

TRANS OLEFINS IN FATTY ACIDS, ESTERS OR TRIGLYCERIDES BY IR SPECTROSCOPY

UOP Method 841-83

SCOPE

This method is for determining isolated (non-conjugated) trans olefin isomers in long-chain carboxylic acids, esters or triglycerides by infrared spectroscopy. As a trend analysis, this method can be used to estimate the relative amount of unsaturation in esters, triglycerides or fatty acids. It is, however, not applicable to any materials containing constituents which have functional groups that would interfere with the band near 970 cm^{-1} , or to fats and oils containing large quantities of conjugated unsaturation (see Note 1).

OUTLINE OF METHOD

A suitable size sample is weighed into a tared volumetric flask and diluted with carbon disulfide. The infrared spectrum is recorded from 1020 to 900 cm^{-1} . The absorbance band near 970 cm^{-1} , which is characteristic of and specific for the C-H deformation of an isolated trans olefin double bond, is determined and the absorptivity calculated. By comparing this to the absorptivity of a standard, the amount of trans olefin isomer present is calculated.

APPARATUS

Balance, readability of 0.1-mg

Cell, absorption, matched KBr or NaCl, 1.0-mm path length, Perkin-Elmer Corp., Cat. No. 0126-1251, or equivalent

Chart paper, continuous grid, linear transmission

Flask, volumetric, 5- and 10-mL

Infrared spectrophotometer, Perkin-Elmer Corp. Model 580B, Cat. No. 0280-0001, or equivalent

Pipets, uncalibrated, disposable, capillary, Pasteur, 145- and 230-mm

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UOP Methods are available through ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken PA 19428-2959, United States. The Methods may be obtained through the ASTM website, www.astm.org, or by contacting Customer Service at service@astm.org, 610.832.9555 FAX, or 610.832.9585 PHONE.

REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified.

Carbon disulfide, dry, Fisher Scientific, Co., Cat. No. C-573, or equivalent (see Note 2)

Standards. One or more of the following chemicals are required depending upon whether an acid, ester or triglyceride is to be analyzed:

9-Octadecenoic acid, (elaidic acid), 98% purity, Pfaltz & Bauer, Inc., Cat. No. E-00480, or equivalent

9-Octadecenoic acid, methyl ester, (methyl elaidate), 98%, purity, Pfaltz & Bauer, Inc., Cat. No. M-16480, or equivalent

9-Octadecenoic acid, 1,2,3-propanetriyl ester, (trielaidin), 98% purity, Analabs, Inc., Cat. No. LGL-018, or equivalent

CALIBRATION OF APPARATUS

The standard chosen for calibration of the instrument is dependent upon the type of sample to be analyzed. Acid samples are calibrated with elaidic acid, esters with methyl elaidate and triglycerides with trielaidin. Weigh 0.1 g to the nearest 0.1 mg of the appropriate standard into each of three tared 10-mL volumetric flasks. Dilute to the mark with carbon disulfide (CS₂). Fill one of the matched 1.0-mm KBr cells with CS₂ and place it in the reference beam of the instrument (see Note 3). Fill the other cell with one of the standard solutions and place it in the sample beam of the instrument. Allow the cells to remain for 5 minutes (see Note 4). For the Perkin-Elmer 580B, use the following instrument settings.

	100% T
Ordinate Expansion:	1
Single Scan:	Limited Range
Range:	1020 to 900 cm⁻¹
Abscissa Expansion:	5
Integrated Scan Mode:	2

For settings of other instruments, consult the operator's manual (see Note 5).

Obtain the infrared spectrum, from 1020 to 900 cm⁻¹, of each of the standard solutions (see Note 6). Measure the absorbance for each as described in *CALCULATIONS* and determine the average absorbance of the three solutions for each standard. Then calculate the absorptivity and use this value as "a" in *CALCULATIONS* to determine the mass-% trans olefin in the sample.

PROCEDURE

Weigh a portion of the liquid, or carefully melted sample, into a tared 5-mL volumetric flask and fill to volume with CS₂. Usually 0.1 - 1.0 g is satisfactory. Fill one of a matched pair of 1.0-mm cells with CS₂ and place it in the reference beam of the instrument (see Note 3). Fill the other matched 1.0-mm cell with the sample solution and place it in the sample beam of the instrument. When the highest point in the spectrum is set to 95% transmission, the lowest point of the band near 970 cm⁻¹ should fall between 20 and 70% transmission. If this does not occur, weigh a new portion of the sample using either more or less material so that the band falls within this range. Allow the cells to remain in the instrument for 5 minutes (see Note 4). Then obtain the infrared spectrum of the sample from 1020 to 900 cm⁻¹ (see Note 5).

CALCULATIONS

Draw the baseline of the transmittance spectrum (see Fig.). For acids this is a straight line between 990 and 939 cm^{-1} , for esters from 998 to 944 cm^{-1} and for triglycerides the baseline is drawn from 995 to 937 cm^{-1} . Determine the percent incident radiation which is equivalent to distance XZ (I_0) shown on the elaidic acid spectrum of the attached Typical Spectra. Determine also the percent transmitted radiation which is equivalent to distance XY (I). Convert these percentages to absorbance as follows:

$$\text{Absorbance} = \log_{10} \left(\frac{I_0}{I} \right)$$

where:

I_0 = incident radiation, %

I = transmitted radiation, %

Calculate the absorptivity of the standard or sample using the following equation:

$$a = \frac{A}{bc}$$

where:

A = absorbance

a = absorptivity

b = path length of the cell, mm

c = concentration, g/L

Calculate the mass percent trans olefin using the following equation (see Note 6):

$$\text{Trans olefin, mass-\%} = 100 \frac{a(\text{sample})}{a(\text{standard})}$$

where:

a is as previously defined

Example Calculations

$$I_0 = 70.2\%$$

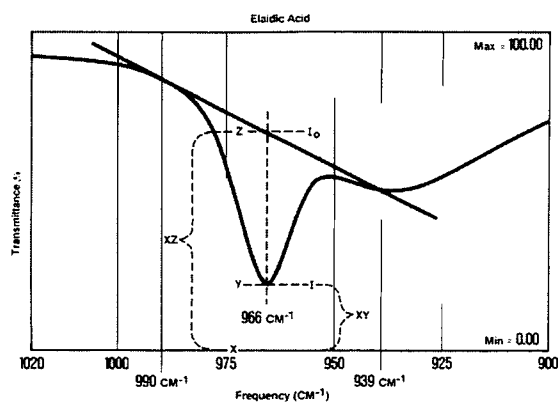
$$I = 20.6\%$$

$$\text{Sample weight} = 0.1902 \text{ g/5 mL CS}_2$$

$$A = \log_{10} \left(\frac{70.2}{20.6} \right) = 0.5325$$

$$a = \frac{0.5325}{1.0(\text{path length}) \times 38.04 \text{ g/L(conc.)}} = 0.0140$$

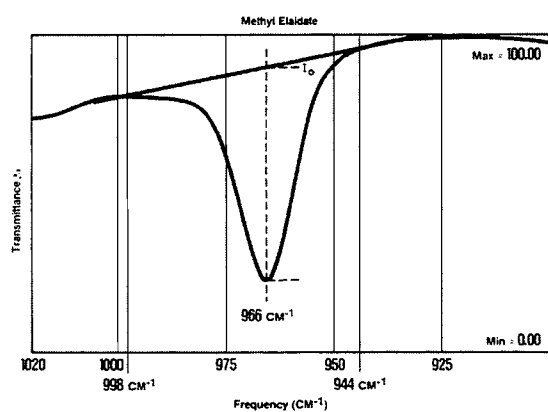
$$\text{Trans olefin, mass-\%} = \frac{0.0140 \text{ (a of sample)}}{0.0574 \text{ (a of standard)}} \times 100 = 24.4\%$$



$XZ = I_0 =$ Incident Radiation, %

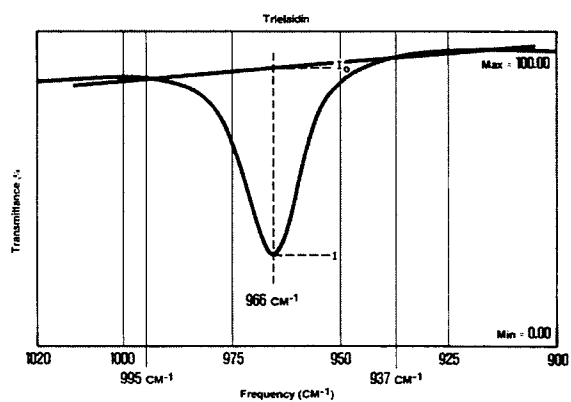
$XY = I =$ Transmitted Radiation, %

Acid



Typical Spectra

Ester



Triglyceride

NOTES AND PRECAUTIONS

1. Long-chain fatty acids containing less than 15 mass-% isolated trans olefin isomers must be converted to methyl esters before analysis by the method published by Schlenk and Gellermann.²

2. Carbon disulfide is a hazardous material. Appropriate precautions regarding the flammability and toxicity through skin absorption or prolonged exposure to vapors at high concentration should be taken. A well ventilated area equipped with a fume hood for sample preparation is recommended in addition to other precautions taken.

3. Carbon disulfide is a very volatile liquid and may eventually evaporate from the cell upon prolonged exposure to the heating effect of the infrared beam. The reference cell should be checked before each standard or sample is run to ensure that the cell is full.

4. It is recommended that N₂ purging of the instrument sample and reference compartments be used to minimize atmospheric absorptions in the range of interest.

5. Samples must be run in the same instrument at identical parameters and conditions as the standards for reproducible data. Any change in operating parameters or conditions of the instrument necessitates recalibration.

6. In calculating the mass-% trans olefin in triglycerides, the absorptivity of the standard triglyceride is used. If acids are being analyzed, the acid standard must be used. The same applies for esters.

PRECISION

Based on 6 pairs of analyses, the estimated standard deviation (esd) for trans olefin at the 15 to 30 mass-% level was calculated to be 0.15%. Duplicate results by the same operator should not differ by more than 0.50 mass-% (95% probability) at the stated level.

TIME FOR ANALYSIS

The elapsed time for one analysis is 0.75 hour. The labor requirement is 0.75 hour. The elapsed time and labor requirement for a series of similar samples, which are run one after another, is somewhat less.

REFERENCES

1. ASTM Method E 169, www.astm.org
2. Schlenk, H., and Gellermann, J. L., "Esterification of Fatty Acids with Diazomethane on a Small Scale," *Anal. Chem.* **32**, 1412-1414 (1960)
3. Tentative Method Cd 14-61 (reapproved 1973); Official and Tentative Methods for American Oil Chemists Society (3rd Edition)

SUGGESTED SUPPLIERS

Analabs, Inc., 80 Republic Dr., North Haven, CT 06473
Fisher Scientific Co., 1600 W. Glenlake Ave., Itasca, IL 60143
Perkin-Elmer Corp., Instrument Div., Norwalk, CT 06856
Pfaltz & Bauer, Inc., 375 Fairfield Ave., Stamford, CT 06902