

DOWNLOAD PDF OFFICIAL METHODS FOR THE DETERMINATION OF TRANS FAT

Chapter 1 : AOAC Analytical Methods

This monograph describes the most common gas chromatographic and infrared spectroscopic official methods required for the determination of trans fatty acids for food labeling purposes. The authors review the current status and limitations of the latest methods and discuss the numerous factors that may have an impact on accuracy and precision.

Abstract Trans fat is a saturated fatty acid with trans configuration and separated double bonds. Analytical methods have been introduced to analyze trans fat content in foods including infrared IR spectroscopy, gas chromatography GC, Fourier transform-infrared FT-IR spectroscopy, reversed-phase silver ion high performance liquid chromatography, and silver nitrate thin layer chromatography. Trans fat content in 6 vegetable oils were analyzed and processing effects including baking, stir-frying, pan-frying, and frying on the formation of trans fat in corn oil was evaluated by GC. Among tested vegetable oils, corn oil has 0. Among cooking methods, stir-frying increased trans fat in corn oil whereas baking, pan-frying, and frying procedures did not make changes in trans fat content compared to untreated corn oils. However, polyunsaturated fatty acids having double bonds with conjugated forms such as conjugated linoleic acid are not categorized as trans fat due to their health beneficial influence. Hydrogenation, which uses hydrogen gas and metal catalysts to reduce the degree of unsaturation and provide proper physical characteristics in oils since s, is a major process to generate trans fat in our daily diet. Margarine, shortening, and butters are well-known solid fat containing trans isomers. Other sources of trans fat in our daily diet are microorganisms in the rumens of ruminant animals through biohydrogenation. *Roseburia hominis* AT, *Roseburia inulinivorans* AT and *Ruminococcus obeum*-like strain A can produce trans fatty acids from ruminant sources 1, 2. The profiles of trans fat from commercially available partially hydrogenated oils and natural resources are not the same. Detrimental health implication of trans fat diet has been filed up for more than four decades. Epidemiologic and biochemical evidence confirmed that excessive trans fats in the diet are a significant risk factor for cardiovascular disease. Health effects of trans fat could be due to the structures of trans fat 1, 2. The melting point of stearic acid C However, this IR method has some limitations. The advanced techniques for trans fat analysis in fats and oils are using FT-IR spectroscopy. These FT-IR methods used carbon disulfide like IR method, which may lead to stratification, vapor and air bubbles within cells. FT-IR can determine the content of trans fat with high correlation coefficient without using carbon disulfide. Some disadvantages of FT-IR method for determining trans fat are overestimating the content of trans fat, interference from conjugated fatty acids, and the expressing results as equivalent of elaidic acid C When light strikes two different medium at normal condition, some light partially transmitted and partially reflected. However, in internal reflection mode, light is reflected inside the crystal and evanescent wave is then propagating away from the surface of the crystal like trans fat. This technique requires prior extraction of fat from food samples while further derivatization and use of harmful solvents were not necessary 8. Conventional GC for analysis of FAME use a slightly polar column using polyglycol Carbowax because natural oils have majorly cis form of double bonds. American Oil Chemists Society AOCS 11 approved official method Ce 1h for the determination of cis, trans-saturated, monounsaturated, and polyunsaturated fatty acids in vegetable or non-ruminant animal oils and fats by capillary GC with a flame ionization detector FID. Also, Official Method of Analysis Different detectors like mass spectrometry MS coupled with GC were introduced for trans fat analysis In case of foods from ruminant animals like dairy products, different extraction or GC conditions are needed due to a large number of short chained fatty acids and the variety of different molecular structures Total trans fat measured by IR can be corrected using a formula: Reversed-phase HPLC can separate fatty acids based on the apparent carbon number, which is the number of carbons - 2x number of double bonds. Silver salts can form more stable complex with cis isomers and AgNO₃ rather than trans isomer. Analysis of fatty acid profiles in one of typical procedures in food industry because providing information on the content of trans fat should be included in food label. Accurate information on the content of trans fat in foods can come from proper techniques for

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extracting lipids, saponification, and methylation of fatty acids. Several official methods have been approved by any official national or international entity. As more accurate and precise methods and technologies are developed, some official methods are not frequently used whereas some are still valid. In Korea, officially approved analysis methods have been suggested in Korea Food Code. All the government agencies and laboratories from food industry should use this official method to certify the content of trans fat in the products. Many reports have conducted on the fatty acid profiles and trans fat content in edible oils. However, effects of conventional cooking procedures including baking, stir-frying, pan-frying, and frying on the formation of trans fat in edible oils are scarce in the literature. The objective of this study was to analyze trans fat content in commonly consumed vegetable oils and to find out effects of conventional cooking treatment on the formation of trans fat. Triundecanoin, elaidic acid, mixtures of standard fatty acids, and BF₃ were purchased from SigmaAldrich St. Other chemicals were purchased from Daejung Chemical Co. Vegetable oils including corn, rapeseed, soybean, olive, perilla, and sesame oils were obtained from 18 large glossary markets located in 9 different cities in Korea for the purpose of Korean TDS from the year of to the year of Best-selling branded vegetable oils, which were purchased from 18 different sampling sites, were sampled equal amount and blended to make a pool of oil. Each oil sample was provided in triplicate with separated bottles. Corn oil in the pan was stirred continuously by a spatula for 2 min. Frying was done using a shallow pan diameter of 30 cm and height of 20 cm. Corn oils sample with different cooking procedure were prepared in separated 3 bottles. Fatty acid analysis by gas chromatography with a flame ionization detector FID. The flow rate of helium carrier gas was 0. Peaks of GC chromatograms were identified comparing the retention times of a mixture of standard fatty acid methyl esters SigmaAldrich. Each peak of fatty acid was quantified using an equivalent of the concentration of the internal standard. Samples were separately analyzed in triplicate. The retention times for internal standard C Mixtures of standard fatty acids were well separated using current analysis method.

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Chapter 2 : Analysis of Trans Fat in Edible Oils with Cooking Process

This monograph describes the most common gas chromatographic and infrared spectroscopic official methods required for the determination of trans fatty acids for food labeling purposes. The authors review the current status and limitations of the latest methods and discuss the numerous factors that.

Since the first infrared spectra of fatty acids FA and vegetable oils were published by Coblentz in , there has been a growing recognition by researchers and the food industry of the value of vibrational spectroscopy in lipid analysis. Besides the fundamental role of mid-IR spectroscopy in basic research on lipids, its single most notable application for producers and processors of fats and oils has been the determination of total trans FAs. Near-IR NIR spectroscopy cm^{-1} offers other advantages, which account for its widespread use in the agricultural and food industries [2]. NIR spectra exhibit low-absorptivity combination and overtone bands of the fundamental vibration modes. As a result, longer pathlengths can be used that allow for the measurement of opaque, intact test samples e. Besides transmission and diffuse reflection, the transflection mode with fibre optic probes is commonly used in NIR measurements. These advantages have made it possible for NIR spectroscopy in conjunction with multivariate statistical analysis to be routinely applied to compositional determinations, classifications, and prediction of functionality. Advanced chemometrics, such as partial least squares PLS regression, are required for the analysis of observed NIR spectra due to the overlap of broad combination and overtone bands. It also increased interest in the development of more rapid and equally reliable methods as gas chromatography GC for the determination of the FA composition of fat and oil products to monitor compliance with food regulations, such as those that require the declaration of the total fat, trans fat, and saturated fatty acid SFA contents, and to verify the values provided on the product labels. Currently there is no unanimous consent among countries as to which trans FAs are to be included in the definition of total trans FA content [3]. Based on a strict chemical definition this would include all FAs having one or more double bonds in the trans configuration, irrespective of whether it is industrially produced during partial hydrogenation, naturally produced in ruminants, or as a result of food processing. The USA, Canada, the EU and Codex Alimentarius have also attempted to differentiate between the origins of the trans FAs by excluding from the definition of total trans FA content those that contain conjugated trans double bonds [3]. This is not supported by scientific evidence; for example the isolated trans While most regulatory policies assign manufacturers the responsibility of selecting an appropriate method for the determination of FA composition, the selected method has ensuring the validity of the declared nutrient values on labels of food products. In addition, the selected methods must allow the identification of the different trans FA isomers to meet various regulatory standards. The body of literature on vibrational spectroscopy is vast and the number of applications is extensive. However, the present article on the application of IR to the determination of the FA composition of fats and oils is limited to two recent developments. It is focused specifically on: Rapid Determination of Total trans Fat by Mid-infrared Spectroscopy A significant number of mid-infrared IR spectroscopic procedures and several official methods for quantifying total trans FAs with isolated double bonds have been published over the past sixty years as interest in an optimized methodology steadily increased. An introduction to the scope of this mid-IR determination as well as a discussion about its strength and limitations is given below. The determination of total isolated trans FA in fats and oils by various mid-IR procedures [11,12] and official methods [] is based on the measurement of the unique C-H out-of-plane deformation band observed at cm^{-1} Fig. In partially hydrogenated fats and oils, these trans double bonds are found mostly in trans More complex mixtures of trans-containing PUFA and their positional isomers may occur in partially hydrogenated fish oils. Mid-IR methodology has been extensively used in the edible fats and oils industry. Expanded region of mid-IR absorption spectra for trans fats. The deformation band for isolated trans double bonds is observed at cm^{-1} Many modifications have been proposed to improve accuracy, but only some transmission [] and more recently ATR [] mid-IR official methods succeeded in partially improving this

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determination. This is because several previously undetected interferences, due to SFA, conjugated FA, and other minor products, adversely affected accuracy and precision. These issues were significantly minimized when the so-called negative second derivative ATR-FTIR procedure was proposed [12], validated, and approved as official method in [17]. The approach required the analysis of all fats and oils as FAME irrespective of the trans fat content. A brief description is given next. This approach requires the accurate weighing of FAMES and their dissolution in known volumes of volatile and toxic carbon disulfide solvent. FAME solutions are subsequently measured in non-demountable transmission cells with a 1-mm fixed-pathlength. FAME calibration standards consisting of known mixtures of methyl elaidate trans The total concentration of unknown test samples is also set at this concentration. This method assumes that the trans FA to be determined are only methyl elaidate, and that the absorptivities of the cm^{-1} band for all trans FAs and their positional isomers are identical. This methodology requires the generation of two linear regression calibration functions, one for a first set of standards with t To measure the cm^{-1} band height, a straight line is first drawn between two points along the sloping baseline of the infrared spectrum Fig. The positions of these two minima points are not the same and have to be moved closer to each other as the intensity of the trans FAME band at cm^{-1} decreases. The judgment of analysts in drawing baselines has been a significant source of error, and more reliable alternative procedures were subsequently developed. This ATR method was successfully used to rapidly 5 min measure the cm^{-1} trans fat absorption as a symmetric band on a nearly horizontal baseline Fig. The experimental setup of this ATR-FTIR official method offered several advantages and was simpler than the one used for transmission measurements. This method requires the measurement of trans fats relative to a reference material consisting of a trans-free oil. The internal reflection approach eliminated the need to weigh test portions and quantitatively prepare solutions in CS_2 . Traditionally, CS_2 had been used as the reference material in the vast majority of transmission procedures and official methods [11,13,14]. When a trans-free oil reference material was used instead of CS_2 , the sloping baseline of the cm^{-1} trans band became nearly horizontal [11,15,16,18,19]. Therefore, the contributions of the TAG absorptions that led to an elevated and sloping baseline in the first place were removed, and the requirement to convert TAG to their corresponding FAME was eliminated. Having a nearly horizontal baseline significantly reduced the uncertainty in measuring the band height or area at all trans fat levels. Expanded region of mid-IR absorption spectra for trans fats measured relative to a trans-free reference oil. The band areas are integrated in the fixed range between and cm^{-1} It is noted that larger ATR crystals allow the infrared beam to bounce multiple times through the test portion under investigation. This means that the larger the number of internal reflections will result in greater sensitivity and will enhance the signal-to-noise ratio. The ATR element surface must be completely covered for accurate quantification and extreme care should be taken in order to avoid cross contamination. To fully satisfy this last requirement, cleaning the ATR element with ethanol is recommended. The cm^{-1} trans band for trans The area of the trans band could then be integrated electronically between the limits of and cm^{-1} Fig. The resulting regression function relating the integrated area of the absorption band and the amount of trans Similarly, unknown fats and oils test portions are measured relative to the same trans-free reference oil used for calibration. The trans fat or oil level as percent of total fat is then calculated by substituting the value of the integrated area of the trans band in the linear regression function. As stated above, this ATR-FTIR method also assumes that the major component to be determined in unknown melted fat or oil test samples is trielaidin. The FA composition of the trans-free reference oil is a critical aspect of this method. The FA composition of this trans-free reference oil should match as much as possible that of every unknown fat or oil being determined. If the selected trans-free oil is significantly different in composition from that of the fat under investigation, it will adversely impact the accuracy of quantification. For a complex matrix such as milk fat [20], finding a matching trans-free reference material proved to be difficult. This procedure did not require the use of a trans-free oil, instead it referenced the measurement of trans fat relative to air open-IR beam. This aspect of the procedure eliminated both the baseline offset and slope of the trans IR band cm^{-1} as well as the need to measure test samples relative to a trans-free reference fat. Because second derivative

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spectroscopy enhances the curved features of an absorbance band and exhibits narrower bandwidths, for the first time it was possible to observe the presence of weak interference bands close to the trans absorption band at cm^{-1} . One type of interference was due to bands near or below cm^{-1} , which were confirmed to be due to the presence of saturated fat components [24]. For a fully hydrogenated soybean oil test sample [21] a band at cm^{-1} was erroneously attributed to a 1. It was subsequently recognized that it should have been attributed to the saturated tristearin TS, The saturated trilaurin TL, Expanded region of the negative second derivatives of mid-IR absorption spectra for trans fats. For coconut oil dotted line that is high in saturated fat, and contains a trace level of trans fat not detectable by mid-IR, a band near cm^{-1} was observed. An analytical solution to this problem based on standard addition was proposed [26]. In the case of conjugated FAs, however, second derivative spectra led to the total elimination of any band overlap [24]. This negative second derivative ATR-FTIR procedure was subsequently validated in an international collaborative study [24], and recently applied to the rapid determination of the total trans fat content of commercial fats and oils [25] for compliance verification with food labeling regulations. The FT-NIR procedure was proposed [] and recently evaluated in a limited collaborative study [31], but has not been fully validated. A discussion is given next about the development of this procedure and its scope, advantages, and limitations. Although ATR-FTIR has made significant strides towards the accurate determination of the total trans fat content, one still needed to rely on GC official methods for the determination of the total saturated fat content in order to comply with regulatory requirements. On the other hand, recent advances in FT-NIR spectroscopy have made it possible to determine not only the total trans FA content, but also the FA composition of a fat or oil [] without the need for prior derivatization to volatile FAME derivatives as required for GC analysis. While this FT-NIR procedure shows great potential, it still needs to be collaboratively studied in multiple laboratories and approved as an official method. All GC peaks must be reported and accounted for, the resolution of all GC components should be optimized, and quantification should be accurate. This is necessary because FT-NIR spectra are collected for the undiluted and underivatized neat fats and oils. They are strongly matrix dependent and represent all the constituents of the matrix including non-TAG components. Two assumptions were made during method development: The currently available very long or m flexible fused silica capillary columns coated with highly polar stationary phases have made it possible to maximize the resolution of most FAME, including all trans FAMES and their positional isomers. All GC peaks should be accounted for, including unidentified minor ones that may be attributed to unknown FAME components or non-lipid matrix constituents. While the identification of all unknown GC peaks is not essential, their calculated concentrations must however be accounted for, included in the total fat content, and incorporated into PLS calibration models. If GC peaks attributed to non-lipid constituents are produced e. It is therefore recommended to remove such artifacts by using thin layer chromatography to purify the prepared FAME products prior to GC analysis [35]. With regard to the resolution of overlapping GC peaks, it is important to make every effort to resolve co-eluting or overlapping pairs of peaks. One recommended protocol entails the use of two different temperature programs [36] with the same GC instrument and column. The GC profiles from the two temperature programs should be sufficiently different to produce recognizable changes in the relative elution pattern of overlapping components, such that a co-eluting pair of peaks under one set of conditions do not co-elute again under a second temperature program. To improve accuracy of GC determinations, theoretical correction factors are recommended for the quantification of the flame-ionization detector response [9,37]. For FA profiling, the most important bands are due to CH_2 , $-\text{CH}_2$ and $-\text{CH}_3$ groups that originate from either the FA chain or the glycerol moiety of the TAG and phospholipid molecules, or other constituents present in a lipid test portion. Thus, it is difficult to attribute or assign an individual band to a particular functional group, as it is customarily done for mid-IR spectra. In order to extract relevant information on FA composition from these NIR spectral fingerprints, multivariate statistical analysis is required. A common quantitative chemometrics tool used for data analysis is PLS regression. To develop PLS calibration models several issues have to be considered.

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Chapter 3 : AOAC Method for Trans Fats Detection by Gas Chromatography -

Official Methods for the Determination of trans Fat Magdi M. Mossoba, John K.G. Kramer, Pierluigi Delmonte, Martin P. Yurawecz, and Jeanne I. Rader This monograph describes the most common gas chromatographic and infrared spectroscopic official methods required for the determination of trans fatty acids for food labeling purposes.

Trans fats contain one or more double bonds in the trans configuration instead of the usual cis configuration. Vanaspati is rich in trans fats. The trans fats impart solidity to margarine. They are also responsibly imparting stability to emulsions in shortenings. It has been established that trans fatty acids can increase the risk of cardiovascular diseases, diabetes, obesity, premature births, and even cancer. As a result of these research findings, trans fats intake should be negligible or preferably zero. This is utilized for converting raw data into the actual spectrum that can be detected by infrared spectroscopy; hence the term FTIR. The underlying principle of FTIR is based on the fact that most molecules absorb light in the infrared region of the electromagnetic spectrum. This absorption corresponds specifically to the bonds present in the molecule. The FTIR technique can be used to obtain an infrared absorption spectrum or emission spectrum of a solid, liquid or gas. It is a sensitive technique for identifying organic substances, including Trans fats. In the case of trans fats, which are present in partially hydrogenated fats, their structure has multiple double-bonds, which can easily be detected and analyzed by FTIR. This data is essential for confirming the presence of trans fats in the food sample. How Can We Help? The instrumentation is operated by well-trained and competent professionals to carry out food testing with utmost precision. We will be happy to provide you with a proposal for testing the trans fats in your food samples. Official Methods of Analysis. Analysis of trans fat in edible oils with the cooking process. Trans fats—sources, health risks and an alternative approach—a review. J Food Sci Technol. Determination of the degree of degradation of frying rapeseed oil using Fourier-Transform Infrared Spectroscopy combined with partial least-squares regression. Int J Anal Chem.

Chapter 4 : Official Methods for Determination of trans Fat, Second Edition - CRC Press Book

This monograph describes the most common gas chromatographic and infrared spectroscopic official methods required for the determination of trans fatty acids for food labeling purposes.

Chapter 5 : Official Methods for the Determination of Trans Fat (2nd Edition) - Knovel

Official Methods for Determination of trans Fat, Second Edition / Edition 2 This monograph describes the most common gas chromatographic and infrared spectroscopic official methods required for the determination of trans fatty acids for food labeling purposes.

Chapter 6 : AOAC Method for Trans Fats Detection — FTIR -

Abstract. Adequate analytical tools are required for labelling food products for trans fatty acid (TFA) contents. The choice of the fatty acid (FA) derivatisation is a crucial step in obtaining good results, in particular with short-chain and conjugated FA.