Raw Material Identification
to Quality Assurance

Edible oil workflows

Compositional and trace contaminant analysis

- Near-Infrared Spectroscopy • Liquid Chromatography • Gas Chromatography
- Inductively Coupled Plasma-Optical Emission Spectroscopy

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Edible oil production

Edible oils are among the most abundant cooking ingredients in the world. They are extracted from plants (e.g., soybean, canola, and chili); seeds (e.g., sesame and sunflower); nuts (e.g., walnut and macadamia); and fruits (e.g., palm, olive, and coconut). Depending on oil type, they are used in baking and frying food and for noncooking products such as salad dressing, margarine spreads, and dips. In addition, edible oils are used to produce nonfood products such as cosmetics and as a feedstock for making biodiesel fuel.

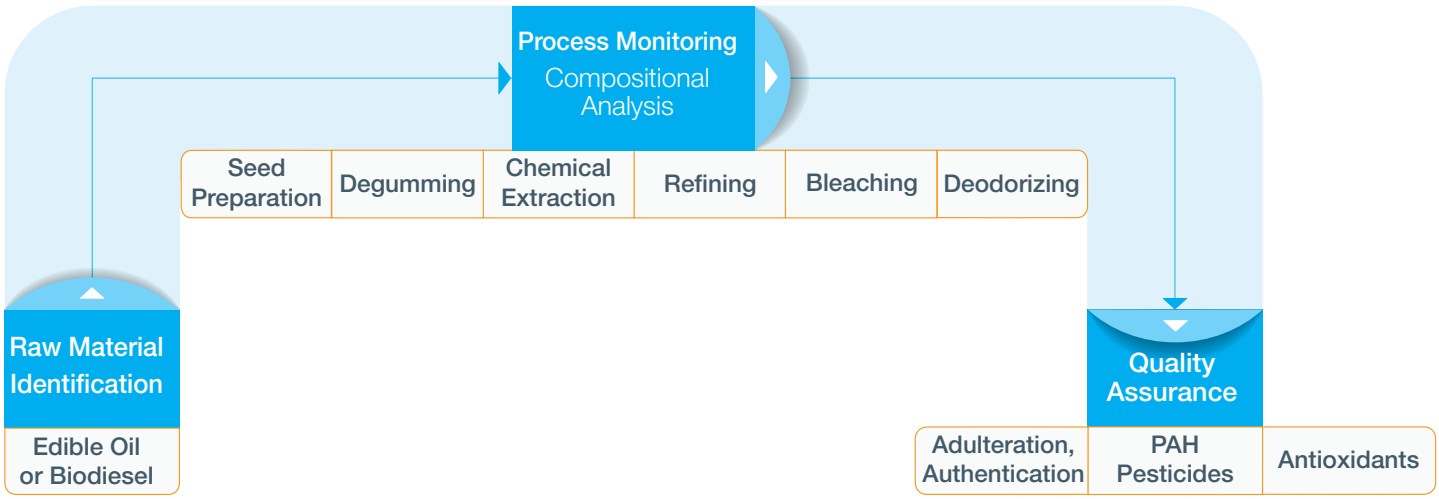
With demand for edible oils increasing due to a growing world population, improved living standards, and changing diets, it is essential to monitor the production process, understand the characteristics of the raw feedstock, and validate the specifications of the finished product to ensure a safe and regulatory-compliant food product.

A typical process for producing edible cooking oils without additives, preservatives, or flavoring includes the following steps:

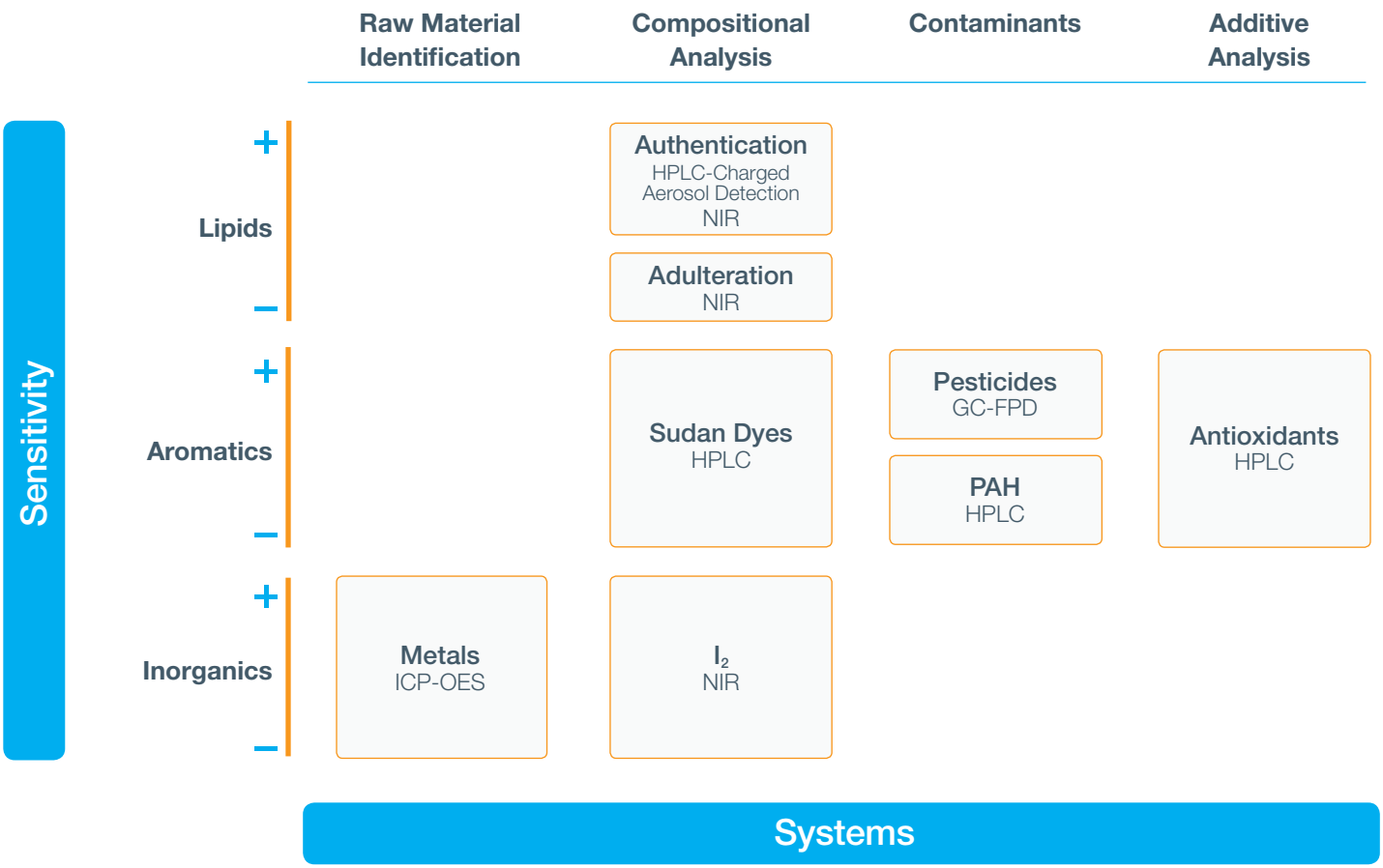
- **Seed preparation**, including cleaning, grinding, and pressing when the oil seeds are deskinned or dehulled, ground into course meal, and pressed to separate the oil.
- **Degumming** crude cooking oils (such as soybean), which have relatively high levels of phosphatides or gums that need to be removed to prevent a loss in refining efficiencies that result from their emulsifying properties and thermal instabilities.
- **Chemical extraction** using a hydrocarbon solvent to selectively dissolve and extract the cooking oil from the pressed mass, followed by combustion to dry the seeds.
- **Refining** to remove color, odor, and bitterness by treating the oil with sodium hydroxide to saponify and remove the fatty acid contaminants.
- **Bleaching** for oil used in cooking, which has an additional filtering step through activated carbon or clays to absorb any pigments. Oils that will not be heated (such as those used for salad dressing) are chilled to remove waxes and ensure that the oils will not partially solidify in the frigerator.
- **Deodorizing** by steam to remove any taste or odor components as a final step. Typically, trace amounts of citric acid are then added to inactivate any trace metals that might promote oxidation and shorten shelf life.

Thermo Fisher Scientific can provide product solutions for every step of your workflow process. If the solutions employ near-infrared (NIR) spectroscopy, inductively coupled plasma-optical emission spectroscopy (ICP-OES), liquid chromatography (LC), or gas chromatography (GC), Thermo Fisher Scientific can deliver critical information about your process—whether it involves raw material identification, compositional analysis, contaminant testing, or additive analysis—with a choice of products to best suit your specific application needs and requirements.

Analysis workflow by application



Analysis workflow by product



System solutions

Liquid Chromatography

The Thermo Scientific™ Dionex™ Corona™ Veo™ charged aerosol detector combined with a Thermo Scientific™ Dionex™ UltiMate™ 3000 LC system provides consistent analyte response independent of chemical structure for high-resolution characterization and quantitation of a variety of lipids from different lipid classes (Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System compatible).

Thermo Scientific™ Accucore™ HPLC Columns

Featuring solid core particles, Accucore columns provide fast, high-resolution separations plus a range of conventional and unique chemistries to meet all your needs.

Gas Chromatography

The Thermo Scientific™ TRACE™ 1300 series gas chromatograph and Thermo Scientific™ TSQ 8000 Triple Quadrupole GC-MS System encompass the latest breakthrough technology to substantially elevate performance in QA/QC and routine laboratories (Chromeleon Chromatography Data System compatible).

Thermo Scientific™ TraceGOLD GC Columns

Providing a leap forward in performance, TraceGOLD GC Columns deliver ultralow bleed, superior inertness, and a high level of reproducibility.

Inductively Coupled Plasma

The Thermo Scientific™ iCAP™ 7000 Series ICP-OES is ideal for direct oil analysis, providing high matrix tolerance and excellent sensitivity for raw material identification.

Near-Infrared Spectroscopy

The Thermo Scientific™ Antaris™ II FT-NIR Analyzer provides robust and reliable data collection for at-line, on-line, and in-line analysis. Analyze raw feedstock by reflection using the internal integrated sphere, liquids with the internal temperature-controlled transmission module, or process monitoring with fiber optics probes—all in one turnkey system.

Raw material identification

The elemental analysis of oils such as canola and palm is important to determine their potential use as either foodstuffs or fuel. The maximum concentrations of elemental contaminats allowed in oils used as foodstuff are much lower than those used as biodiesel. Elevated concentrations can affect flavor, color, and the shelf life of the oil. For edible oils, the maximum concentrations for calcium and magnesium are 0.05 ppm each according to American Oil Chemists' Society (AOCS) Official Method Ca 17-01, Determination of Trace Elements (Calcium, Copper, Iron, Magnesium, Nickel, Silicon, Sodium, Lead, and Cadmium) in Oil by Inductively Coupled Plasma Optical Emission Spectroscopy. For automotive fuels, the maximum concentration is 5 ppm for calcium and magnesium combined according to EN 14214, which provides requirements and test methods for fatty acid methyl esters (FAME) for diesel engines.

	Crude	Refined	Bleached	Deodorized	Detection Limit
Ca 317.9 nm	162.1	1.6	0.22	0.05	0.0019
Cu 324.7 nm	0.036	<DL	<DL	<DL	0.0011
Fe 259.9 nm	1.17	<DL	<DL	<DL	0.00092
Mg 280.2 nm	61.57	0.611	0.07	0.006	0.00025
Na 589.5 nm	0.122	1.145	0.28	<DL	0.011
Ni 231.6 nm	<DL	<DL	<DL	<DL	0.0013
P 177.4 nm	282.109	3.018	1.213	0.579	0.0072
Pb 220.3 nm	<DL	<DL	<DL	<DL	0.010
S 180.7 nm	7.031	2.93	1.484	3.495	0.058
Sn 189.9 nm	0.112	0.075	0.089	0.01	0.0037

Table 1. Determination of the elemental content of canola oil by ICP-OES at four stages of the refining process. As expected, the concentration of the elements decreases with a higher degree of processing. The concentrations for calcium and magnesium are below the AOAC Official Method Ca 17-01 allowable threshold in the deodorized canola, making it a good candidate for producing either biodiesel or foodstuffs.

Compositional analysis

Determining the composition of cooking oil provides a range of valuable information, including authenticating its origin and quality by its unique acylglyceride fingerprint, elucidating its nutritional value by its fatty acid composition, detecting adulteration with a lower quality oil, and providing insight into its thermal stability and resistance to oxidation by its iodine value.

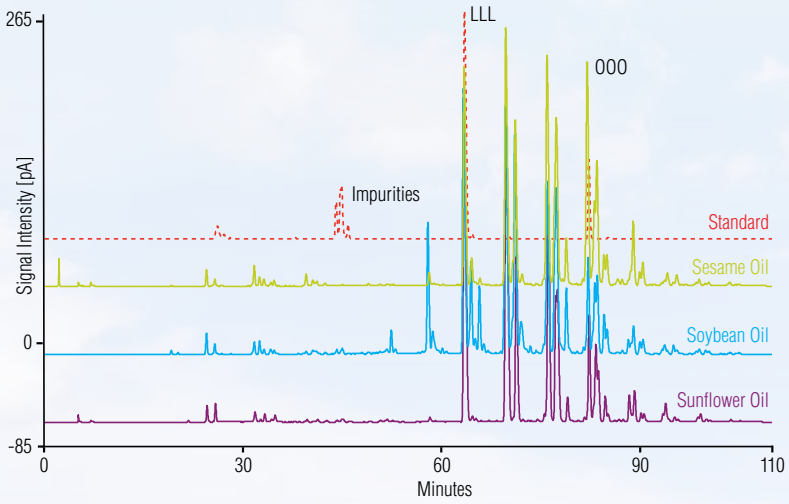


Figure 1. Triglyceride profile showing a clear separation of three different edible oils—sesame, soybean, and sunflower—by reversed-phase HPLC with charged aerosol detection. The overlay of peaks for glycerol trilinoleate (LLL) and glycerol trioleate (000) demonstrate the reproducibility of the method.

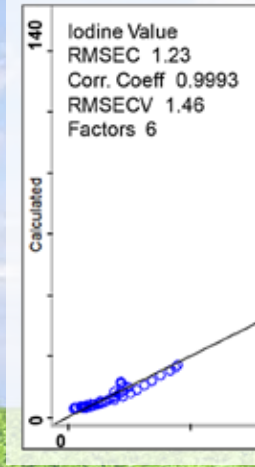


Figure 2. This 3D principal component scores plot demonstrates the capability of NIR to discriminate and identify common edible oils (olive, soybean, canola, sunflower, peanut, and corn) in seconds.

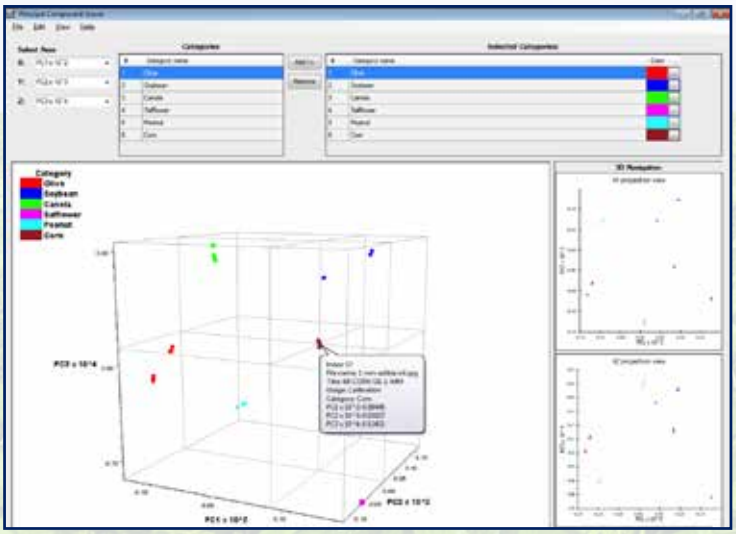
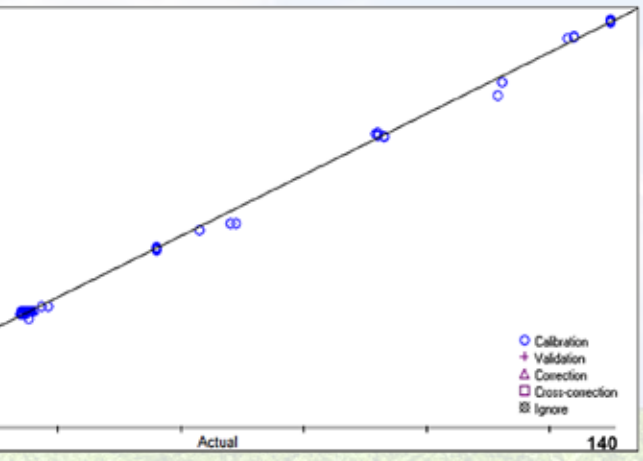
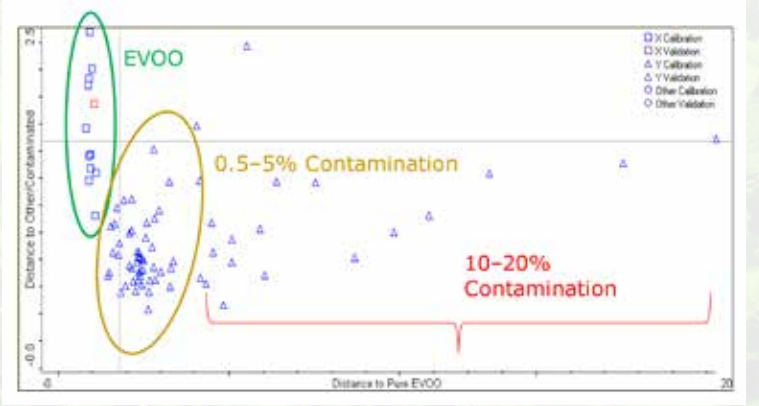


Figure 4. This discriminant analysis distance plot shows how NIR can be used to detect adulteration by discriminating between extrapure virgin olive oil (EVOO) and olive oil contaminated with a number of lower value oils in less than two minutes, and at adulterant levels as low as 0.5%.

Figure 3. Iodine value calibration plot illustrates the capability of NIR to quickly determine the iodine value (unsaturation) for a number of edible oils over a wide range of iodine values.





Sudan dyes are a class of synthetic dyes that are mainly used for industrial applications, such as the coloring of plastics. These dyes are banned as a food-coloring agent because they are classified as carcinogens. For economic reasons, however, they are sometimes illegally used to color food to improve its appearance. Therefore, methods are needed to determine if oil products have been adulterated with these dyes.

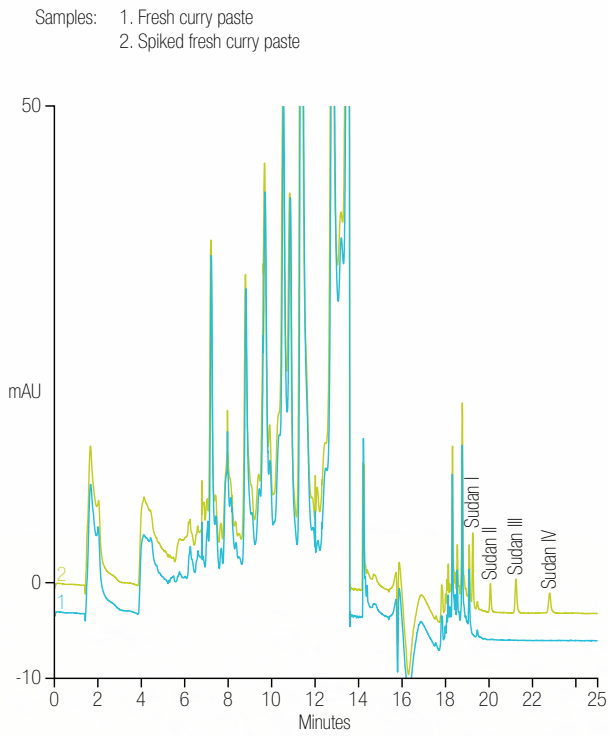
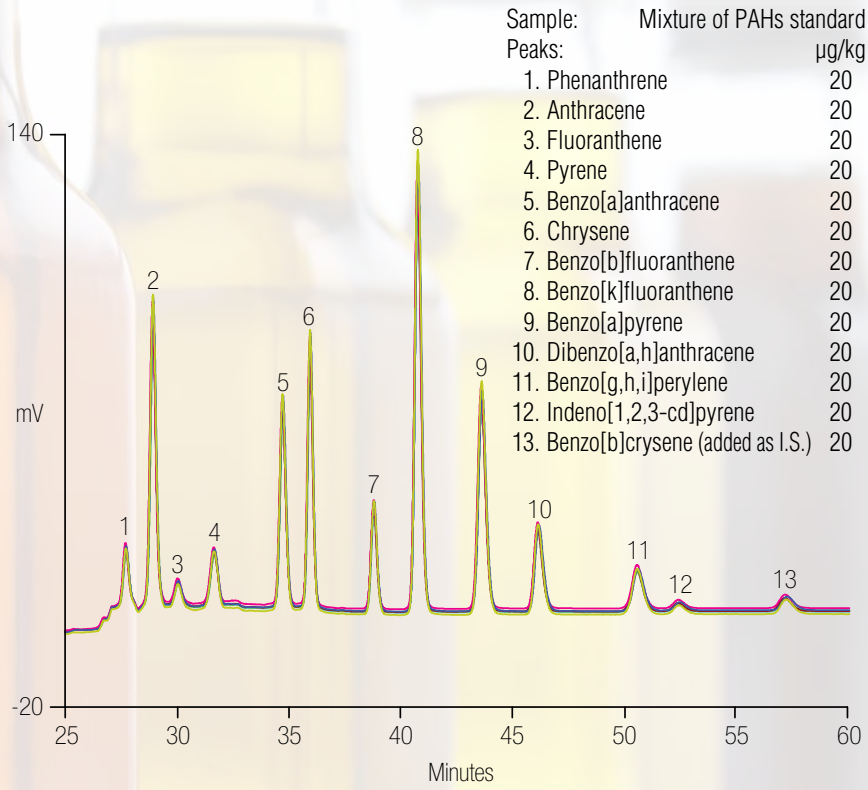


Figure 5. Determination of Sudan dyes I-IV in chili oil by overlay chromatograms of fresh curry paste and spiked fresh curry paste samples (5 mg/mL Sudan I and II and 10 mg/L Sudan III and IV) using reversed-phase, two-dimensional HPLC-UV without off-line sample preparation. A Thermo Scientific™ Acclaim™ PolarAdvantage II Column is used in the first dimension and an Acclaim Phenyl-1 Column in the second dimension.

Contaminant analysis

Analyzing crude oil is of vital interest to manufacturers and consumers of edible oils in the commodity market. Concern over carcinogenic polycyclic aromatic hydrocarbons (PAHs) formed during incomplete combustion of the seed material is a health issue. And as a result of the widespread use of pesticides, residues can also be introduced into the oil. In addition to the presence of contaminants, the functionality of the oil is also of concern. Determining the free fatty acid (FFA) content of the oils demonstrates that the oil has been properly refined and serves as a quality check for oils traded on the commodities market.

Figure 6. Determination of PAHs, shown as an overlay of chromatograms of seven serial injections of an olive oil sample spiked with a PAH standard mixture (20 ug/kg) using donor-acceptor complex chromatography (DACC) HPLC, an automated process for on-line sample preparation and analysis.



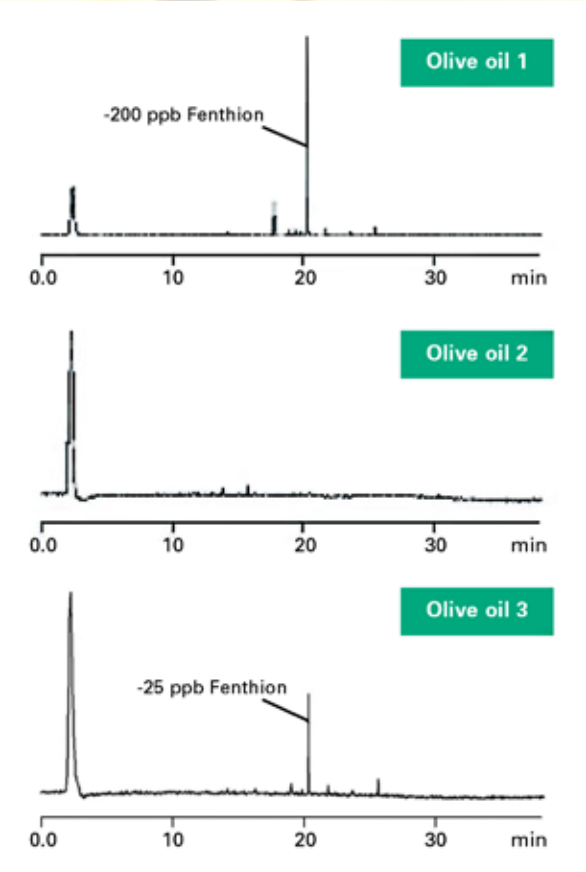
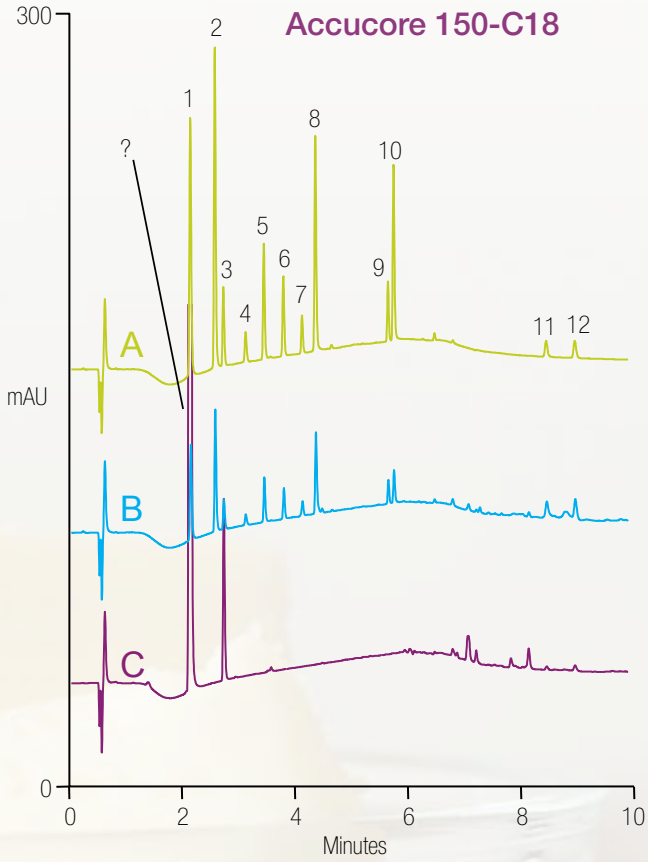


Figure 7. Determination of organo-phosphorous pesticides in olive oil by GC. The chromatograms show the detection of fenthion (an insecticide) in three commercial oils using GC with a programmable temperature vaporizing (PTV) injector and phosphorus-selective flame photometric detector (FPD).

Additive analysis

The shelf life of edible oils and their applicability in industrial situations is greatly dependent on their resistance to oxidation that can lead to rancidity. While the amount of saturated fats present in the oil plays a significant role in providing oxidative stability, the addition of antioxidants can further enhance it.

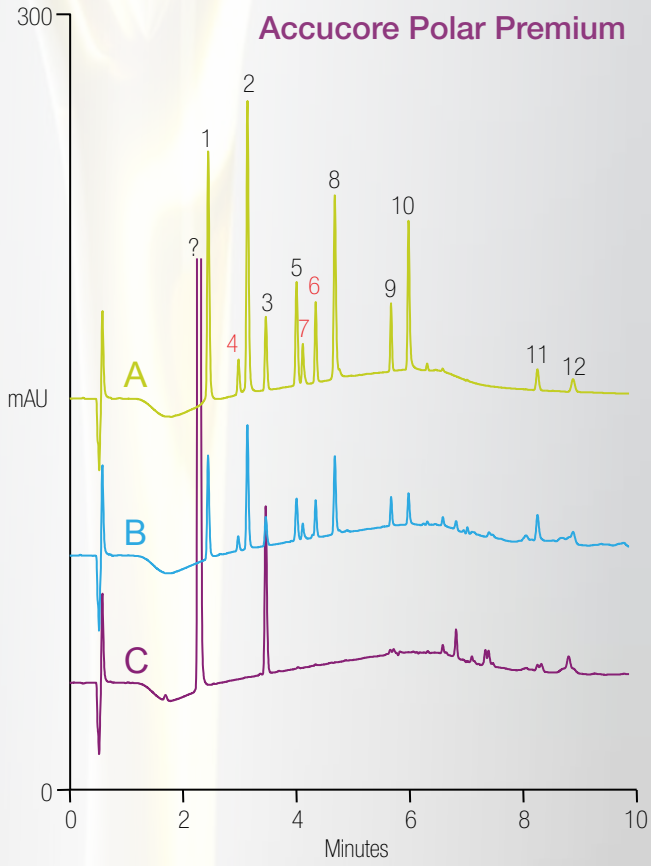
Figure 8. Phenolic antioxidants are commonly used as preservatives for edible oils and fats to prevent rancidity. AOAC Official Method 983.15 on Phenolic Antioxidants in Oils, Fats, and Butter Oil was developed to assay levels in finished food products. Here, margarine preserved with *tert*-Butylhydroquinone (TBHQ) is compared to a peanut oil extract fortified with 12 antioxidants. Results obtained using an Accucore 150-C18 column revealed a peak eluting at the same time as propyl gallate, although not listed as an ingredient (left chromatogram). Analysis with a second column type, an Accucore Polar Premium (right chromatogram), shows the unidentified peak elutes earlier than proply gallate and that the TBHQ in the margarine overlays with the TBHQ in the peanut oil. The results suggest the peak is an unidentified constituent. Note the different selectivities of the Accucore 150-C18 and Accucore Polar Premium, a polar-embedded C18 phase, resulting in a change of the elution order for Peaks 4, 6, and 7.



Samples:
A. 12 antioxidants, 50 ppm each in acetonitrile
B. Peanut oil extract fortified with 12 antioxidants
C. Margarine preserved with TBHQ

Peaks:

1. Propyl Gallate
2. THBP
3. TBHQ
4. Ethoxyquin
5. NDGA
6. BHA
7. Ionox-100
8. Octyl Gallate
9. BHT
10. Lauryl Gallate
11. Vitamin E
12. Vitamin E Acetate



Applications for Edible Oils

Analyte	Application
Alkaline & Earth Alkali Metals	AN 40876: Elemental Analysis of Canola Oil Using the Thermo Scientific iCAP 6500 ICP
Pesticides	AN 10049: Trace Determination of Organo-Phosphorous Pesticides in Olive Oil by GC Analysis through PTV Backflush/FPD
Pesticides	AN 52102: Analysis of Pesticides in Citrus Oil Using PTV Backflush with GC-MS/MS Triple Quadrupole for High Sample Throughput
Polycyclic Aromatic Hydrocarbons	AN 196: Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Edible Oils by Donor-Acceptor Complex Chromatography (DACC)-HPLC with Fluorescence Detection
Sudan Dyes	AN 1023: Determination of Sudan Dyes I–IV in Curry Paste
Triacylglycerides	LPN 2881-01: Towards Standard-Free Quantitative and Qualitative Analysis in Liquid Chromatography

Australia +61 3 9757 4486
Austria +43 1 333 50 34 0
Belgium +32 53 73 42 41
Brazil +55 11 3731 5140
China +852 2428 3282
Denmark +45 70 23 62 60
France +33 1 60 92 48 00
Germany +49 6126 991 0
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