
13C Position-specific isotopic analysis of vanillin: from industrial production to shopping carts.

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Résumé

Vanillin produced from vanilla pods represents less than 1% of the vanillin global demand, but this part is likely to grow due to increasing consumer taste for natural product. Therefore, because of the recent climate events impacting vanilla crops and the high price difference between vanillin (main molecule of the vanilla aroma) ex-vanilla pods or from other sources, this market is sensitive and subject to economically motivated adulterations. Isotopic analysis is a premium tool for vanillin authenticity assessment, particularly through position-specific methods. A new 13C-SNIF-NMR method has been proposed (Guyader et al., 2019) and routinely applied. The method designed as ‘single pulse’ approach was developed with appropriate reproducibility (Caytan et al., 2007). Despite the great performance of this method to discriminate vanillin sources, it suffers from long acquisition time (8h / sample) and requires high quantity of pure vanillin, making it unrealistic for commercially available vanilla-flavoured products whose vanillin concentration is often lower than 100 mg/kg. Developments on NMR pulse sequence offers solution to tackle these issues.

INEPT (Insensitive Nuclei Enhanced by Polarization Transfer) refocused adiabatic pulse sequence has been developed (Merchak et al., 2015) and implemented for vanillin authentication. Sample preparation and acquisition parameters were optimized. A significant number of samples was analyzed using both single pulse (Guyader et al., 2019) and INEPT sequence in order to predict full isotopic 13C distribution from INEPT data through modelisation, enabling the prediction of the 3 unobserved quaternary carbons isotopic composition.

Required quantity or acquisition time (depending on the application) was significantly reduced, without change in terms of discriminative power for authentication purposes. This method was also applied on commercially-collected vanilla sugars as a proof of feasibility. Ongoing work on vanillin extraction aims to extend this approach to complex transformed products such as ice creams, yoghurts and biscuits amongst other.

References

Caytan et al. (2007) DOI: 10.1021/ac070826k.

Guyader et al. (2019) DOI: 10.1002/ffj.3486.

Merchak et al. (2015) DOI: 10.1039/C5AY01250C.

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