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High-temperature high-resolution molecular absorption spectrometry – a supplementary technique to ICP-MS for the determination of halogens

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Inductively coupled plasma mass spectrometry (ICP-MS) is without doubt one of the most sensitive techniques for the determination of trace elements in a variety of matrices. However, there are also some limitations, and the most serious ones are with the determination of the halogens. Fluorine, which has an ionization energy of 17.42 eV, can essentially not be determined by ICP-MS, as the ionization energy of the plasma gas argon is only 15.76 eV. Chlorine and bromine have lower ionization energies of 12.97 eV and 11.81 eV, respectively, and can be determined by ICP-MS; however, severe matrix and memory effects can often be observed when conventional sample introduction with a nebulizer/spray chamber is used. One way out of this problem is using electrothermal vaporization (ETV) for sample introduction.High-temperature high-resolution molecular absorption spectrometry (HR-CS MAS) is ideally suited for the determination of non-metals, and can therefore be used as a supplementary technique to ICP-MS. The sample – in many cases a solid sample – is directly introduced into a small graphite tube furnace, a 'molecule-forming reagent' is added, and the furnace is heated to a previously optimized temperature – typically between 1500 °C and 2500 °C. At this temperature, diatomic molecules are formed in the gas phase, such as CaF, SrCl or CaBr, which exhibit a characteristic absorption spectrum, which can be used for the sensitive determination of these halogens. Plant materials, coal, copper concentrate, cosmetics and fish oil were investigated with this technique and the results compared with those of ICP-MS where possible.

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Validation of a rapid liquid chromatography tandem mass spectrometry method for serum 25OHD and evaluation of the necessity to separate 3-epi $25OHD_3$

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The objective of this study is to develop a rapid liquid chromatography tandem mass spectrometry (LC-MS/MS) method with ability to separate 3-epi $25OHD_3$ (EPI-LC-MS/MS) from $25OHD_3$, and evaluate the effects of 3-epi $25OHD_3$ on routine LC-MS/MS that cannot separate 3-epi $25OHD_3$ (NEPI-LC-MS/MS). Performance of the newly built EPI-LC-MS/MS was validated, and 982 samples were analyzed and compared by the two methods. Both methods showed a linearity coefficient correlation exceeding 0.999 in the 6.25–500 nmol/L range for $25OHD_2$ and $25OHD_3$. Moreover, they showed a between run coefficient variation (CV) and total CV of < 5% for 25OHD2 and $25OHD_3$. The results of the accuracy test showed that the bias was below 6.19% in the absence of 3-epi $25OHD_3$. Comparison of the 25OHD results obtained by the two methods for 982 patients (age 1-100 years) revealed excellent clinical agreement (Cohen's kappa = 0.875) and correlation (R2 = 0.973). Among the 982 patients, only 73 patients had 3-epi $25OHD_3$ (> 6.25 nmol/L); out of these 73 patients, the 3-epi $25OHD_3$ level in 58 patients was between 6.25 and 12.5 nmol/L. In patients with < 375 nmol/L 25OHD ($25OHD_2$ + $25OHD_3$), only 8 had 3-epi $25OHD_3$ levels exceeding 12.5 nmol/L (range: 13.3 - 27.5 nmol/L). Among samples containing 3-epi $25OHD_3$, only three were separated into different 25OHD-deficiency groups using the above methods. In conclusion, a rapid and precise EPI-LC-MS/MS method for measuring 25OHD with efficient separation of 3-epi $25OHD_3$ was developed. Our results showed that 3-epi $25OHD_3$ had little effect on the routinely used NEPI-LC-MS/MS.

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