Glyceryl Dibehenate

DEFINITION

Glyceryl Dibehenate is a mixture of diglycerides, mainly glyceryl dibehenate, together with variable quantities of monoglycerides and triglycerides. It contains NLT 15.0% and NMT 23.0% of monoglycerides, NLT 40.0% and NMT 60.0% of diglycerides, and NLT 21.0% and NMT 35.0% of triglycerides. It is obtained by esterification of glycerin with behenic (docosanoic) acid. The fatty acid may be of vegetable or synthetic origin.

IDENTIFICATION

• **A.** It meets the requirements for the content of diglycerides in the *Assay*.

• **B.** It meets the requirements for *Fats and Fixed Oils ⟨401⟩, Fatty Acid Composition* in *Specific Tests*.

• **C.** **Melting Range or Temperature,** *Class II ⟨741⟩*: 65°−77°

ASSAY

• **Procedure**

Mobile phase: Tetrahydrofuran

System suitability solution: 40.0 mg/mL of USP Glyceryl Dibehenate RS in tetrahydrofuran. Heat gently at about 35°.

Sample solution: 40.0 mg/mL of Glyceryl Dibehenate in tetrahydrofuran. Heat gently at about 35°. Determine the total mass of solvent and substance.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: Refractive index

Column: 7.5-mm × 60-cm; 5-µm 100-Å packing L21

[Note—Two 7.5-mm × 30-cm L21 columns may be used in place of one 60-cm column, provided system suitability requirements are met.]

Temperatures

Detector: 35°

Column: 35°

Flow rate: 1.0 mL/min

Injection volume: 40 µL

[Note—Maintain the temperature of the sample at 35° to avoid precipitation.]

System suitability

Samples: *System suitability solution* and *Sample solution*

Suitability requirements: The retention times of the major peaks of the *Sample solution* correspond to those of the *System suitability solution*.

[Note—The relative retention times for triglycerides, diglycerides, monoglycerides, and glycerin are about 0.73, 0.76, 0.82, and 1.0, respectively.]

Analysis

Sample: *Sample solution*

Calculate the percentage of monoglycerides in the portion of Glyceryl Dibehenate taken:

$$Result = [(r_{U1}/r_S) \times (100 - A - B)] - D$$

r_{U1} = peak area of monoglycerides

r_S = sum of the peak areas of monoglycerides, diglycerides, and triglycerides

A = content of free glycerin from *Limit of Free Glycerin* (%)

B = content of water from the *Water Determination* test in *Specific Tests* (%)

D = content of free fatty acids (%) using the following formula:

$$D = AV \times [M_{r1}/(M_{r2} \times 1000)] \times 100$$

AV = acid value, determined in the *Acid Value* test in *Specific Tests*

M_{r1} = molecular weight of behenic acid, 340.58

M_{r2} = molecular weight of potassium hydroxide, 56.11

1000 = conversion of mg to g

Calculate the percentage of diglycerides in the portion of Glyceryl Dibehenate taken:

$$Result = (r_{U2}/r_S) \times (100 - A - B)$$

r_{U2} = peak area of diglycerides

r_S = sum of the peak areas of monoglycerides, diglycerides, and triglycerides

A = content of free glycerin from *Limit of Free Glycerin* (%)

B = content of water from the *Water Determination* test in *Specific Tests* (%)

Calculate the percentage of triglycerides in the portion of Glyceryl Dibehenate taken:

$$Result = (r_{U3}/r_S) \times (100 - A - B)$$

r_{U3} = peak area of triglycerides

r_S = sum of the peak areas of monoglycerides, diglycerides, and triglycerides

A = content of free glycerin from *Limit of Free Glycerin* (%)

B = content of water from the *Water Determination* test in *Specific Tests* (%)

Acceptance criteria
Monoglycerides: 15.0%–23.0%
Diglycerides: 40.0%–60.0%
Triglycerides: 21.0%–35.0%

IMPURITIES

• **RESIDUE ON IGNITION ⟨281⟩:** NMT 0.1%

• **LIMIT OF FREE GLYCERIN**
  Mobile phase, System suitability solution, and **Chromatographic system:** Proceed as directed in the *Assay*.
  Standard stock solution: 4.0 mg/mL of USP Glycerin RS in tetrahydrofuran
  Standard solutions: To four 15-mL flasks transfer 0.25, 0.5, 1.0, and 2.0 mL of the *Standard stock solution*, respectively, and add 5.0 mL of tetrahydrofuran. Determine the concentration for each (mg/g).
  Sample solution: Transfer 0.2 g of Glyceryl Dibehenate to a 15-mL flask, and add 5.0 mL of tetrahydrofuran. Heat gently at about 35°. Determine the total mass of solvent and substance.
  System suitability
    Samples: *System suitability solution* and *Sample solution*
    Suitability requirements: The retention times of the major peaks of the *Sample solution* correspond to those of the *System suitability solution*.
    [NOTE—The relative retention times for triglycerides, diglycerides, monoglycerides, and glycerin are about 0.73, 0.76, 0.82, and 1.0, respectively.]
  Analysis
    Samples: *Standard solutions* and *Sample solution*

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Using the chromatograms from the *Standard solutions*, measure the responses for the glycerin peaks. Plot the concentration, in mg/g, of USP Glycerin RS in the *Standard solutions* versus the glycerin peak responses obtained. From the standard curve so obtained, determine the glycerin concentration, *C*, in mg/g, in the *Sample solution*.

Calculate the percentage of free glycerin in the portion of Glyceryl Dibehenate taken:

$$Result = (C/C_U) \times 100$$

C = concentration of glycerin determined from the calibration curve in the *Sample solution* (mg/g)

C_U = concentration of Glyceryl Dibehenate in the *Sample solution* (mg/g)

Acceptance criteria: NMT 1.0%

SPECIFIC TESTS

• **FATS AND FIXED OILS,** *Acid Value, Method I* ⟨401⟩

Sample: 1 g

Analysis: Suspend the *Sample* in 50 mL of a mixture of alcohol and toluene (1:1) with gentle heating. Continue as directed in *Fats and Fixed Oils ⟨401⟩, Acid Value*, beginning with "Add 1 mL of phenolphthalein TS".

Acceptance criteria: NMT 4

• **FATS AND FIXED OILS,** *Fatty Acid Composition* ⟨401⟩**:** Glyceryl Dibehenate exhibits the composition profile of fatty acids in *Table 1*, as determined in the chapter.

Table 1

Carbon-Chain Length	No. of Double Bonds	Percentage (%)
16	0	≤3.0
18	0	≤5.0
20	0	≤10.0
22	0	≥83.0
22	1	≤3.0
24	0	≤3.0

• **WATER DETERMINATION,** *Method Ia* ⟨921⟩**:** NMT 1.0% on 1.0-g sample. Use pyridine as the solvent.

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight containers at a temperature not higher than 35°.

• **LABELING:** Label to indicate the origin of behenic acid and glycerin.

• **USP REFERENCE STANDARDS** ⟨11⟩
  USP Glycerin RS
  USP Glyceryl Dibehenate RS

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

Topic/Question	Contact	Expert Committee
GLYCERYL DIBEHENATE	Documentary Standards Support	CE2020 Complex Excipients

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Chromatographic Database Information: Chromatographic Database

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