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Oral: Stable isotope ratio analysis to assess pharmaceuticals, cosmetics and dietary supplements authenticity

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The stable isotope ratio analysis of the mayor bio-elements (hydrogen, carbon, nitrogen, oxygen and sulphur) makes it possible to authenticate pharmaceuticals, cosmetics and dietary supplements. This technique, applied to bulk samples and/or to specific compounds, can be used to detect the origin of an ingredient (synthetic or natural), the substitution of one ingredient with another, as well as the geographical and/or botanical origin of the products. The $\delta^{13}C$ and $\delta^{2}H$ values of vanillin can determine whether this product is natural (deriving from the expensive CAM plant *Vanilla*), biotechnologically produced or synthetic [1]. Moreover, the $\delta^{13}C$ values of specific components of *Rosa damascene mill.*, one of the most expensive essential oils in the global market, can indicate the fraudulent addition of cheaper oil from C4 plants (e.g., *Cymbopogon martinii, palmarosa*) [2]. Finally, the $\delta^{13}C$ analysis is a suitable tool to discriminate between Monacolin K (contained in red yeast rice-based dietary supplements) and the marketed statin [3] and between natural L-theanine (extracted from *Camellia Sinensis*) and the biosynthetically produced one [4].

These examples show that the isotopic fingerprint represents an effective tool for the authenticity assessment of economically relevant pharmaceuticals, cosmetics and dietary supplements.

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[2] F. Pellati, G. Orlandini, K.A. van Leeuwen, G. Anesin, D. Bertelli, M. Paolini, S. Benvenuti, F. Camin (2013). Gas chromatography combined with mass spectrometry, flame ionization detection and elemental analyzer/isotope ratio mass spectrometry for characterizing and detecting the authenticity of commercial essential oils of Rosa damascena Mill. Rapid Communications in Mass Spectrometry 27(5), 591–602. DOI: 10.1002/rcm.6489.

[3] M. Perini, G. Carbone, F. Camin (2017). Stable isotope ratio analysis for authentication of red yeast rice. Talanta 174, 228–233. DOI: 10.1016/j.talanta.2017.05.057.

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Oral: First Use of Triply Labelled Water analysis for energy expenditure measurements in mice

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The Doubly Labelled Water (DLW) method is widely used to determine energy expenditure of (free roaming) animals, and of humans. In this work, we demonstrate the addition of the third stable isotope, ^{17}O, to turn it into Triply Labelled Water (TLW), exploiting the modern three isotopes measurement capabilities of optical spectrometry. We performed TLW measurements for the analysis of the CO_2 production (r_{CO2}) of mice on different diets. Triply highly enriched water (with abundances of 30%, 55% and 8% for ^{2}H, ^{18}O and ^{17}O, respectively) was injected into mice, and the isotope enrichments of the distilled blood samples taken after 2, 24 and 48 hours, respectively, were measured by an Off-Axis Integrated Cavity Output Spectroscopy instrument (LGR LWIA 912-0050). Typical δ -values for the 2-hours blood samples of the mice were 13000‰, 1800‰ and 1600‰ for $\delta^{2}H$, $\delta^{18}O$ and $\delta^{17}O$, respectively. Analysis of the measurements was done using a bespoke data analysis program (written in R), which includes a sophisticated memory correction algorithm [1]. For these enriched samples such an algorithm is indispensable. For calibration of the measurements, we extended the range of available DLW reference waters [2] by preparing ^{17}O enriched reference waters on a gravimetrical basis.
We found that the values of the r_{CO2} (which are proportional to the energy expenditure), calculated based on ^{18}O-^{2}H, and on ^{17}O-^{2}H, agreed very well, increasing the reliability and redundancy of the measurements and lowering the uncertainty in the calculated r_{CO2} to ±3%. However, like in a previous study using DLW [3], the TLW method overestimated the r_{CO2} compared to the indirect calorimetry measurements that we also performed. Thanks to the addition of ^{17}O, we could now unambiguously identify ^{2}H isotope effects as the culprit. We hypothesize an extra loss or exchange mechanism with a high fractionation for ^{2}H to explain this difference.

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