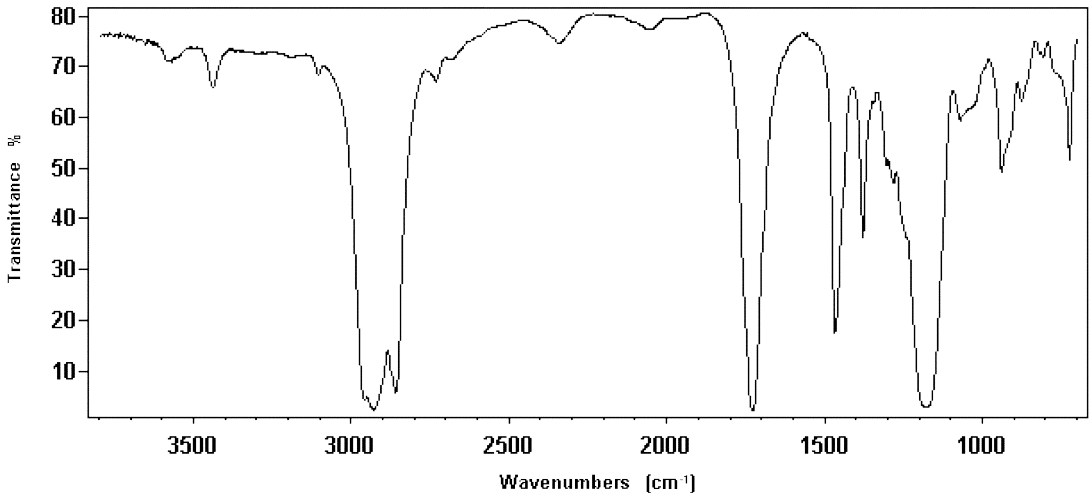
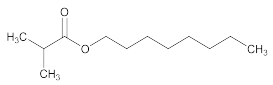


Octyl Formate

Octyl Isobutyrate

First Published: Prior to FCC 6

Octyl 2-Methylpropanoate



$C_{12}H_{24}O_2$ Formula wt 200.32
FEMA: 2808
UNII: T5819VWJ3T [octyl isobutyrate]

DESCRIPTION

Octyl Isobutyrate occurs as a colorless to pale yellow liquid.
Odor: Refreshing, herbaceous
Boiling Point: ~245°
Solubility in Alcohol, Appendix VI**:** One mL dissolves in 1 mL of 95% alcohol.
Function: Flavoring agent

ASSAY

- **PROCEDURE:** Proceed as directed under *M-1a,* Appendix XI.
  Acceptance criteria: NLT 98.0% of $C_{12}H_{24}O_2$

SPECIFIC TESTS

- **ACID VALUE, FLAVOR CHEMICALS (OTHER THAN ESSENTIAL OILS),** *M-15,* Appendix XI
  Acceptance criteria: NMT 1.0
- **REFRACTIVE INDEX,** Appendix II**:** At 20°
  Acceptance criteria: Between 1.420 and 1.425
- **SPECIFIC GRAVITY:** Determine at 25° by any reliable method (see *General Provisions*).
  Acceptance criteria: Between 0.853 and 0.858

Add the following:

▲Fully Hydrogenated Oils and Fats

First Published: FCC 10

Completely Hydrogenated Oils and Fats
Hydrogenated Oils and Fats

DESCRIPTION

Fully Hydrogenated Oils and Fats occur as solids at room temperature. They are produced through the hydrogenation of individual food grade oils and fats, or through the hydrogenation of mixtures of food grade oils and fats to achieve an iodine value of NMT 4, representing the saturation of double bonds from the source oils and/or fats. Oils and fats used to manufacture Fully Hydrogenated Oils and Fats are expressed or extracted from a range of seeds, nuts, fruits, and animal fatty tissues, including canola (low erucic acid rapeseed), coconut, corn, cottonseed, lard, palm, palm kernel, peanut, safflower, soybean, sunflower, and tallow.[1] The fatty acid chain length distribution for these oils and fats varies widely, however, they are typically classified as either lauric (coconut and palm kernel, both of which are characterized as having NLT 35% of fatty acids with a carbon chain length of 12) or non-lauric (all other oils and fats characterized by having NMT 2% of fatty acids with a carbon chain length of 12). The resultant mixture of saturated triglycerides reflects the fatty acid chain length distribution of the precursor oils/fats with the unsaturated native fatty acids largely converted to saturated fatty acids (these are generally fatty acids with an 18-carbon chain length). Fully Hydrogenated Oils and Fats are insoluble in water.

[1] This standard is intended to represent all of the listed animal fats and vegetable oils as source oils, including all varieties and fractions of these fats and oils. Marine sourced oils and high erucic acid rapeseed oils are not represented by the standard.

Function: Ingredient used in the manufacture of a variety of foods and food ingredients; or as a component for the production of a wide array of shortening products, baking fats, frying fats, confectionary fats, margarines, spreads, etc.

Packaging and Storage: Store in tight containers at ambient temperature. [NOTE—Bulk, molten Fully Hydrogenated Oils and Fats may also be stored in properly covered containers at 10°–15° above the melting point.]

IDENTIFICATION

• **A. DROPPING POINT**

[NOTE—This method is based on AOCS Standard Procedure Cc 18-80 (reapproved 2009).[2] The dropping point is defined as the temperature at which a test sample will become fluid to flow under the conditions of the test below.]

Apparatus: Use the Mettler DP90[3] dropping point system with accessory sample cups and sample cup holder intended for use with this system (drip hole diameter 2.8 mm), or an equivalent dropping point system.

Standard: Lauric acid doped with 0.5% powdered carbon[4]

Standard performance: Transfer a portion of the *Standard* to a small beaker, and heat on a warm hot plate (60°–70°) until the lauric acid melts. Stir the melted *Standard* well to ensure that the carbon is in suspension. Transfer the melted *Standard* to a pre-conditioned sample cup, filling the cup entirely. (A pre-conditioned sample cup is one that has been thoroughly cleaned, dried, and held in a freezer on a clean, smooth surface for NLT 10 min immediately prior to use.) Place the melted *Standard* in the sample cup in a freezer held at NMT –5° for exactly 15 min. Preheat the dropping point furnace to 39°. Immediately after 15 min, remove the sample cup from the freezer and place it into the furnace for exactly 1 min, then initiate the heating program of the dropping point system to completion of the analysis. (The system should be set at a heating rate of 1.0°/min.) The dropping point of the *Standard* should be 44.90°–47.30°. [NOTE—The time for which the sample cup is held in the furnace prior to initiation of the heating program may vary based on the individual instrument used. The instructions included are based on the use of the Mettler DP90 system, but adjustments will need to be made when using a system that has a built-in hold time.]

Sample preparation: Melt a sample in a small beaker on a warm hot plate and mix thoroughly. Using a pre-conditioned sample cup, as described in the *Standard performance*, transfer a sufficient amount of the melted sample to the sample cup to fill the cup entirely. Place the melted sample in the sample cup in a freezer held at NMT –5° for exactly 15 min.

Analysis: Preheat the dropping point furnace to a temperature of NLT 5° below the expected dropping point of the *Sample preparation*. Insert the sample preparation cup containing the cooled sample into the sample cup holder, and remove from the freezer, avoiding repeated handling of the cooled sample cup. Immediately place the sample holder into the furnace, ensuring that the holder is properly seated, and initiate the heating of the dropping point system to completion of the analysis. (The system should be set at a heating rate of 1.0°/min.) Record the dropping point temperature from the display of the dropping point control unit.

Acceptance criteria

Lauric type: 30°–55°

Non-lauric type: 50°–75°

• **B. FATTY ACID COMPOSITION**

[NOTE—This method is based on AOCS Official Methods Ce 2-66 and Ce 1h-05.[2]]

Apparatus: Use a gas chromatograph (see *Chromatography*, Appendix IIA) suitable for use with capillary columns, a temperature-controlled split/splitless injector operated in split mode, and a flame-ionization detector (FID). The capillary column should be of fused silica, 100-m × 0.25-mm with a 0.20-µm coating of a 100% cyanopropyl-silicone stationary phase.[5]

Operating conditions: The carrier gas should be gas chromatography-grade hydrogen or helium (99.99% or better purity) that has been dried, and from which the oxygen has been removed using suitable filters. Do not use nitrogen as a carrier gas for this method. The flame gases should be gas chromatography-grade hydrogen and air, and the make-up gas should be gas chromatography-grade nitrogen or helium. Use a 78.5-mm × 4-mm (i.d.) × 6.3-mm (o.d.) injection port split liner with glass wool. The injection port and the detector should be operated at 250°. The column (oven) temperature should be held at 180°. When hydrogen carrier gas is used, the column head pressure is 170 kPa (25 psi) with a constant flow rate of 1.0 mL/min, a linear velocity of 26 cm/s, and a split ratio of 100:1. When helium is used as the carrier gas, the column head pressure is 286 kPa (41 psi) with a constant flow rate of 1.0 mL/min, a linear velocity of 19 cm/s, and a split ratio of 100:1.

Reagents and solutions[6]

Internal standard solution: 5.0 mg/mL of triheneicosanoin (C21:0 triacylglycerol, TAG) in chloroform. Use triheneicosanoin with a purity of NLT 99%.

Standard solution: Use a suitable mixture of fatty acid methyl esters covering the range of fatty acids expected in the sample material. [NOTE—Use USP FAME Standard Mixture RS or a mixture of methyl esters of pure fatty acids, in particular *cis*- and *trans*-

[2] Full text of the method is available from the American Oil Chemists' Society (AOCS) at www.aocs.org.

[3] Available from http://us.mt.com. An equivalent system with a furnace and control unit may be used.

[4] Use lauric acid catalog number N-12-A available from NuCheck Prep, Inc. (http://www.nu-chekprep.com) and powdered carbon catalog number SN-20 available from Westvaco Nuchar (http://mwv.com), or equivalent products.

[5] Supelco SP-2560 (available at http://www.supelco.com), Agilent CP-Sil88 (available at http://www.agilent.com), or equivalent.

[6] Pure fatty acids, methyl esters of pure fatty acids, and mixtures of fatty acid methyl esters for use in the *Internal standard solution* and the *Standard solution* can be obtained commercially from Nu-Chek Prep, Elysian, MN, or an equivalent supplier.

isomers of octadecenoic (oleic), *trans*-isomers of octadecadienoic (linoleic) and octadecatrienoic (α-linolenic) acids, with a reference chromatogram.]

Sample preparation: Transfer a sufficient amount of the *Internal standard solution* into a 50-mL or 125-mL reaction flask so that the concentration in the final solution, after the sample is added, is 0.05–0.10 mg of internal standard per 1 mg of sample. Evaporate the chloroform (from the *Internal standard solution*) from the flask, then introduce 100–1000 mg of the sample to the reaction flask. Add 4–10 mL of 0.5 N methanolic sodium hydroxide, and add a boiling chip. Attach a condenser, and heat the mixture on a steam bath until the fat globules go into solution. This step should take 5–10 min. Add 5–12 mL of 12.5% boron fluoride–methanol reagent (this reagent contains 125 g of boron fluoride per liter of methanol and is available commercially) through the condenser, and boil for 2 min. Add 2–5 mL of heptane through the condenser, and boil for 1 min longer. Remove from heat, remove the condenser, and add about 15 mL of saturated sodium chloride solution. Stopper the flask, and shake vigorously for 15 s. Dilute the fatty acid methyl esters (FAME) so obtained in *n*-heptane or *n*-hexanes to a concentration of approximately 15–20 mg/mL of FAME in solvent.

System suitability: Using an injection size of 1 µL, check the performance of the column using the *Standard solution*. Adjust the test portion size, test portion concentration, or oven temperature (in 1° increments) if necessary to produce a chromatogram with optimal separation that matches the example chromatogram provided with the mixture of fatty acid methyl esters used in the *Standard solution*.

Theoretical correction factors: Theoretical correction factors (TCF) represent the theoretical flame ionization detector response factor for fatty acids (as methyl esters) with respect to the C21:0 FAME internal standard. The TCF should be applied to the analytical data for optimum accuracy and to minimize variation between laboratories because of differences in calculating response factors. Using a microsyringe suitable for gas chromatography (10 µL capacity), inject 1 µL of the *Standard solution* into the chromatograph, and record the resulting chromatogram.

Calculate the TCF for each fatty acid:

$$TCF_X = MW_X/(N_X - 1) \times (AWC \times TCF_{IS})$$

TCF_X = TCF for the methyl ester of fatty acid X with respect to the internal standard
MW_X = molecular weight of the methyl ester of fatty acid X
N_X = number of carbon atoms in the methyl ester of fatty acid X
AWC = atomic weight of carbon, 12.011
TCF_{IS} = TCF for the methyl ester of the C21:0 internal standard, 1.3503

Procedure: Using a microsyringe suitable for gas chromatography (10 µL capacity), inject 1 µL of the *Sample preparation* into the chromatograph, and record the resulting chromatogram.

Calculate the amount, in grams, of each of the individual fatty acids for which a peak appears in the chromatogram, expressed as the FAME (W_{FAMEx}) equivalents:

$$W_{FAMEx} = (A_X \times W_{TAG\text{-}IS} \times F \times TCF_X)/A_{IS}$$

W_{FAMEx} = weight of fatty acid X in the *Sample preparation*, expressed as the fatty acid methyl ester (g)
A_X = peak area count for fatty acid X from the chromatogram of the *Sample preparation*
$W_{TAG\text{-}IS}$ = weight of the internal standard in the *Sample preparation* (g)
F = factor converting the weight of the C21:0 internal standard from triacylglycerol form to the weight of its corresponding fatty acid methyl ester form (1.0040)
TCF_X = TCF for the methyl ester of fatty acid X with respect to the internal standard
A_{IS} = peak area count for the internal standard from the chromatogram of the *Sample preparation*

Calculate the weight, in grams, of each of the individual fatty acids for which a peak appears in the chromatogram:

$$W_X = W_{FAMEx} \times F_{FAx}$$

W_X = weight of fatty acid X in the *Sample preparation* (g)
W_{FAMEx} = weight of fatty acid X in the *Sample preparation*, expressed as the fatty acid methyl ester (g)
F_{FAx} = factor converting the fatty acid methyl ester of X to the corresponding fatty acid X (from *Table 1*)

Table 1. Factors for Conversion of FAME to Fatty Acid Equivalents

Fatty Acid (Shorthand Notation)	F_{FAx}
4:0	0.8627
6:0	0.8923
8:0	0.9114
10:0	0.9247
11:0	0.9300
12:0	0.9346
13:0	0.9386
14:0	0.9421
15:0	0.9453
16:0	0.9481
17:0	0.9507
18:0	0.9530
18:1	0.9527
18:2	0.9524
20:0	0.9570
21:0	0.9588
22:0	0.9604
23:0	0.9620
24:0	0.9633

Calculate the amount of each individual fatty acid present in the chromatogram of the *Sample preparation* as the percentage of the total fatty acids present in the *Sample preparation*:

$$P_X = (W_X/\Sigma W_X) \times 100$$

P_X = percentage of fatty acid X present in the *Sample preparation* (as the percent of total fatty acids)
W_X = weight of fatty acid X in the *Sample preparation* (g)
ΣW_X = sum of the weights of every fatty acid present in the chromatogram of the *Sample preparation* (g)

Report the amount of each fatty acid listed in the *Acceptance criteria* as the percentage of the fatty acid composition (g of individual fatty acid per 100 g of total fatty acids present in the sample taken).

Acceptance criteria: Fully Hydrogenated Oils and Fats exhibit the following composition profile of fatty acids with the classification of Lauric Type and Non-Lauric Type based on the source oils/fats, as described in the *Description*:

Table 2. Fatty Acid Composition

Fatty Acid (Shorthand Notation)	Lauric Type (%)	Non-Lauric Type (%)
6:0	<1.5	—
8:0	<9	—
10:0	<9	—
12:0	>35	<2
14:0	10–25	<2
16:0	<12	>2
18:0	7–30	>15
18:1	<4	<4
18:2	<1	<1
20:0	<1	<4
22:0	—	<4
24:0	—	<3

- **C. Iodine Value**

Analysis: Using the chromatogram obtained in *Identification B. Fatty Acid Composition*, calculate the iodine value as:

$$\text{Result} = \Sigma(P_x \times I_x)$$

P_x = percentage of fatty acid X present in the *Sample preparation* (as the percent of total fatty acids)
I_x = iodine conversion factor for the methyl ester of unsaturated fatty acid X (triglyceride form, see *Table 3*)

Table 3. Iodine Value Conversion Factors[2]

Unsaturated Fatty Acid (Shorthand Notation)	MW, Triglyceride	Moles of Iodine	MW, Iodine	Factor (Triglyceride)
12:1	633.01	6	761.4270	1.2029
14:1	717.18	6	761.4270	1.0617
15:1 *trans*	759.16	6	761.4270	1.0030
15:1	759.16	6	761.4270	1.0030
16:1 *trans*	801.34	6	761.4270	0.9502
16:1	801.34	6	761.4270	0.9502
17:1 *trans*	843.42	6	7614270	0.9028
17:1	843.42	6	761.4270	0.9028
18:1 *trans*	885.50	6	761.4270	0.8599
18:1	885.50	6	761.4270	0.8599
18:2 *trans*	879.50	12	1522.8540	1.7315
18:2	879.50	12	1522.8540	1.7315
18:2 CLA	879.50	12	1522.8540	1.7315
18:3 *trans*	873.50	18	2284.2810	2.6151
18:3	873.50	18	2284.2810	2.6151
20:1	969.66	6	761.4270	0.7853

[NOTE—The iodine value is a message of unsaturation and represents the number of grams of iodine absorbed, under prescribed conditions, by 100 g of the test substance.]

Acceptance criteria: NMT 4

IMPURITIES

Inorganic Impurities

- **Lead,** *Lead Limit Test, Atomic Absorption Spectrophotometric Graphite Furnace Method, Method II***,** Appendix IIIB
  Acceptance criteria: NMT 0.1 mg/kg
- **Nickel,** *Elemental Impurities by ICP, Method I***,** Appendix IIIC
  [NOTE—The method referenced in Appendix IIIC is modified, as indicated in the following sections, based on AOCS Official Method Ca 17-01 (reapproved 2009).[2]]
  Sample preparation: Melt a sample at 10° above the melting point. The relative maximum temperature for analysis of this sample (and all hardened fats) is 60°.
  Sample solution: Transfer 2.5 ± 0.02 g of the *Sample preparation* to an autosampler tube, and dilute the sample with 2.5 g of 1-butanol. Cap the tube and invert it 40–50 times on a mixing table. Keep the *Sample solution* warm to ensure that it stays in solution during analysis.
  Blank oil: A refined and bleached soybean oil or other oil (which is known to be free of trace elements). [NOTE—Use Base 20 Oil or Base 75 Oil available from Accu-Standard, New Haven, CT, or an equivalent absolute reference blank oil to check the level of trace elements in the *Blank oil*.]

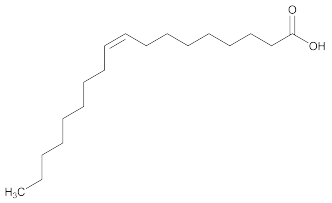
Blank: Dilute one part of the *Blank oil* with one part of 1-butanol, by volume.
Calibration solution 1: Dilute a portion of a commercially available nickel standard,[7] equivalent to 300 µg of elemental nickel, to 50.0 g with the *Blank oil*. Further dilute this mixture with 50.0 g of 1-butanol to achieve a solution containing 3.0 µg/g of nickel.
Calibration solution 2: Dilute a portion of a commercially available nickel standard, equivalent to 15.0 µg of elemental nickel, to 50.0 g with *Blank oil*. Further dilute this mixture with 50.0 g of 1-butanol to achieve a solution containing 0.15 µg/g of nickel.
Check standard solution: Dilute a portion of commercially available nickel standard, equivalent to 100 µg of elemental nickel, to 50.0 g with *Blank oil*. Further dilute this mixture with 50.0 g of 1-butanol.
System suitability: Proceed as directed.
Analysis: Analyze according to the instrument manufacturer's instructions at the major nickel emission wavelength of 231.6 nm. Run the *Sample solution*, *Calibration solution 1*, *Calibration solution 2*, and the *Blank* in triplicate and average the results for each sample.
Calculate and report the results on the basis of the dilution factor used in preparation of the *Sample solution*.
Acceptance criteria: NMT 1.5 mg/kg

OTHER REQUIREMENTS
- **LABELING:** Label as "hydrogenated","completely hydrogenated", or "fully hydrogenated" immediately followed by the name(s) of the source oil(s)/fat(s).▲ *FCC10*

Oleic Acid

First Published: Prior to FCC *6*
Last Revision: First Supplement, FCC 9

(Z)-9-Octadecenoic Acid



$C_{18}H_{34}O_2$ **Formula wt:** 282.47
CAS: [112-80-1]

UNII: 2UMI9U37CP [oleic acid]

DESCRIPTION
Oleic Acid occurs as a colorless to pale yellow, oily liquid when freshly prepared, but upon exposure to air it gradually absorbs oxygen and darkens. It is an unsaturated acid obtained from fats. When strongly heated in air, it decomposes and produces acrid vapors. Its specific gravity is about 0.895. It is practically insoluble in water, but is miscible with alcohol, with ether, and with fixed and volatile oils.
Function: Component in the manufacture of other food-grade additives; defoaming agent; lubricant; binder
Packaging and Storage: Store in tight containers.

IDENTIFICATION
- **FATTY ACID COMPOSITION,** Appendix VII
  Sample preparation: Transfer 100–1000 mg of Oleic Acid to a 50-mL or 125-mL reaction flask. Proceed as directed beginning with "Add 5–12 mL of 12.5% boron trifluoride-methanol reagent".
  Acceptance criteria: A sample exhibits 95%–105% of the amount of each individual fatty acid reported by the manufacturer.

ASSAY
- **OLEIC ACID**
  Acceptance criteria: A sample contains 95%–105% of the amount of oleic acid reported by the manufacturer when tested according to the *Identification* procedure.

IMPURITIES
Inorganic Impurities
- **LEAD,** *Lead Limit Test, Atomic Absorption Spectrophotometric Graphite Furnace Method, Method II,* Appendix IIIB
  Acceptance criteria: NMT 0.1 mg/kg

SPECIFIC TESTS
- **ACID VALUE (FATS AND RELATED SUBSTANCES),** *Method I,* Appendix VII
  Acceptance criteria: 196–204
- **IODINE VALUE,** Appendix VII
  Acceptance criteria: 83–103
- **RESIDUE ON IGNITION (SULFATED ASH),** Appendix IIC
  Sample: 10 g
  Acceptance criteria: NMT 0.01%
- **SAPONIFICATION VALUE,** Appendix VII
  Sample: 3 g
  Acceptance criteria: 196–206
- **TITER (SOLIDIFICATION POINT),** *Solidification Point,* Appendix IIB
  Acceptance criteria: Not above 10°
- **UNSAPONIFIABLE MATTER,** Appendix VII
  Acceptance criteria: NMT 2.0%
- **WATER,** *Water Determination,* Appendix IIB
  Acceptance criteria: NMT 0.4%

OTHER REQUIREMENTS
- **LABELING:** Label to indicate the amounts and types of fatty acids present.

[7] Elemental standards are available from SPEX-Certiprep, Metuchen, NJ, or an equivalent supplier. A suitable elemental standard should be present in solution as organic material.