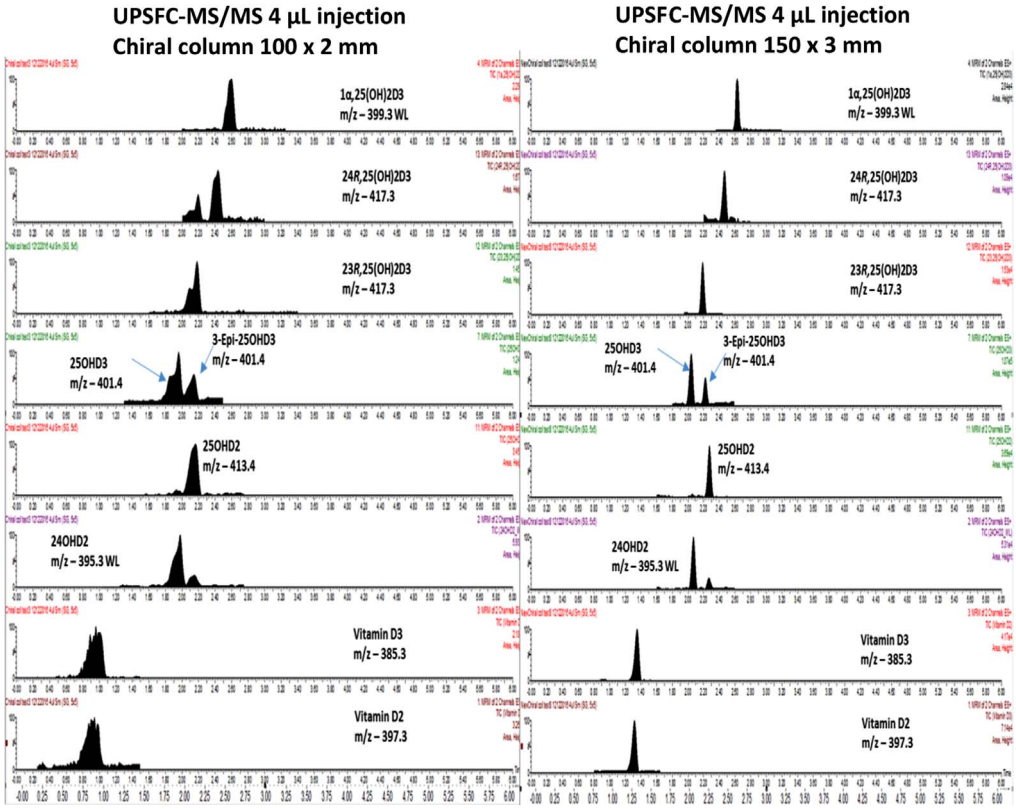

Fig. 6. Chromatogram of vitamin D analytes on Lux Cellulose-2 chiral columns with different dimensions: 100 × 2 mm and 150 × 3 mm.

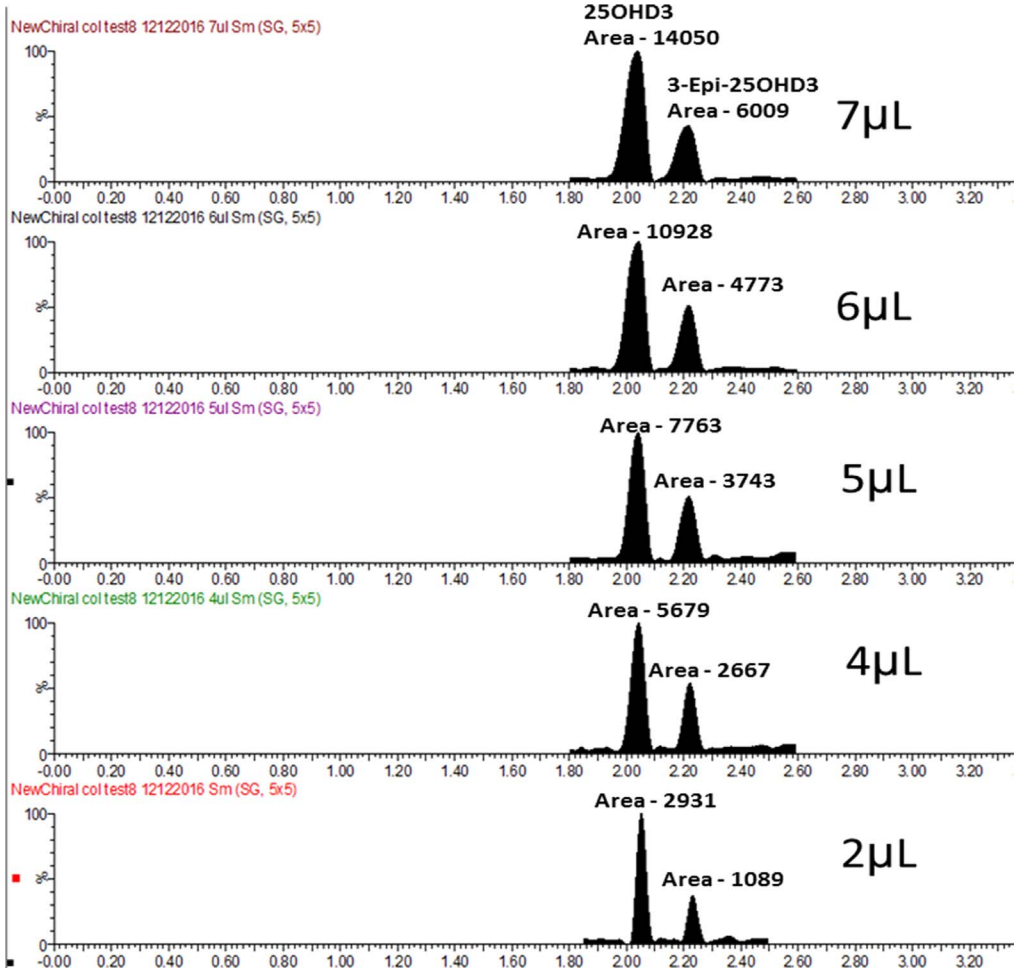

Fig. 7. Separation and signal intensity (area) of 25OHD3 and 3-epi-25OHD3 with increasing sample injection volume (2–7 µL) using a 150 × 3 mm Lux Cellulose-2 chiral column.

$(OH)_2$D3 and 24,25$(OH)_2$D3 (Fig. 1). The elution order of vitamin D analytes is compared between UPSFC and UPLC in Fig. 2.

1.2. Optimization of mass spectrometry conditions

The data in Section 2 was obtained during the method development and optimization of the UPSFC-MS/MS method. Section 2.1 presents data from the optimization of multiple reaction mode (MRM) parameters using 1α,25$(OH)_2$D3 derivatized with DMEQ-TAD as an example. The mass spectra from full scan and daughter scan of m/z 762.6 > 247.5 are shown in Fig. 3. The signal intensity of 1α,25$(OH)_2$D3 DMEQ-TAD is compared with a range of cone voltage and collision energies in Fig. 4 to determine the optimal values.

1.3. Optimization of UPSFC column conditions

The chromatograms and figures in section 2.2 relate to the optimization of UPSFC column screening and selection for optimized separation of vitamin D metabolites. The chromatograms

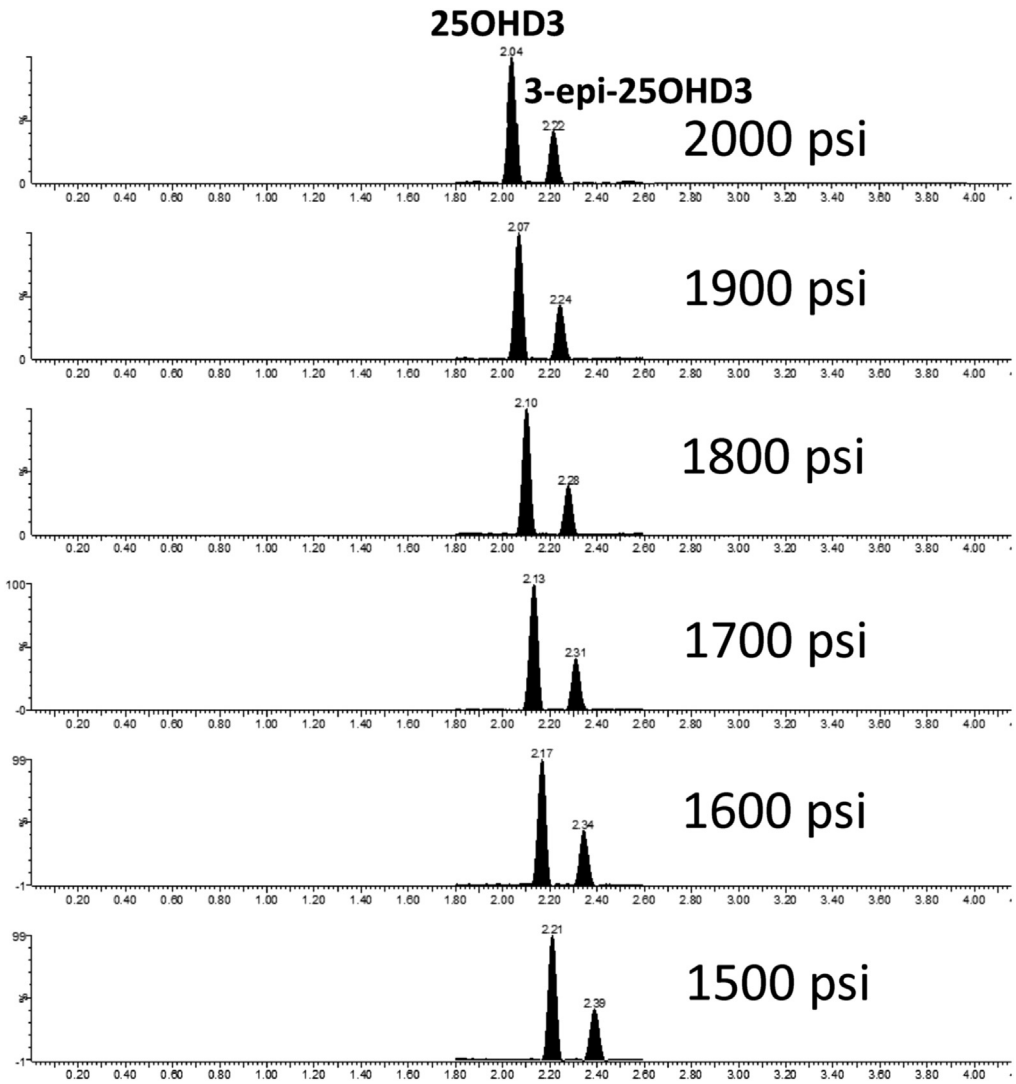

Fig. 8. Elution of 25OHD3 and 3-epi-25OHD3 with increasing ABPR (1500–2000 psi) using a 150 × 3 mm Lux Cellulose-2 chiral column.

displayed in Figs. 5 and 6 compare different column chemistries and column size to optimize vitamin D separation by UPSFC.

1.4. Optimization of UPSFC method parameters

The chromatographs and figures in section 2.3 are outputs from the optimization of UPSFC parameters for vitamin D analysis. The chromatograms compare the separation and signal intensity of 25OHD3 and 3-epi-25OHD3 with increasing injection volume and atmospheric back pressure regulator (ABPR) in Figs. 7 and 8, respectively. The signal intensity of vitamin D analytes is compared for the optimization of the inlet flow rate, ABPR, column temperature and solvent for sample reconstitution in Figs. 9–11 respectively (Fig. 12).

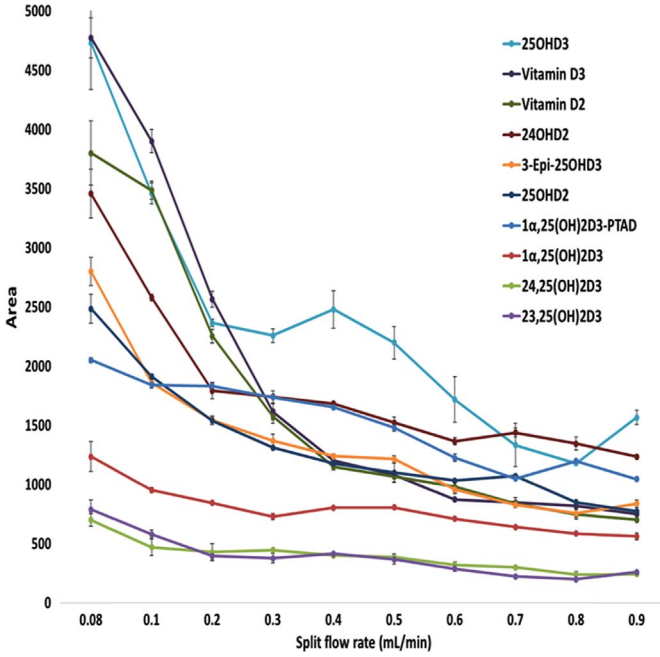

Fig. 9. Analyte areas of vitamin D metabolites with increasing split flow rate (0.08–0.9 mL/min) containing methanol 0.1% formic acid.

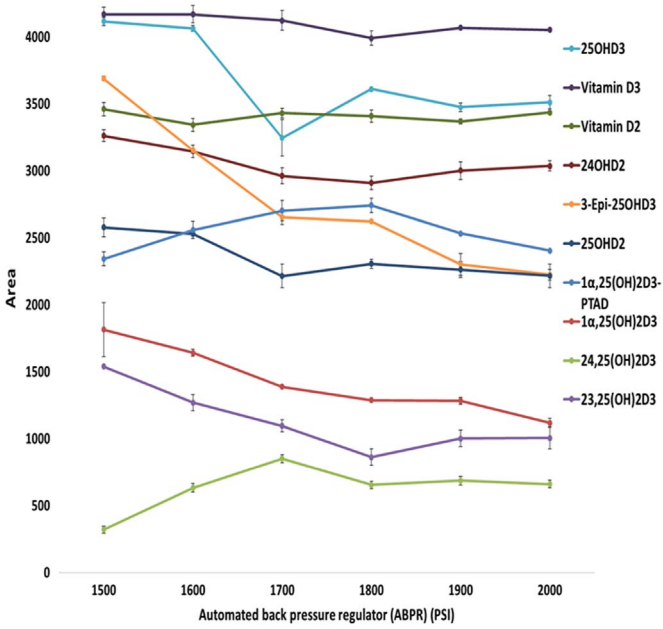

Fig. 10. Analyte areas of vitamin D metabolites with increasing ABPR (1500-200 psi).

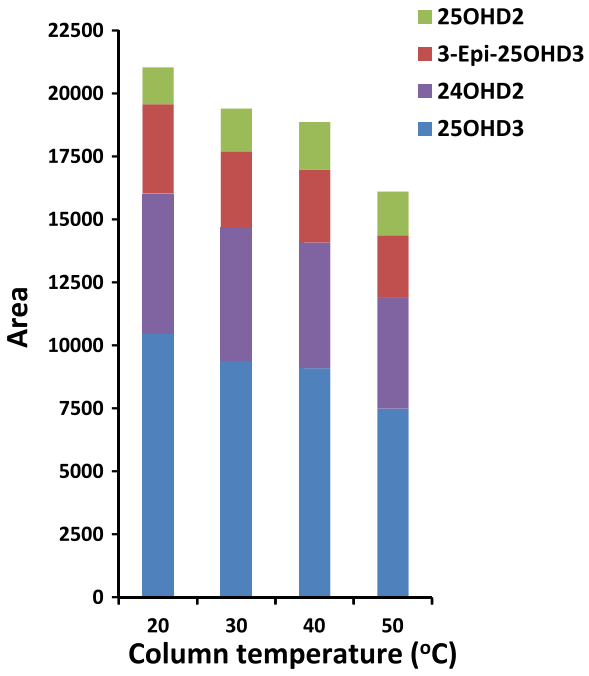

Fig. 11. Analyte areas of vitamin D metabolites with increasing column temperature (20–50 °C).

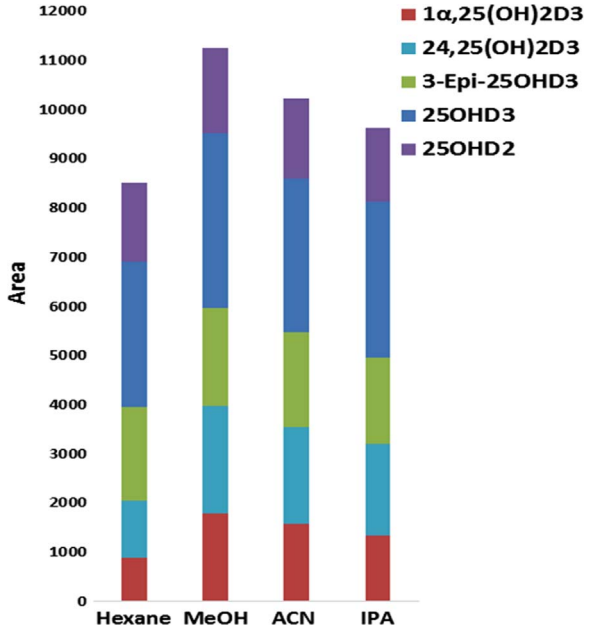

Fig. 12. Analyte areas with different solvents used for sample reconstitution.

2. Experimental design, materials, and methods

The sample preparation, UPSFC-MS/MS and UPLC-MS/MS methodologies for the data presented here have been previously described and cited [1]. The DMEQ-TAD sample preparation method is described previously [2].

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Transparency document. Supplementary material

Transparency document associated with this article can be found in the online version at https://doi.org/10.1016/j.dib.2018.08.027.

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[1] C. Jenkinson, A. Taylor, K.H. Storbeck, M. Hewison, Analysis of multiple vitamin D metabolites by ultra performance supercritical fluid chromatography-tandem mass spectrometry (UPSFC-MS/MS), J. Chromatogr.. B 1087–1088 (2018) 43–48.
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