

Beeswax Characterization / Adulteration Detection

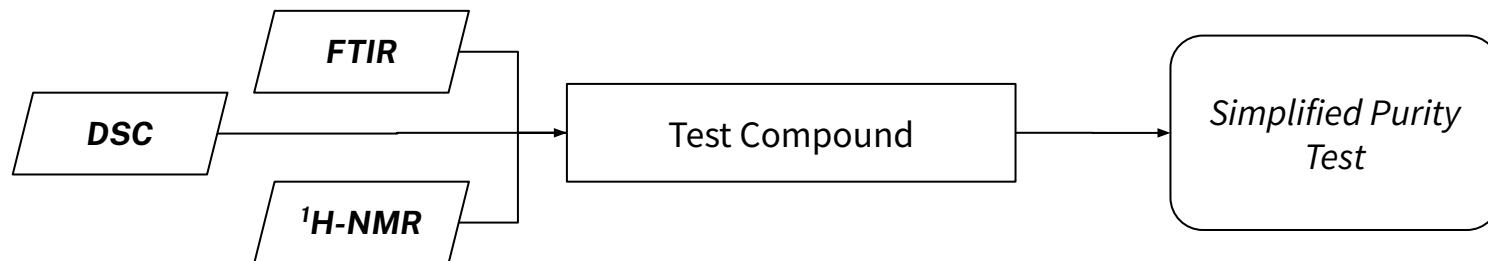
Thermochemical and Spectroscopic Analysis

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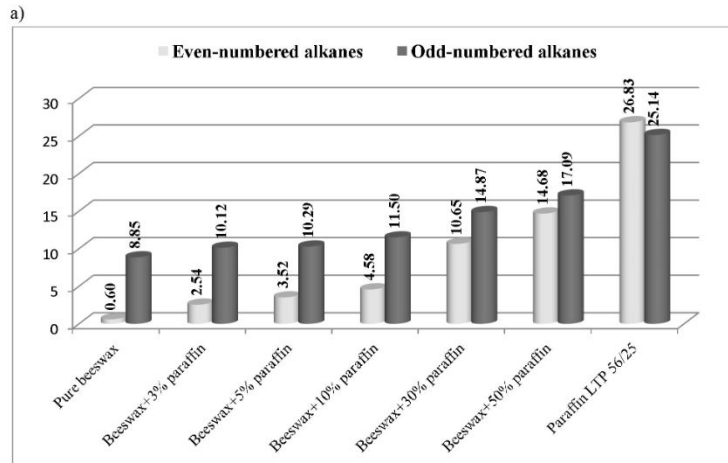
Abstract

- During the production of beeswax products, specific synthetic chemicals may be introduced to reduce manufacturing costs. These “adulterants,” often derived from petroleum and related byproducts, reduce the quality of the product and may represent health risks when used in the food and cosmetic industry.
- Current methods for in-situ purity analysis often rely on complex analytical instruments that are prohibitively expensive and require specialty knowledge and experience.
- Previously, an attempt at analytical model creation was made using Differential Scanning Calorimetry (DSC). To extend past the evident limitations of DSC, a combination of Nuclear Magnetic Resonance (NMR) and Fourier-Transform Infrared spectroscopy (FT-IR) data is used to create a basic platform for a new variant of purity test.



Baseline / Foundational Data

- Multiple previous analyses have been done.
 - Excellent paper that serves as the baseline for this study: “Efficiency of GC-MS Method in Detection of Beeswax Adulterated with Paraffin”
- GC/MS (Gas Chromatography - Mass Spectroscopy) is typically used in analysis as it is well-suited to this environment. However, it is expensive and extremely time-consuming for wax.



Waś, E., Szczęśna, T., & Rybak-Chmielewska, H. (2016). Efficiency of GC-MS method in detection of beeswax adulterated with paraffin. *Journal of Apicultural Science*, 60(1), 145–162. <https://doi.org/10.1515/jas-2016-0012>

Preparation

FTIR

- Multiple wax samples are created.
 - Each sample placed in containment tube, and heated with agitated water bath set at 110 C until homogenized, careful to avoid breakdown of beeswax.
- For FTIR, no other special prep requirements exist.

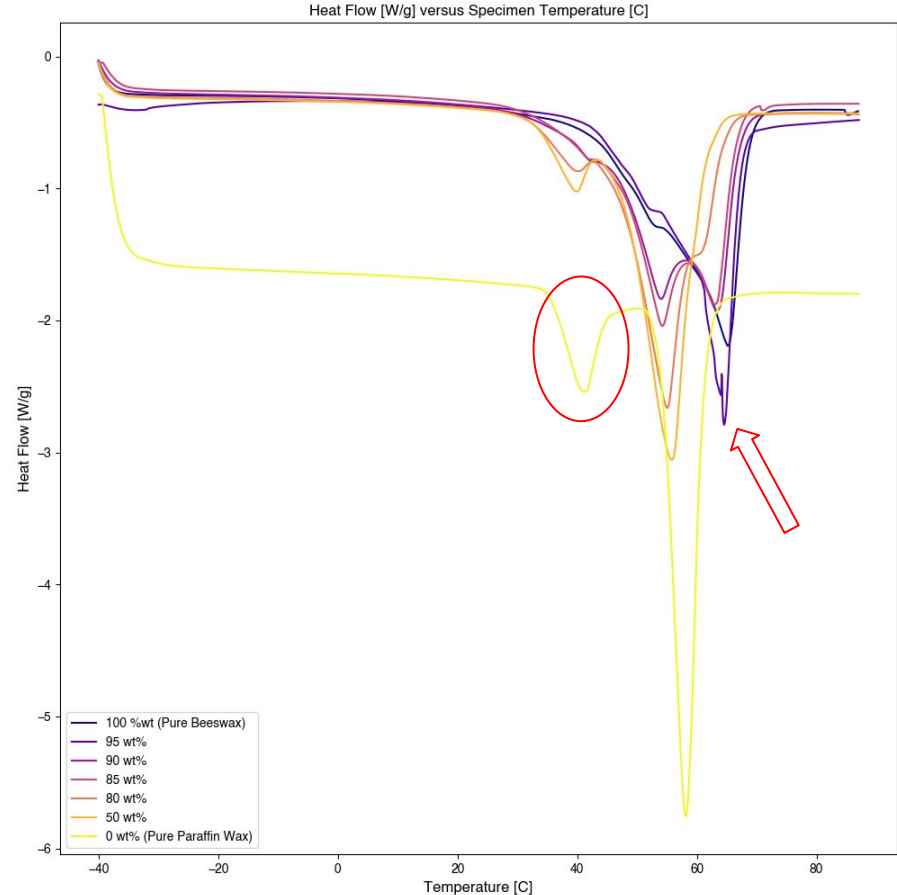
¹H-NMR

- 0.05g NMR samples are treated with 7.5 mL C²HCl₃ w/ 2% Tetramethylsilane (standard) in a test tube to form the analytical solution (¹³CNMR weighed out 300 mg, treated with 15.0 ml).
- Samples are then loaded into NMR tubes for analysis.

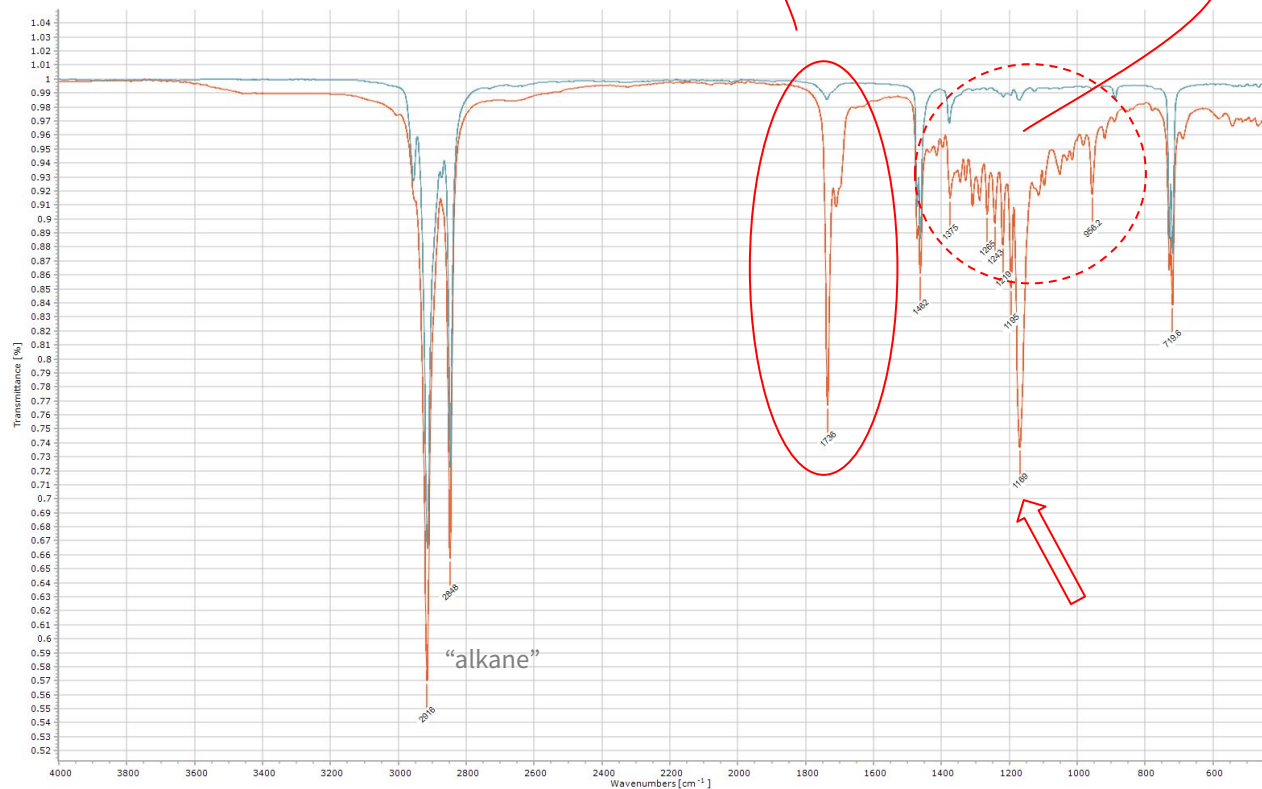
Sample	Paraffin [g]	Beeswax [g]	Total [g]
Pure Paraffin [0]	1.674	N/A	1.674
Eutectic Blend [50]	0.886	0.846	1.732 [51.1]
Pure Beeswax [100]	N/A	1.536	1.536

DSC : Differential Scanning Calorimetry

- Method of analysis where a sample is characterized through plotting of heat flow (W/g) as the sample is raised in temperature.
- This functions due to materials requiring excessive energies to overcome phase changes. Each material has specific behaviors as a function to temperature.
- Kind of useless, as an estimate of melting point for mixture of complex compounds cannot be found.
 - “Quantum effects in crystalline grains...”



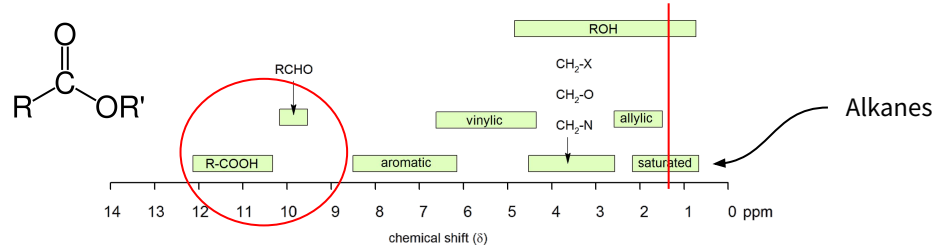
FTIR : Fourier Transform Infrared spectroscopy



" δ -lactone" = 6-membered cyclic ester

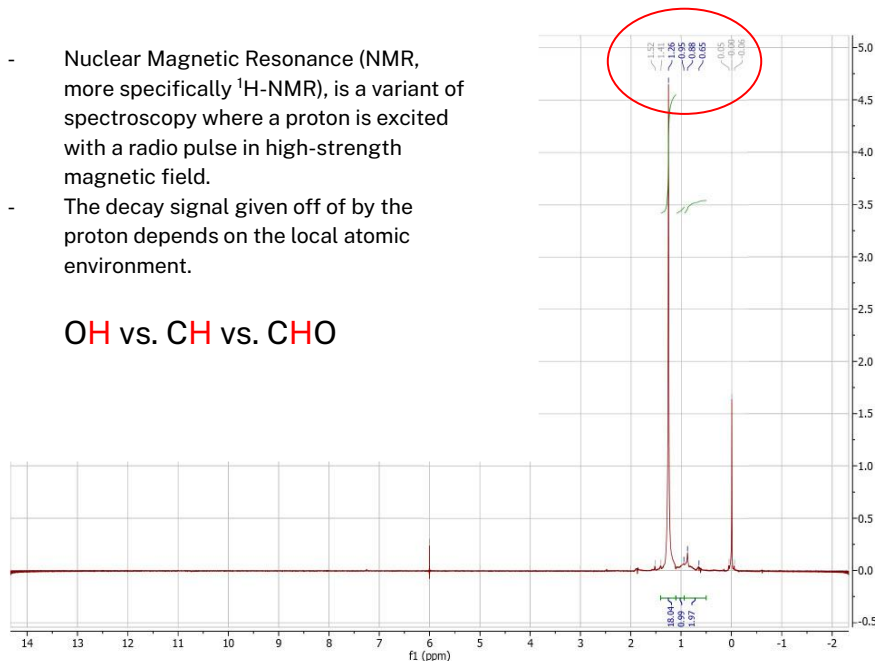
- Fourier-Transform Infrared spectroscopy (FT-IR) is similar to optical spectroscopy in where a samples' absorption to varying IR wavelengths is measured.
- Different bonds resonate at a different energy, which requires a photon of different wavelength.
- "Fourier-Transform": Deconstruct a signal into a function that shows the relative frequency of certain signals.

^1H -NMR : Proton Nuclear Magnetic Resonance spectroscopy

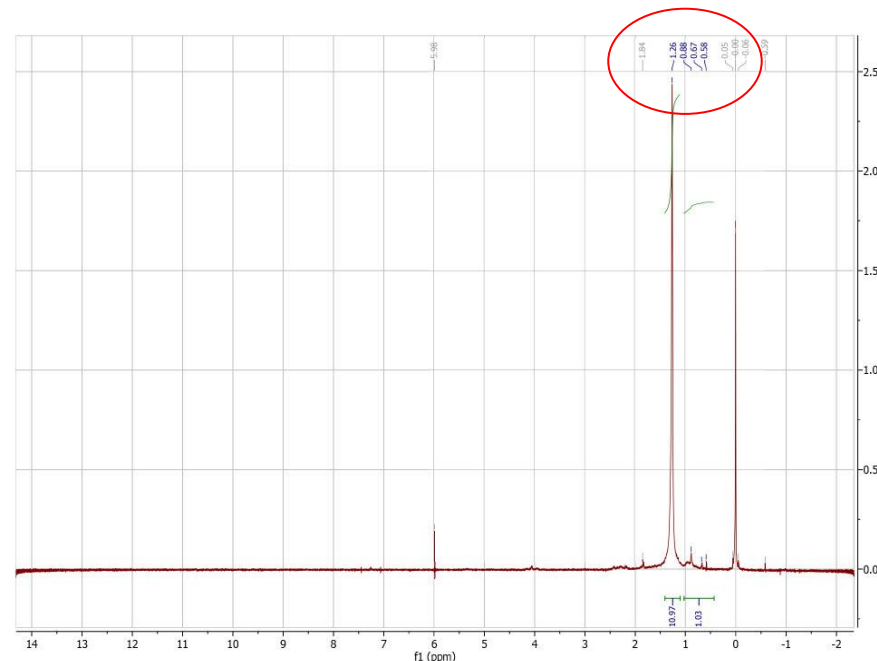


- Nuclear Magnetic Resonance (NMR), more specifically ^1H -NMR, is a variant of spectroscopy where a proton is excited with a radio pulse in high-strength magnetic field.
- The decay signal given off by the proton depends on the local atomic environment.

OH vs. CH vs. CHO

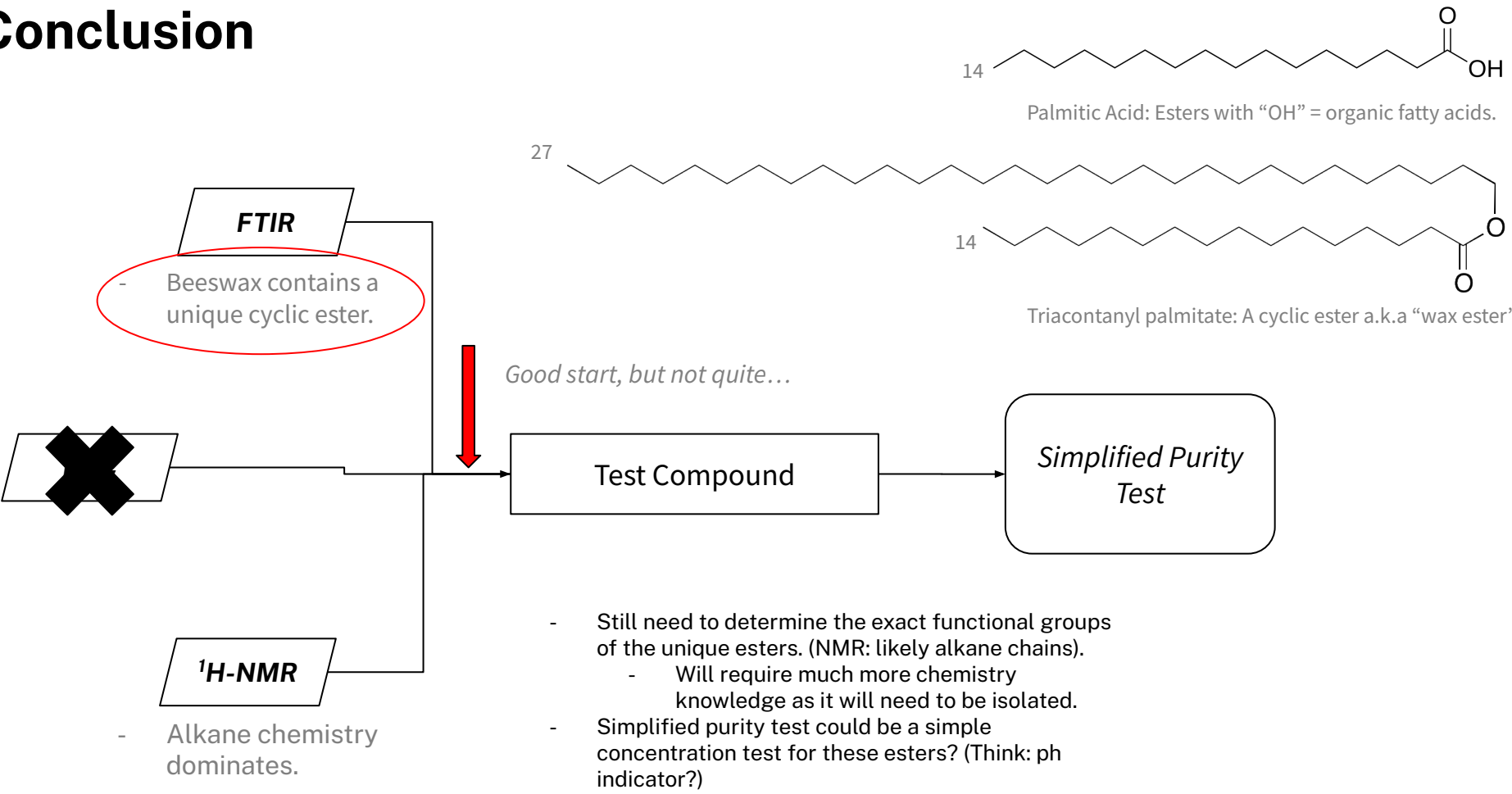


Pure Paraffin ^1H shifts.



Pure Beeswax ^1H shifts.

Conclusion



Bonus: Import vs. Domestic

