Analysis of Canola and Palm Oils via ^{1}H-NMR and FTIR January 28 - February 6, 2025
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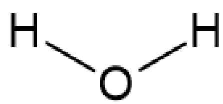
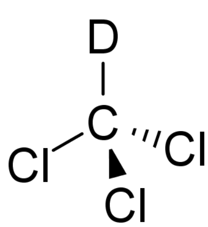
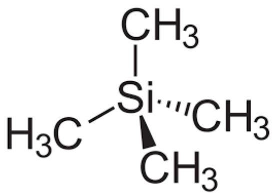
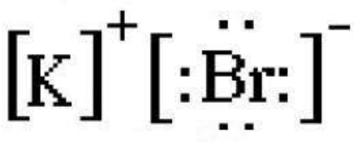
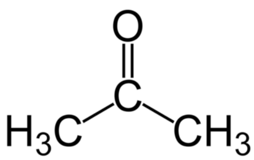
Objectives:
This experiment aims to analyze the composition, quality, and relative compositions of canola oil (CO), palm oil (PO), and a 2:1 CO-PO mixture in relation to the molecular structures of the fatty acids present in both oils via ^{1}H-NMR and FTIR.

Reagents:

Reagent Name and Structure	***Physical and Chemical Properties***	***Safety Precautions***
Deionized Water (H_2O) 	- colorless, odorless liquid - MW = 18.02 g/mol	Non-irritant, generally safe to handle Disposal: sink
Canola Oil (CO)	- light yellow liquid with a slight oil odor - less dense than water (specific gravity ~ 0.9) - comprised of oleic, linolenic, and linoleic acids (91%)	- somewhat flammable - wash with water and soap upon direct contact - keep away from drains Disposal: I104
Palm Oil (PO)	- light yellow solid with a characteristic, slight odor - has melting point range from 25 to 40 ºC - less dense than water (specific gravity ~ 0.9) - comprised of palmitic acid (44%) and oleic and linolenic acids (50%)	- somewhat flammable - wash with water and soap upon direct contact - keep away from drains Disposal: I104
Chloroform-d ($CDCl_3$) 	- clear, odorless liquid - somewhat soluble in water - denser than water (density = 1.500 g/cm^3) - does not give ^{1}H-NMR signals when used as a solvent	- can cause serious eye and skin irritation - toxic if inhaled - can cause damage to organs (liver and kidney) if ingested - can cause cancer / carcinogenic - Disposal: G703

Tetramethylsilane (TMS) 	- colorless, odorless liquid - low boiling point from 26 to 28 ºC - less dense than water (density = 0.648 g/cm^3) - yields reference signal at $\delta = 0$ ppm when mixed with sample for ^{1}H-NMR	- extremely flammable in both liquid and vapor forms - Disposal Code: G704
Potassium Bromide (KBr) 	- appears as a white, odorless, crystalline solid - very soluble in water; KBr crystals are destroyed in the presence of moisture - transparent to IR radiation; used as the material for sample holder in IR spectroscopy	- causes skin and eye irritation upon direct contact - should be handled with gloves on - can cause respiratory irritation if KBr dust is inhaled - harms aquatic life - Disposal Code: D499
Acetone 	- colorless liquid with a mildly sweet, fruity odor - soluble in water but will not dissolve KBr crystals - may be used to clean IR-related equipment (ask technicians for cleaning steps)	- highly flammable in liquid and vapor form - causes serious eye irritation - causes respiratory irritation if inhaled - Disposal Code: G704

Required Equipment:

- IRAffinity Machine
- Bruker NMR Machine
- Glassware for Producing 2:1 CO-PO Mixture (e.g. graduated cylinder)
- Glassware for Loading Oil Samples, TMS, and $CDCl_3$ for NMR Spectroscopy
- Sandwich and Pellet Setups for Liquid and Solid Samples for IR Spectroscopy

Literature Data

Table 1. FTIR Absorptions Present in Edible Fats and Oils from v = 4000 to 600 cm^{-1} (Ye & Meng, 2022)

#	Bond(s) Involved	Wavenumber *v* (cm^{-1})	Type of Vibration
1	C-H	720	C-H stretching (rocking)
2	C-O	1166	C-O stretching
3	C-H	1454	C-H bending
4	C=O	1749	C=O stretching
5	C-H	2856	C-H symmetric stretching
6	Alkene C-H	2937	Alkene C-H stretching

Table 2. ^{1}H-NMR Chemical Shifts of Signals Elicited by Edible Oils in $CDCl_3$ (Siudem *et al.*, 2022)

#	Compounds	Group Involved	Chemical Shift δ (ppm)
1	All fatty acids except linolenic acid	$-CH_2-\mathbf{CH_3}$	0.89
2	Linolenic acid	$-CH=CH-CH_2-\mathbf{CH_3}$	0.97
3	Fatty acids with acyl chains	$-(\mathbf{CH_2})_n-$	1.31
4	Fatty acids with acyl chains	$-\mathbf{CH_2}-CH_2-COOH$	1.61
5	Mono- and polyunsaturated fatty acids	$-\mathbf{CH_2}-CH=CH-$	2.04
6	Unsaturated fatty acids with acyl chains	$-\mathbf{CH_2}-COOH$	2.31
7	Linoleic and linolenic acids	$-CH=CH-\mathbf{CH_2}-CH=CH-$	2.77
8	Triacylglycerols	$-\mathbf{CH_2}-OCO-$	4.27
9	Triacylglycerols	$-\mathbf{CH}-OCO-$	5.28
10	Mono- and polyunsaturated fatty acids	$-\mathbf{CH}=\mathbf{CH}-$	5.35

Table 3. Relative Compositions of Canola and Palm Oils (*Fatty Acid Composition*, 2018)

Fatty Acid	**Lipid Number**	**wt% in Palm Oil**	**wt% in Canola Oil (Rapeseed Oil)**
Lauric	12:0	0.3	-
Myristic	14:0	1.0	-
Palmitic	16:0	**44**	4
Stearic	18:0	4	1.5
Oleic	18:1	**40**	**60**
Linoleic	18:2	**10**	**20**
Linolenic	18:3	0.2	**11**
Arachidic	20:0	-	0.5
Gadoleic Eikosadienic	20:1+2	-	1.5
Behenic	22:0	-	0.4
Erucic	22:1	-	0.4

General Observations:

References:

Fatty Acid Composition of Selected Oils and Fats. (2018, June 22). Fediol. https://www.fediol.eu/data/fatty%20acids.pdf.

Siudem, P., Zielinska, A., Paradowska, K. (2022). Application of 1H NMR in the study of fatty acids composition of vegetable oils. *Journal of Pharmaceutical and Biomedical Analysis*, *212*, 114568. https://doi.org/10.1016/j.jpba.2022.114658

Ye, Q., Meng, X. (2022). Highly efficient authentication of edible oils by FTIR spectroscopy coupled with chemometrics. *Food Chemistry*, *385*, 132661. https://doi.org/10.1016/j.foodchem.2022.132661

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