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Optimizing GC Injections when Analyzing δ²H of Vanillin for Traceability Studies

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Column overloading is a problem when analyzing δ²H, due to the low natural abundance of deuterium and poor ionization efficiency of H₂. This problem can be overcome by using split injections instead of splitless. In this study we have compared the influence upon the measured isotopic ratios when using the two injection methods.

Introduction
In traceability studies of vanilla, the main aroma constituent 4-hydroxy-3-methoxybenzaldehyde (vanillin) was analyzed for its δ³H values by Gas Chromatography Pyrolysis Isotope Ratio Mass Spectrometry (GC-P-IRMS). To obtain good precision, it is normally required to have at least 30ng of hydrogen on column depending on the instrument. However, the amount of vanillin in extracts of vanilla pods will overload a DB-5ms column with a film thickness of 0.25µm when injecting 1µL using splitless injection – as seen in figure 1. Chromatography can be improved by using split injections with a split ratio of 1:20. δ¹H should in theory not be affected by sample amount and therefore be the same independently of injection method.

Results
In this study we compared values of δ³H for vanillin using split and splitless injection. Splitless injections maintains a constant level of δ³H (see figure 2) with standard deviation around 3‰ corresponding to the expected precision of the instrument. At low concentrations of vanillin the δ³H values decreases when using split at 1:20.

Accordingly, using split injections at low concentration will lead to discrimination against δ³H compared to splitless injections. It is therefore recommended to use splitless injections for analysis of δ³H when analyzing extracts of vanilla pods for traceability studies.

When injecting 40ng vanillin on column for both methods, a deviation above 10‰ was found (data not shown). This indicates differences in the obtained values is not solely due to amount-dependent isotopic fractionation, but partly also due to fractionation during split injection.

Materials and methods
GC-IRMS: Trace GC Ultra fitted with a DB-5 capillary column (Agilent Technologies, Böblingen, Germany) (30m x 0.250mm i.d., δj 0.25 µm) coupled to Delta V Advantage Isotope Ratio Mass Spectrometer (Thermo Scientific, Bremen, Germany). High Temperature Conversion Reactor consisting of a ceramic tube with no catalyst was operated at 1420 C. Helium was used as carrier gas at 1.2mL/min. Injector temp. 230 C.