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Superglycerinated Fully Hydrogenated Rapeseed Oil

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DEFINITION

Superglycerinated Fully Hydrogenated Rapeseed Oil is the product obtained by refining, hydrogenating, and glycerinating oil obtained from the seeds of *Brassica napus* and *Brassica campestris* (Fam. Cruciferae). The product is a mixture of mono-, di-, and triglycerides, with triglycerides as a minor component.

[Note—Use compendial grade glycerin as a starting material.]

IDENTIFICATION

• **A.** It meets the requirements of the test for *Fats and Fixed Oils, Fatty Acid Composition⟨401⟩*.

COMPOSITION

• **Content of 1-Monoglycerides**

Periodic acid solution: Dissolve 5.4 g of periodic acid in 100 mL of water, add 1900 mL of glacial acetic acid, and mix. Preserve in a light-resistant, glass-stoppered bottle.

Chloroform: Use chloroform that meets the following test. Add 50.0 mL of *Periodic acid solution* to each of three 500-mL flasks. Add 50 mL of chloroform and 10 mL of water to two of the flasks, and add 50 mL of water to the third flask. Add 20 mL of potassium iodide TS to each flask, mix gently, and continue as directed in the *Analysis*, beginning with "and allow to stand at least 1 min, but no longer than 5 min, before titrating". The difference between the volume of 0.1 N sodium thiosulfate VS required in the titrations with and without the chloroform is not greater than 0.5 mL.

Sample solution: Melt Superglycerinated Fully Hydrogenated Rapeseed Oil at a temperature not higher than 10° above its melting point, and mix thoroughly. Transfer an accurately weighed quantity of it, equivalent to about 150 mg of 1-monoglycerides, to a 100-mL beaker, dissolve in 25 mL of *Chloroform*, and mix.

Analysis: Transfer the *Sample solution*, with the aid of an additional 25 mL of *Chloroform*, to a separator. Wash the beaker with 25 mL of water, and add the washing to the separator. Close the separator tightly with a stopper, shake vigorously for 30–60 s, and allow the layers to separate.

[Note—Add 1–2 mL of glacial acetic acid to break emulsions due to the presence of soap.]

Collect the aqueous layer in a 500-mL glass-stoppered Erlenmeyer flask, and again extract the chloroform solution in the separator with two 25-mL portions of water. Retain the combined aqueous extracts, which will be used in the test for *Limit of Free Glycerin*. Transfer the chloroform layer to a 500-mL glass-stoppered Erlenmeyer flask, and add 50.0 mL of *Periodic acid solution* to this flask and to each of two blank flasks containing a mixture of 50 mL of *Chloroform* and 10 mL of water. Swirl the flasks during the addition of *Periodic acid solution*, and allow to stand for at least 30 min, but no longer than 90 min. To each flask, add 20 mL of potassium iodide TS, and allow to stand at least 1 min, but no longer than 5 min, before titrating. Add 100 mL of water, and titrate with 0.1 N sodium thiosulfate VS, using a magnetic stirrer to keep the solution thoroughly mixed, to the disappearance of the brown iodine color. Add 2 mL of starch TS, and continue the titration to the disappearance of the blue color.

Calculate the percentage of 1-monoglycerides in the portion of Superglycerinated Fully Hydrogenated Rapeseed Oil taken:

$$\text{Result} = \{[M_1 \times (V_B - V_S) \times N]/(W \times A)\} \times 100$$

M_1 = molecular weight of glyceryl monostearate, 358

V_B = volume of sodium thiosulfate VS consumed in the blank determination (mL)

V_S = volume of sodium thiosulfate VS required in the titration of the Superglycerinated Fully Hydrogenated Rapeseed Oil (mL)

N = normality of the sodium thiosulfate VS

W = weight of the Superglycerinated Fully Hydrogenated Rapeseed Oil taken to prepare the *Sample solution* (mg)

A = factor number, 2

Acceptance criteria: 90.0%–110.0% of that indicated on the label

IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.5%, when a 5-g sample of Superglycerinated Fully Hydrogenated Rapeseed Oil is ignited at an ignition temperature of 800 ± 25°

• **Limit of Nickel**

Sample solution: Weigh 5.0 g of Superglycerinated Fully Hydrogenated Rapeseed Oil into a previously tared platinum or silica crucible. Cautiously heat the substance, and introduce into it a wick formed from twisted ashless filter paper. Ignite the wick. When the substance ignites, stop heating. After combustion, ignite in a muffle furnace at about 600°. Continue the incineration until white ash is obtained. After cooling, transfer the residue, with the aid of two 2-mL portions of diluted hydrochloric acid, to a 25-mL volumetric flask. Add 0.3 mL of nitric acid, and dilute with water to volume.

Nickel standard solution: Immediately before use, dilute 10 mL of nickel standard solution TS with water to 500 mL. This solution contains the equivalent of 0.2 µg/mL of nickel.

Standard solutions: Into three identical 10-mL volumetric flasks, introduce respectively 1.0, 2.0, and 4.0 mL of *Nickel standard solution*. To each flask, add a 2.0-mL portion of the *Sample solution*, and dilute with water to volume.

Instrumental conditions

(See *Atomic Absorption Spectroscopy ⟨852⟩*.)

Mode: Atomic absorption spectrophotometer equipped with a graphite furnace

Analytical wavelength: 232.0 nm

Lamp: Nickel hollow-cathode

Analysis

Samples: *Sample solution* and *Standard solutions*

Concomitantly determine the absorbances at least three times each, at the wavelength of maximum absorbance. Record the average of the steady readings for each of the *Standard solutions* and the *Sample solution*. Plot the absorbances of the *Sample solution* and the *Standard solutions* versus the added quantity of nickel.

[Note—The *Sample solution* should be plotted as if it had a content of added nickel equivalent to 0 µg.]

Extrapolate the line joining the points on the graph until it meets the concentration axis. The distance between this point and the intersection of the axes represents the concentration of nickel, C, in µg/mL, in the *Sample solution*.

Calculate the content of nickel in the portion of Superglycerinated Fully Hydrogenated Rapeseed Oil taken:

$$\text{Result} = (V \times C)/W$$

V = volume of the *Sample solution*, 25 mL

C = concentration of nickel in the *Sample solution* (µg/mL)

W = weight of Superglycerinated Fully Hydrogenated Rapeseed Oil taken to prepare the *Sample solution* (g)

Acceptance criteria: NMT 1 µg/g (ppm)

• **Limit of Erucic Acid:** NMT 1.0%, as determined in the test for *Fats and Fixed Oils ⟨401⟩, Fatty Acid Composition*

• **Limit of Free Glycerin**

Periodic acid solution and **Chloroform:** Prepare as directed in the test for *Content of 1-Monoglycerides*.

Sample solution: Use the combined aqueous extracts obtained as directed in the test for *Content of 1-Monoglycerides*.

Analysis: Transfer 50.0 mL of *Periodic acid solution* to each of two flasks: a 500-mL glass-stoppered Erlenmeyer flask containing the *Sample solution* and a 500-mL glass-stoppered Erlenmeyer blank flask containing 75 mL of water. Continue as directed for *Analysis* in the test for *Content of 1-Monoglycerides*, beginning with "Swirl the flasks during the addition of *Periodic acid solution*, and allow to stand for at least 30 min, but no longer than 90 min".

Calculate the percentage of free glycerin in the portion of Superglycerinated Fully Hydrogenated Rapeseed Oil taken:

$$\text{Result} = \{[M_1 \times (V_B - V_S) \times N]/(W \times A)\} \times 100$$

M_1 = molecular weight of glycerin, 92

V_B = volume of sodium thiosulfate VS consumed in the blank determination (mL)

V_S = volume of sodium thiosulfate VS required in the titration of the Superglycerinated Fully Hydrogenated Rapeseed Oil (mL)

N = normality of the sodium thiosulfate VS

W = weight of the Superglycerinated Fully Hydrogenated Rapeseed Oil taken to prepare the *Sample solution* as directed in the test for *Content of 1-Monoglycerides* (mg)

A = factor number, 4

Acceptance criteria: NMT 1%

SPECIFIC TESTS

• **Fats and Fixed Oils,** *Acid Value* ⟨401⟩**:** NMT 6.0

• **Fats and Fixed Oils,** *Fatty Acid Composition* ⟨401⟩**:** Superglycerinated Fully Hydrogenated Rapeseed Oil exhibits the fatty acid composition profile shown in *Table 1*.

Table 1

Carbon-Chain Length	Number of Double Bonds	Percentage (%)
14	0	<1.0
16	0	3–5
18	0	38–42
20	0	8–10
22	0	42–50
24	0	1.0–2.0
18	1	≤1.0
18	2	<1.0
20	1	<1.0
22[a]	1	≤1.0

[a] Erucic acid.

• **Fats and Fixed Oils,** *Hydroxyl Value* ⟨401⟩**:** NLT 90.0% and NMT 110.0% of that indicated on the label

• **Fats and Fixed Oils,** *Iodine Value* ⟨401⟩**:** NMT 4

• **Fats and Fixed Oils,** *Peroxide Value* ⟨401⟩**:** NMT 2.0

• **Fats and Fixed Oils,** *Unsaponifiable Matter* ⟨401⟩**:** NMT 1.5%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight, light-resistant containers. No storage requirements specified.

• **Labeling:** Label it to indicate the hydroxyl value and the content of 1-monoglycerides.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
SUPERGLYCERINATED FULLY HYDROGENATED RAPESEED OIL	Documentary Standards Support	SE2020 Simple Excipients

Topic/Question	Contact	Expert Committee
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SE2020 Simple Excipients

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