

# ***Trans Fat Determination in the Industrially Processed Edible Oils By Transmission FT-IR Spectroscopy***



**By**

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# TFA and FDA

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- FDA issued a final rule that requires the declaration of the amount of TFA present in foods, including dietary supplements, on the nutrition label by January 1, 2006.
  - Since there was no scientific basis for establishing a daily value (DV) for TFA, the final rule did not require the listing of a % DV as is required for some of the other mandatory nutrients, such as saturated fat.
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# Determination of Isolated *trans* Isomers

- ❖ Capillary GC Method
- ❖ Single-bounce attenuated total reflectance (SB-ATR) FT-IR spectroscopy method.
- Based on the observation that *trans* double bonds exhibit a characteristic and strong IR absorption band ( $967\text{ cm}^{-1}$ , H-C=C bending vibration)
- Widely employed method, particularly in the analysis of hydrogenated oils



# Factors Limiting Accuracy of Traditional AOCS Method

- All triglycerides exhibit a weak absorption band that underlie the *trans* absorption band
- Intensity of these underlying absorptions varies with triglyceride composition of the oil; can contribute 3-5 percent to the measured *trans* values.
- Sensitivity is limited by inherently short effective pathlength (~4 mm at *trans* measurement wavelength).

# FT-IR Spectral measurements

- The infrared spectra of the *trans* standards and samples were collected using Nicolet 5700 FT-IR spectrometer equipped with a 200  $\mu\text{m}$  KCl transmission cell.
- Trielaidine and partially hydrogenated oil are solids at room temperature.
- Therefore the viscosity of trielaidine standards and test samples were decreased by the addition of odorless mineral oil spirit (OMS) containing the spectral marker (0.03%) in the ratio of 1:2 to facilitate the loading of transmission cell.

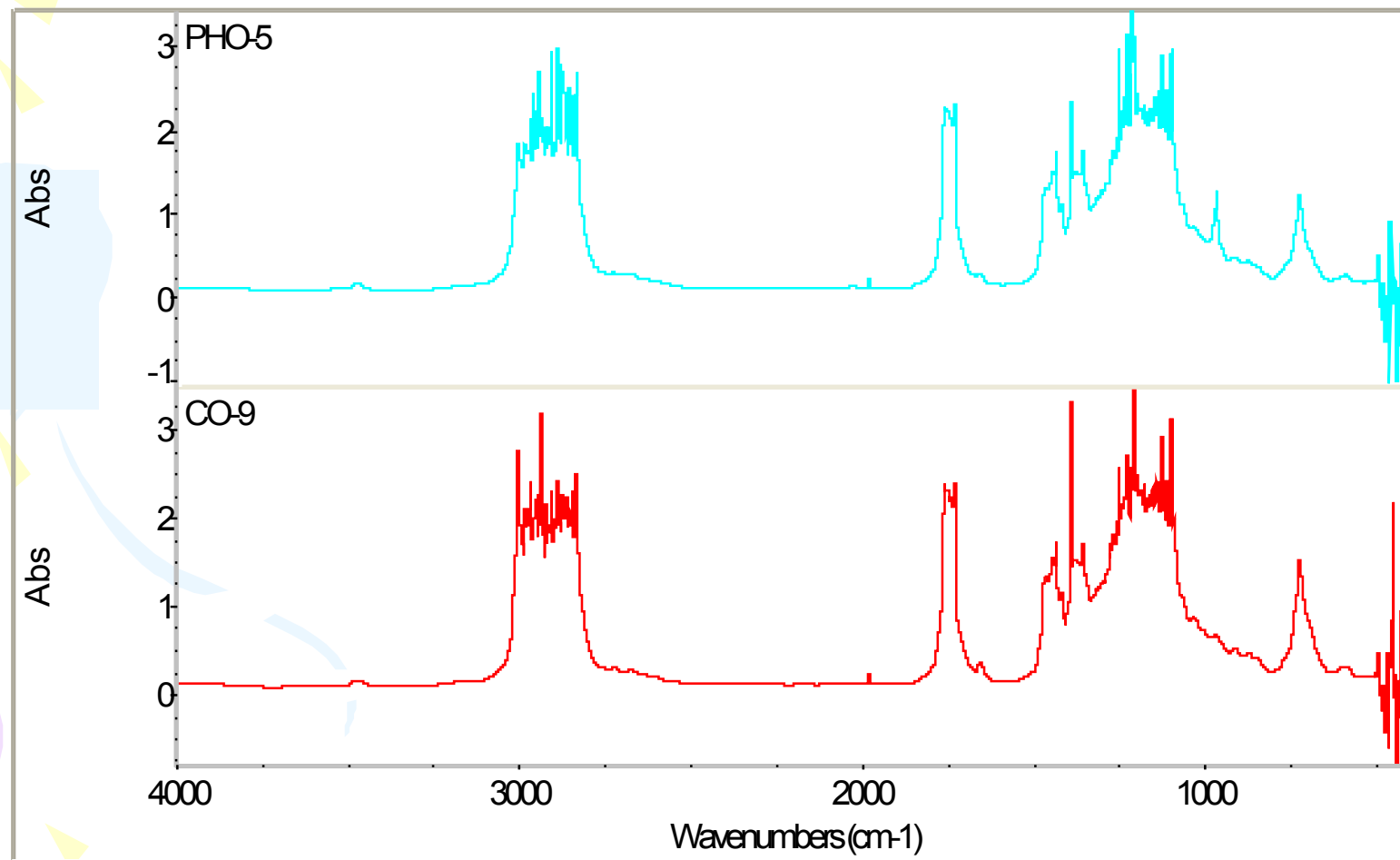
# FT-IR Spectral measurements

- Prior to the loading of 200  $\mu\text{m}$  KCl cell, all standards and samples were heated to 50  $^{\circ}\text{C}$  to avoid any crystallization during the analysis.
- A total of 32 scans were collected in the range 4000–400  $\text{cm}^{-1}$  at a resolution of 4  $\text{cm}^{-1}$ .
- The transmission FT-IR spectrum of all diluted standards and samples with OMS were recorded under the same parameters and fresh background was subtracted from the each for the accuracy in the results.

# Gas Chromatography

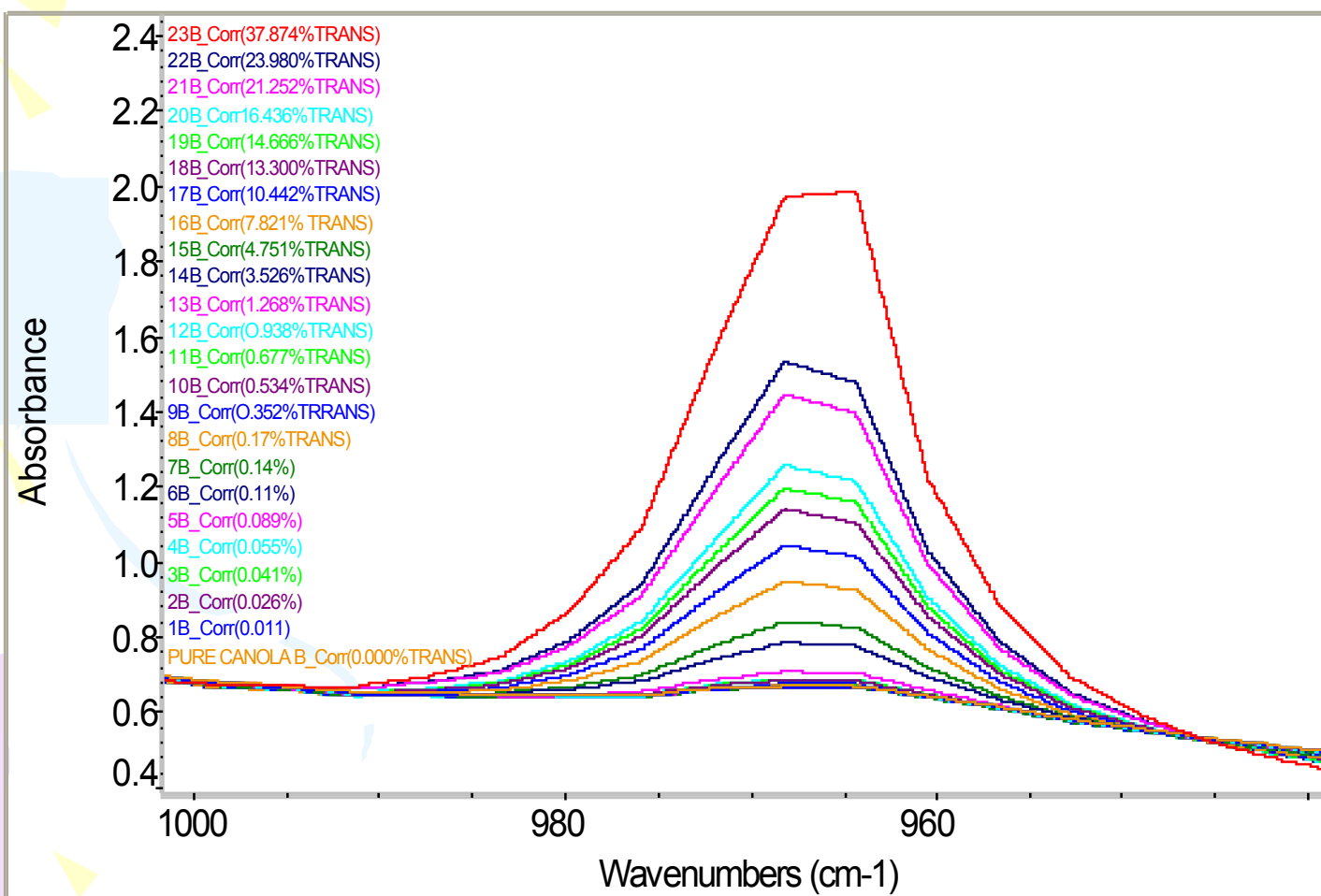
- Fatty acid methyl esters (FAMES) were prepared using standard (IUPAC method 2.301, 1979) and analyzed on a Perkin Elmer gas chromatograph (8700) a flame ionization detector.
- Oxygen-free nitrogen gas was used as mobile phase.
- Oven temperature was programmed as following:
  - The column held initially at 130 °C for 2 min;
  - Increased to final temperature 220 °C with 4 °C/min heating holding for 5 minutes;
- injector temperature, 260 °C;
- detector (FID) temperature, 270 °C;
- column flow rate, 4 ml/min;
- split ratio, 40:1;
- injected volume, 1 µL.

**Representative transmission FT-IR spectrum of partially hydrogenated oil (PH-5) with prominent *trans* peak at 967 cm<sup>-1</sup> and cooking oil (CO-9).**

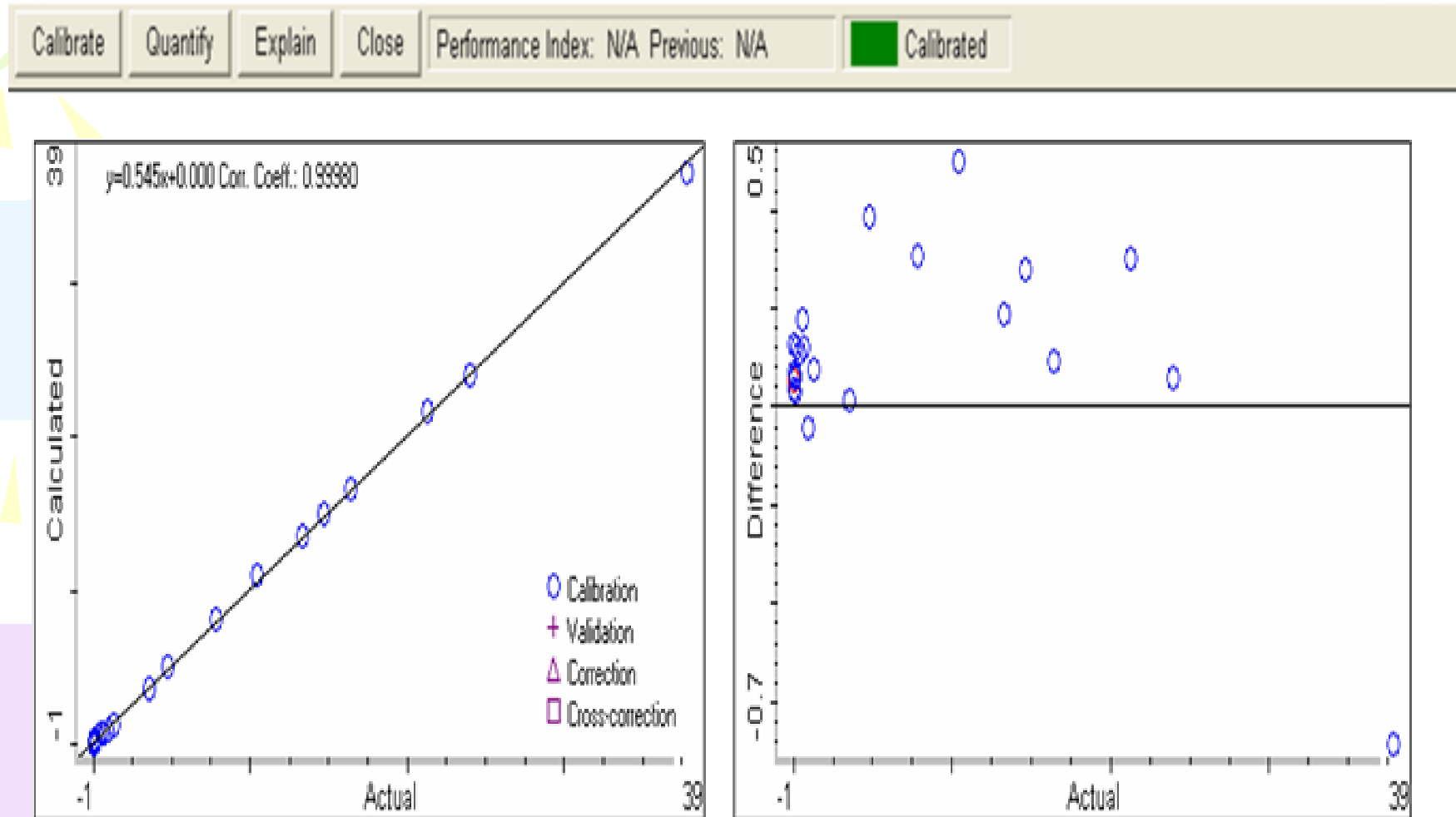




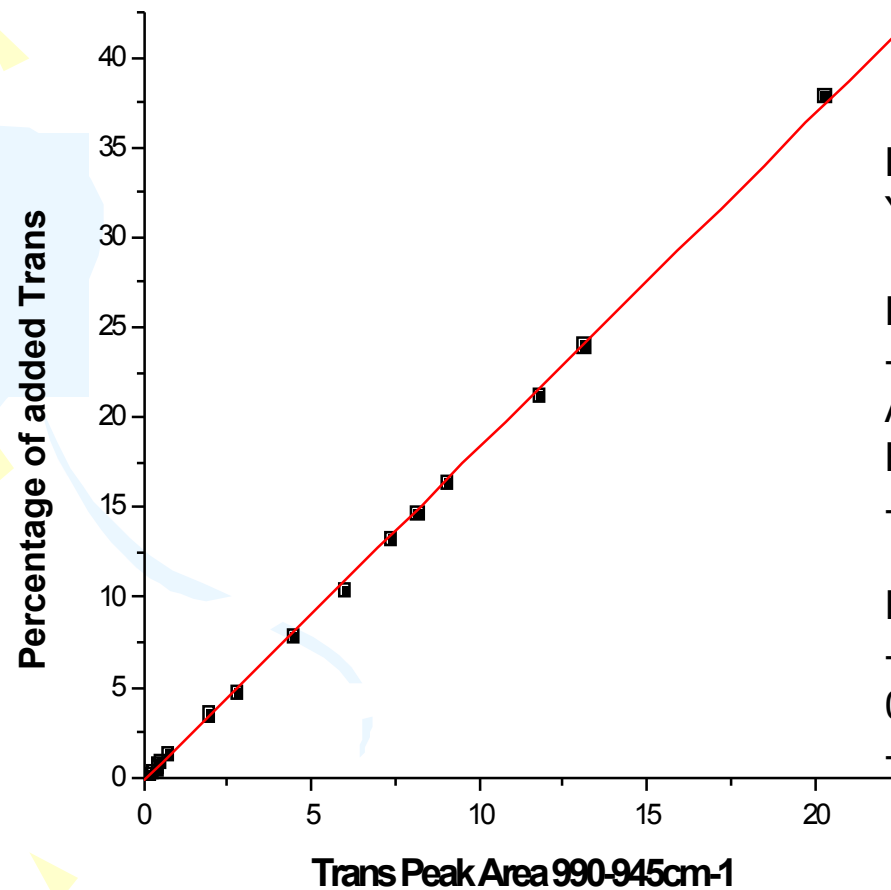
**The absorbance of *trans* band at 967 cm<sup>-1</sup> of prepared standards spiking the trielaidine in canola oil ranging from 0.011 to 37.874%.**



# TQ Analyst calibration of the *trans* standards (trielaidine added in canola oil)



# Plot of *trans* peak area versus added *trans* to canola oil



Linear Regression for Data1\_Trans:  
 $Y = A + B * X$

Parameter	Value	Error	
A	-0.15776	0.05362	
B	1.84724	0.00817	
R	SD	N	P
0.99979	0.20926	23	<0.0001

# ***Trans* fatty acids in partially hydrogenated oils (ghee) and cooking oils by GC and FT-IR**

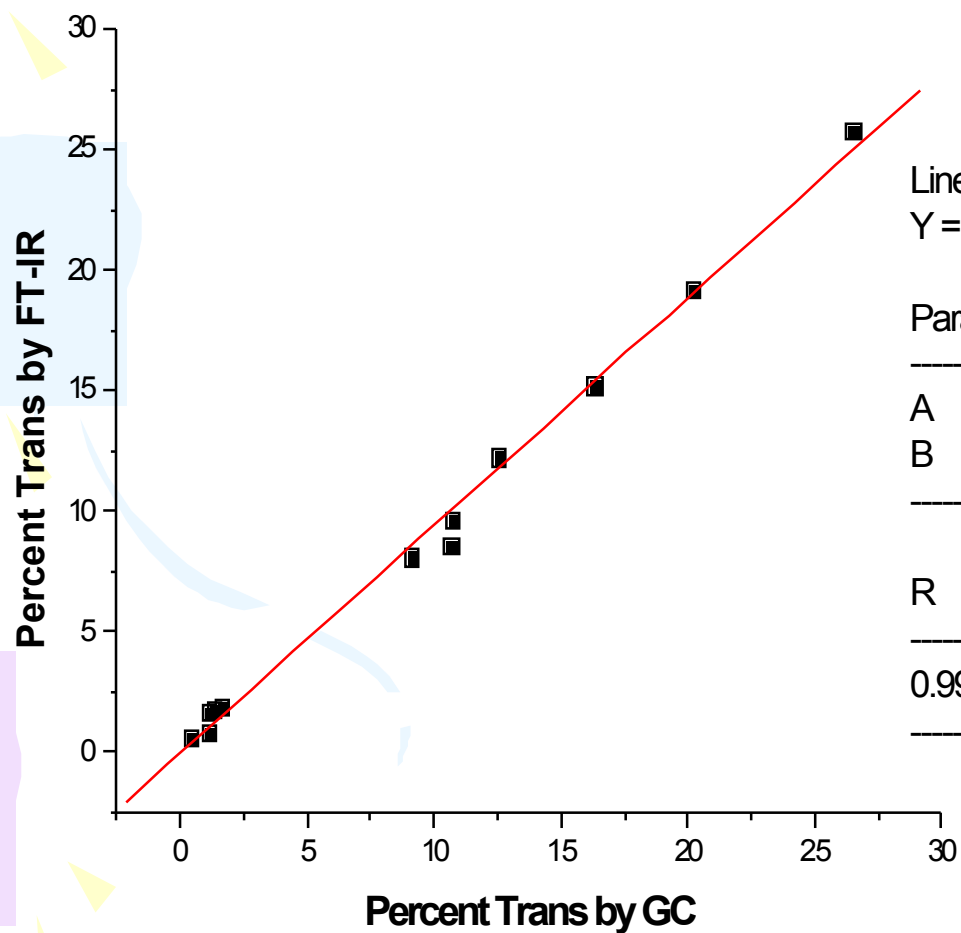
## **Samples**

## **GC**

## **FT-IR**

• PHO-1	9.12 ±0.23	8.06 ±0.02
• PHO-2	26.51 ±0.55	25.74 ±0.05
• PHO-3	10.72 ±0.64	9.61 ±0.03
• PHO-4	10.69 ±0.22	8.58 ±0.01
• PHO-5	16.32 ±0.44	15.17 ±0.04
• PHO-6	12.55 ±0.41	12.21 ±0.04
• PHO-7	20.21 ±0.61	19.19 ±0.03
• CO-8	0.52 ±0.52	0.60 ±0.01
• CO-9	0.45 ±0.16	0.61 ±0.01
• CO-10	0.52 ±0.12	0.56 ±0.01
• CO-11	1.33 ±0.21	1.76 ±0.01
• CO-12	1.63 ±0.23	1.83 ±0.01
• CO-13	1.17 ±0.16	0.83 ±0.01
• CO-14	1.14 ±0.23	1.65 ±0.01

# Plot of GC results versus transmission FT-IR



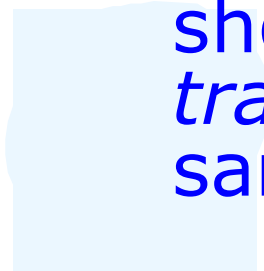

Linear Regression for Data1\_B:  
 $Y = A + B * X$

Parameter	Value	Error
A	-0.02779	0.28685
B	0.94397	0.02307

R	SD	N	P
0.99703	0.65764	12	<0.0001



# Conclusion

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- The results of transmission FT-IR spectroscopy were found in good agreement with the GC results and have shown slightly better sensitivity for low *trans* values in the analyzed edible oil samples.
  - All hydrogenated edible oils have shown higher amount of *trans* fat by the both GC and transmission FT-IR spectroscopy, which is very dangerous for the health of consumers.
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# Thanks



NCAC