



Adulteration of Used Cooking Oils

An NMR Method for the Detection of Fresh Palm Oil in Used Cooking Oils

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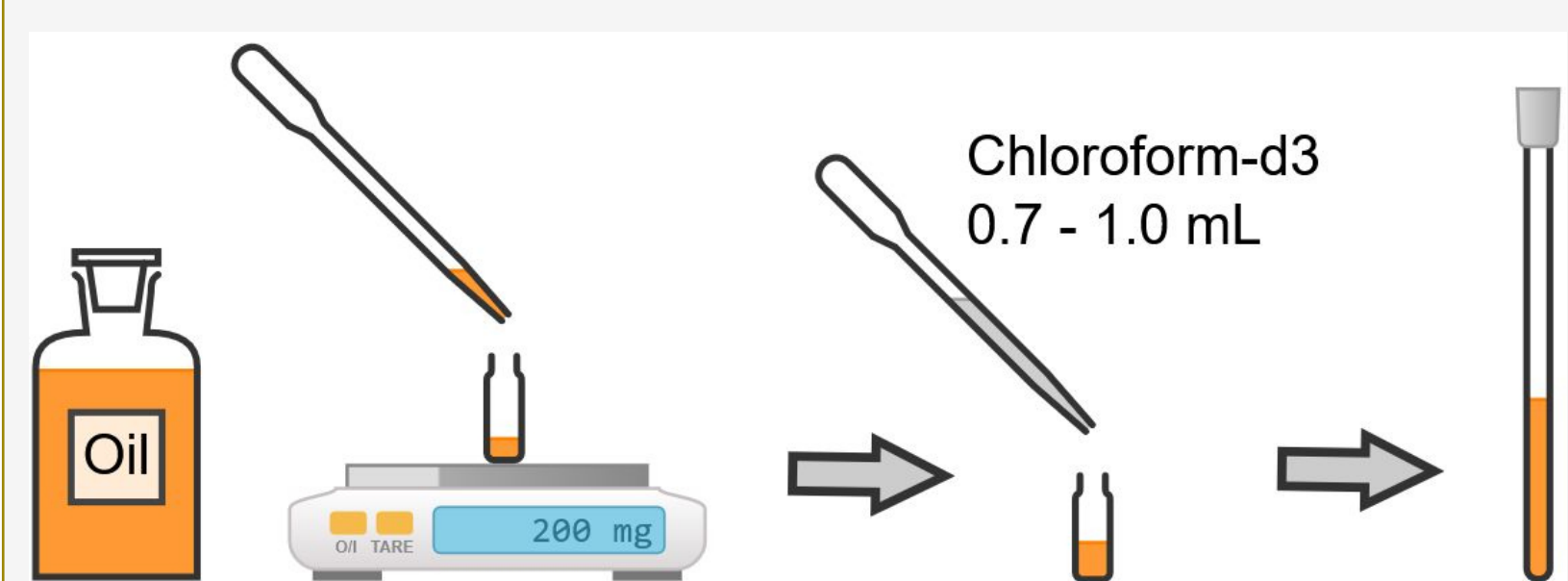
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„Why NMR, why this topic?“

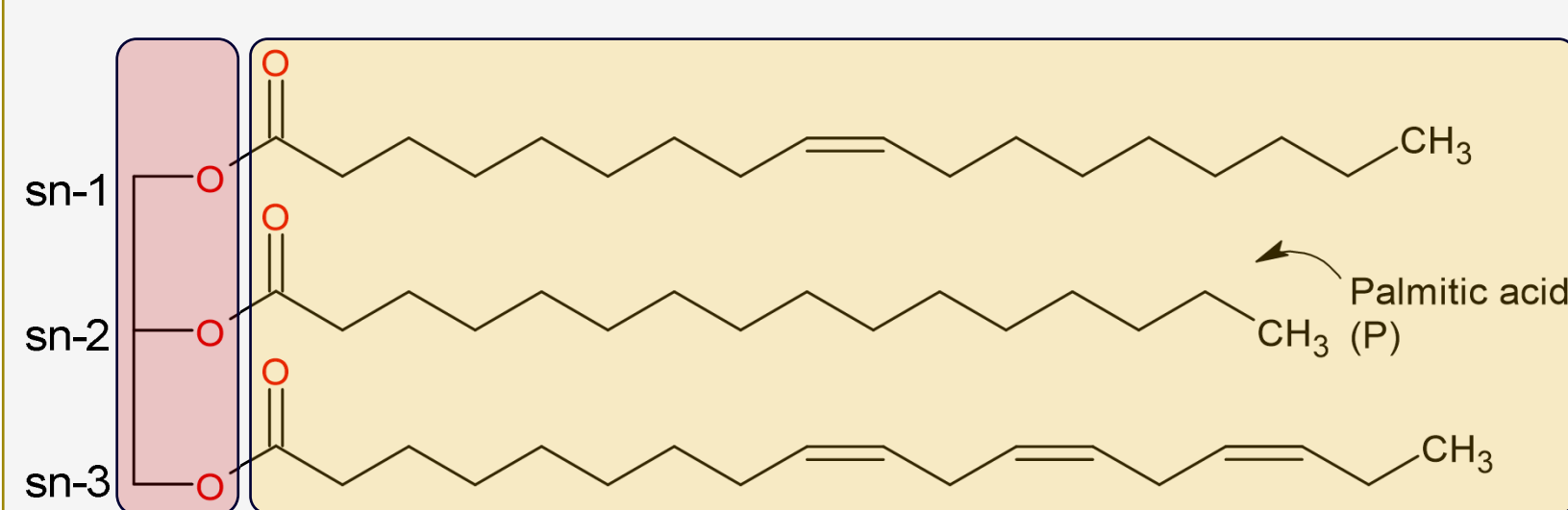
Used cooking oils (UCO) subjected for biodiesel production, stretched with fresh palm oil (PO) is costing millions in EU subsidies. Conventional techniques for lipid analysis (HPLC, GC) can't easily determine the addition of fresh PO after the usage.

¹³C-NMR spectroscopy can distinguish between fatty acid distributions of UCOs with and without PO. Additionally, ¹H-NMR spectroscopy can identify fresh, unrefined PO by specific heat-sensitive substances in concentrations down to approx. 2 % (w/w). The identification of fresh, refined PO proves difficult but is still worked on.

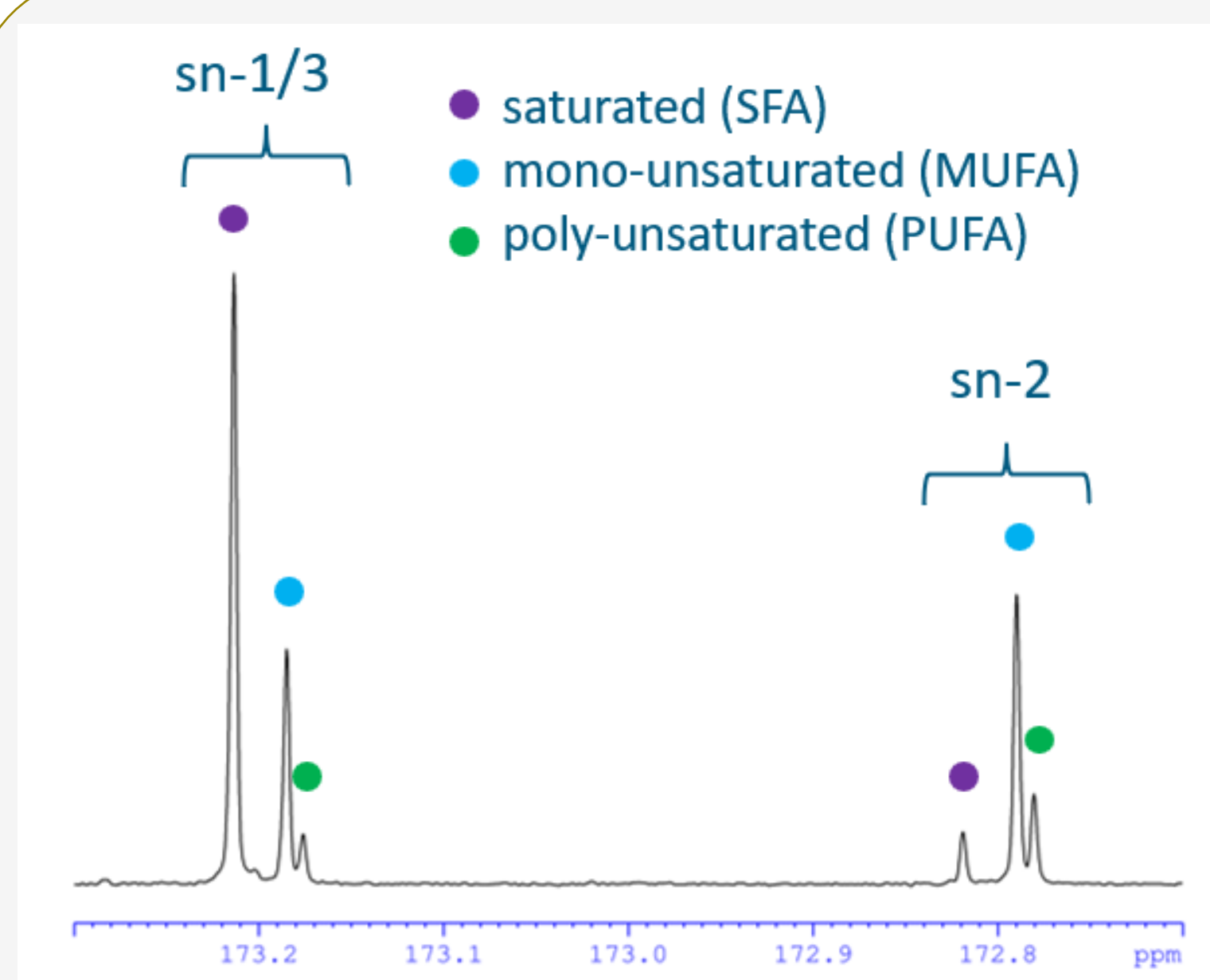
Fatty acids (¹³C-NMR)



Sample preparation for fatty acid analysis

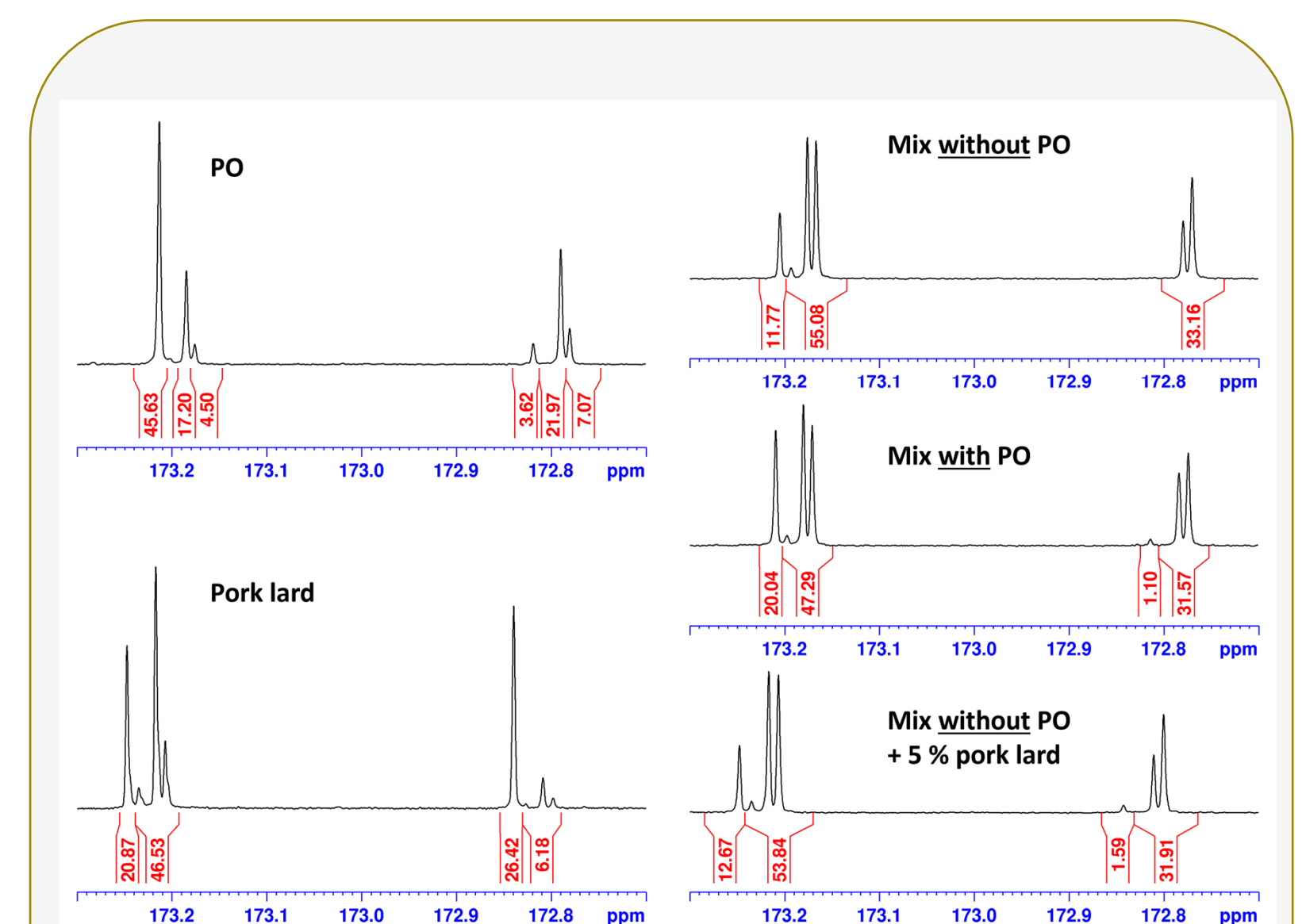


Exemplary structure of triglycerides



¹³C-NMR spectrum of PO, Carbonyl region

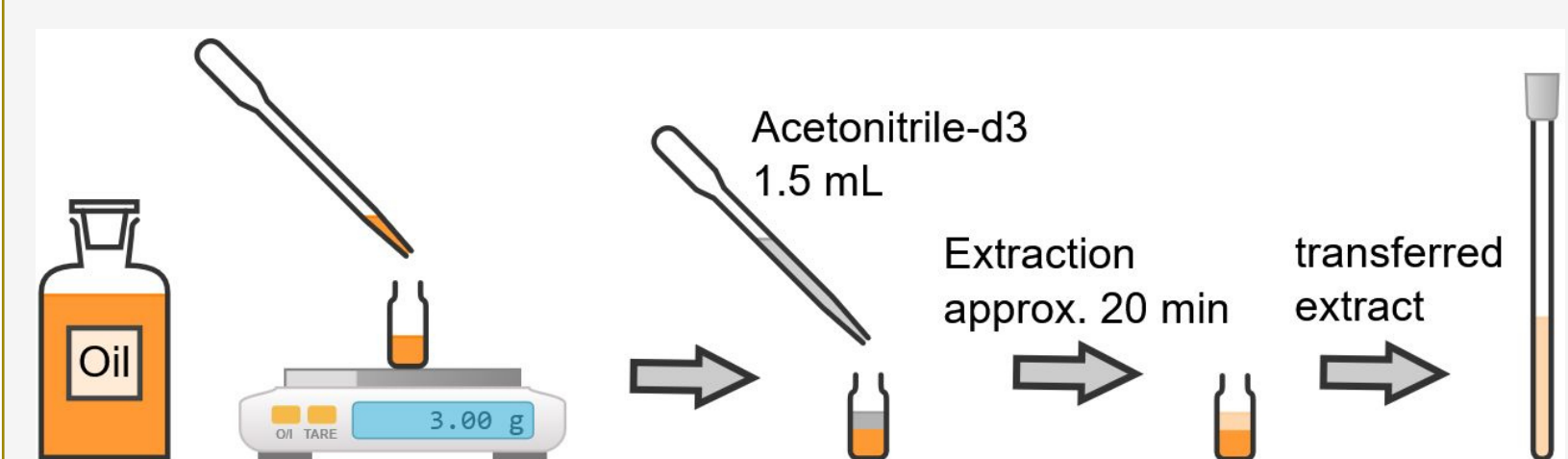
PO is highly saturated (~45 % palmitic acid (P))
→ one of few vegetable oils with saturated fatty acids in sn-2



¹³C-NMR spectra of PO and different oil mixtures, Carbonyl region

Ratios of saturated fatty acids in sn-1/3 and sn-2 are specific for PO, even in mixtures

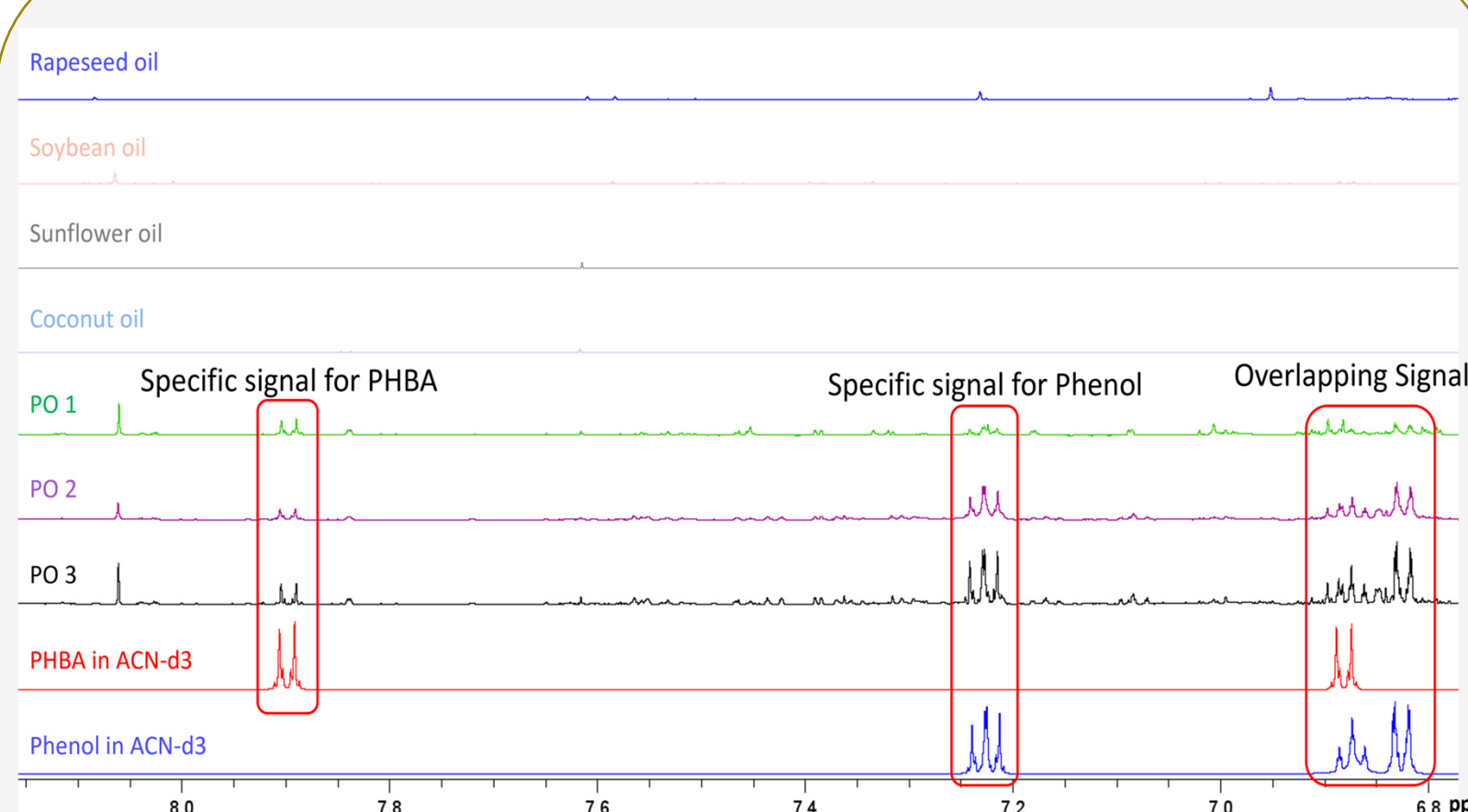
Non-lipid components (¹H-NMR)



Sample preparation for analysis of non-lipid components

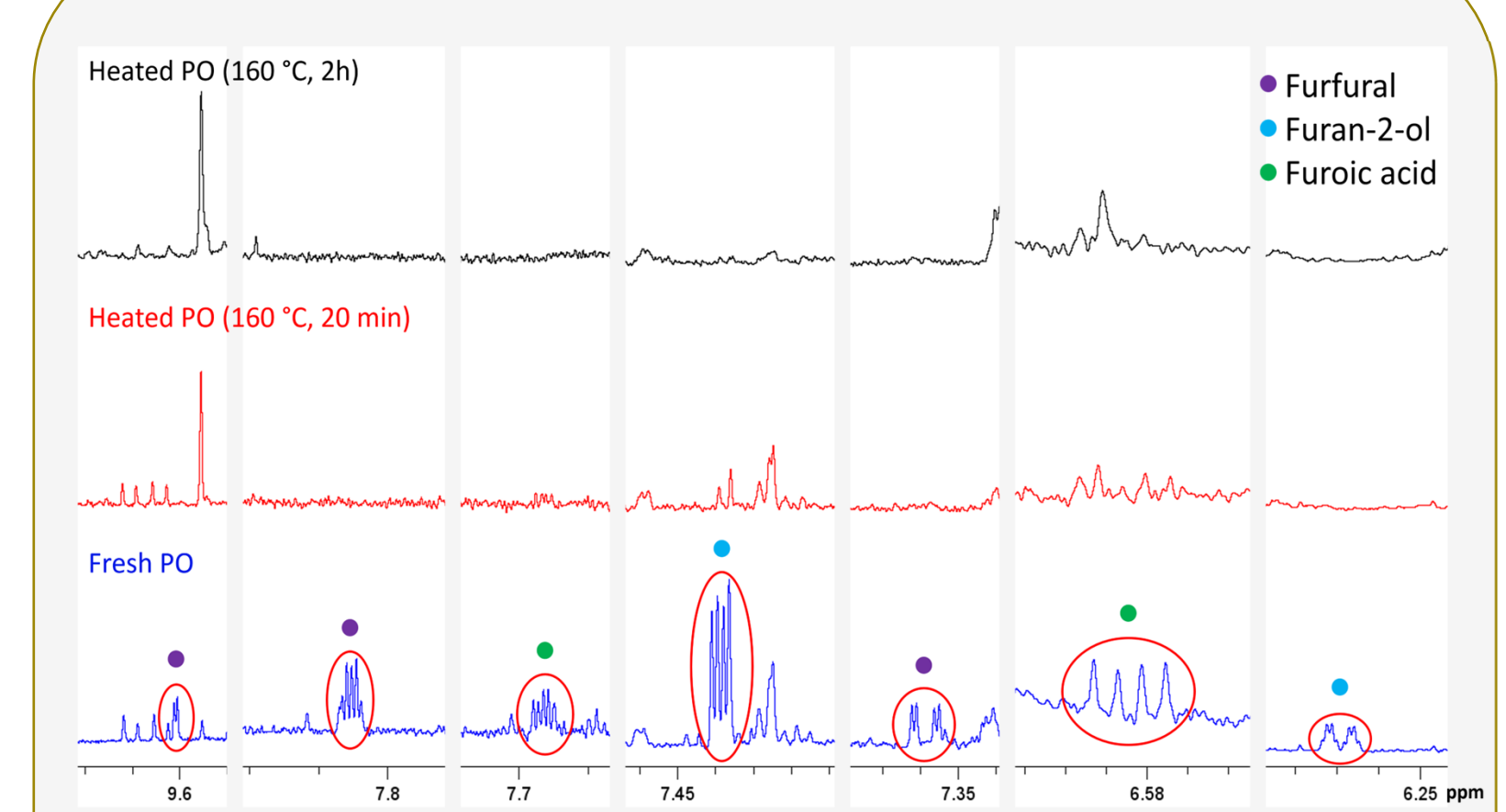
Non-lipid components include sterols, carotenoids, flavonoids, tocopherols, etc.

Two groups identified during heating of oils (simulation of frying conditions):
heat-resistant and heat-sensitive



¹H-NMR spectra of ACN extracts: Phenol and p-Hydroxybenzoic acid (PHBA) are specific for PO

Phenol and PHBA are specific for PO and heat-resistant
→ identification of PO in mixtures



¹H-NMR spectra of ACN extracts of fresh and heated PO: signals of Furfural, Furan-2-ol, and furoic acid

Furfural, furan-2-ol, and furoic acid are specific for PO and heat-sensitive
→ distinguishment of heated and fresh palm oil (adulteration)

Conclusion and Limitations

Identification of (unrefined) palm oil in oil mixtures and used cooking oils is possible from the specific fatty acid profile and arrangement by high-resolution ¹³C-NMR, as well as heat-resistant marker substances (e.g. Phenol, PHBA). The unknown oil fractions in mixtures can make the evaluation of the spectra complex and time-consuming.

The addition of fresh palm oil to used cooking oils after their usage can be identified based on the palm oil-specific, heat-sensitive marker substances (e.g. furfural, furan-2-ol, furoic acid, and others).

Refined palm oil does not contain any of these markers and fatty acid profile is the same as unrefined palm oil → adulteration can't be detected yet.