
Poster

[P27-8] P27-8: Assay and monitoring

Chair: Yoshihiko Hirotsu, Japan

Wed. Sep 27, 2017 12:30 PM - 1:30 PM Annex Hall (1F)

(Wed. Sep 27, 2017 12:30 PM - 1:30 PM Annex Hall)

[P27-8-6] Quantitative analysis of vitamin D2 and D3 using SFC-MS/MS

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Keywords: SFC-MS, Vitamin D2/D3

Background

Vitamin D measurement has become an important component in clinical assays largely because deficiency is associated with a number of disorders, such as rickets, osteomalacia and osteoporosis. LC-MS/MS has become essential tool for monitoring the concentration of Vitamin D2/D3 in biological samples due to its high level of sensitivity and specificity; however, another technic can improve the sensitivity.

Method

In this study, a new supercritical fluid chromatography-mass spectrometry (SFC-MS/MS) method has been developed. The last years have seen considerable activity in the development of SFC, now emerging as a real competitor or complement to traditional high-performance liquid chromatography (HPLC). Addition of a polar co-solvent, such as methanol, increases the solvent strength for hydrophilic compounds, offering versatility in its range of applications. In addition to the decreased usage of toxic organic solvents, a supercritical fluid is readily evaporated once the pressure is released, resulting in an increased sensitivity for many compounds, when using MS detection.

Results

The LCMS-8050 triple quadrupole mass spectrometer was coupled to the Nexera UC system. Sample preparation involved taking 50 L of sample, adding to it 150 L of precipitant solution (containing internal standard). Following incubation and centrifugation, 5 L of supernatant was injected for analysis. The UC-MS instrument was equipped with an electrospray source (ESI). The rapid elution of vitamin D3 and D2 by a gradient (0 to 20% of MeOH) produced excellent peak shape and accuracy with elution in less than five minutes. Sensitivity is increased by more than 5 compared to the classical LC-MS/MS, and the limit of detection is less than 1 pg/ μ L. The calibration curves showed good linearity ($R^2 > 0.999$).

Conclusions

Due to the intrinsic, universal sensitivity and specificity of MS, SFC-MS/MS offers an attractive alternative to reverse phase liquid chromatography (RPLC)-MS. It leads to lower cost per analysis thanks to reduction of solvent consumption together with improvement in sensitivity.