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Coix Seed Powder

DEFINITION

Coix Seed Powder, also known as Job's tears powder, consists of the dried ripe caryopsis, freed from the shell, of *Coix lacryma-jobi* var. *ma-yuen* (Rom.Caill.) Stapf (Fam. Poaceae, alt. Gramineae) collected in the fall and reduced to a fine or very fine powder. It contains NLT 3.5% of triglycerides calculated as the sum of trilinolein ($C_{57}H_{98}O_6$), 1,2-dilinoeloyl-3-palmitin ($C_{55}H_{98}O_6$), 1,2-dilinoeloyl-3-olein ($C_{57}H_{100}O_6$), 1-palmitoyl-2-oleoyl-3-linolein ($C_{55}H_{100}O_6$), 1,2-dioleoyl-3-linolein ($C_{57}H_{102}O_6$), 1,2-dioleoyl-3-palmitin ($C_{55}H_{102}O_6$), and triolein ($C_{57}H_{104}O_6$) on the dried basis.

IDENTIFICATION

• A. HPTLC FOR ARTICLES OF BOTANICAL ORIGIN (203)

Standard solution A: 2 mg/mL of [USP Oleic Acid RS](#) in [methanol](#)

Standard solution B: 40 mg/mL (about 50 µL/mL) of [USP Coix Seed Oil Extract RS](#) in [methylene chloride](#)

Sample solution: Sonicate 0.5 g of Coix Seed Powder in 5 mL of [methylene chloride](#) for 10 min. Centrifuge at 5000 rpm for 5 min and use the supernatant.

Chromatographic system and System suitability

Use [Identification of Fixed Oils by Thin-Layer Chromatography \(202\), Identification, Method II](#).

Derivatization reagent: 25 mg/mL of [phosphomolybdic acid](#) in 96% alcohol

Analysis

Samples: *Standard solution A*, *Standard solution B*, and *Sample solution*

Apply the *Samples* as bands and dry in air. Develop in a saturated chamber up to 7 cm, remove the plate from the chamber, and dry in air.

Treat the plate with *Derivatization reagent*, heat at 120° for 3 min, and examine in white light.

Acceptance criteria: The *Sample solution* exhibits, in the upper half, a band corresponding in R_f and color to the oleic acid band in *Standard solution A*. The *Sample solution* exhibits additional bands that correspond to similar bands in *Standard solution B*, including one band right above oleic acid with the same color, a cluster of four blue bands in the lower half, and some faint bands in the middle third.

• B. HPLC

Analysis: Proceed as directed in the test for *Content of Triglycerides*.

Acceptance criteria: The *Sample solution* exhibits a peak with a retention time corresponding to triolein in *Standard stock solution* and peaks due to trilinolein, 1,2-dilinoeloyl-3-palmitin, 1,2-dilinoeloyl-3-olein, 1-palmitoyl-2-oleoyl-3-linolein, 1,2-dioleoyl-3-linolein, and 1,2-dioleoyl-3-palmitin at retention times corresponding to the same triglycerides in *Standard solution 6*. The 1,2-dilinoeloyl-3-olein and 1,2-dioleoyl-3-linolein are the most intense peaks; 1-palmitoyl-2-oleoyl-3-linolein and triolein are medium-intensity peaks; trilinolein, 1,2-dilinoeloyl-3-palmitin, and 1,2-dioleoyl-3-palmitin are low-intensity peaks (distinction from *Sorghum bicolor* (L.) Moench, which has similar triglyceride peaks, all of low intensity, and *Hordeum vulgare* L., which has four triglyceride peaks, all of low intensity).

COMPOSITION

• CONTENT OF TRIGLYCERIDES

Mobile phase: [Methanol](#)

Standard stock solution: 0.20 mg/mL of USP Triolein RS in [methanol](#). Sonicate to dissolve.

Standard solutions 1–5: Dilute the *Standard stock solution* with [methanol](#) to obtain solutions of 0.0125, 0.025, 0.05, 0.10, and 0.20 mg/mL of USP Triolein RS. Pass each through a suitable membrane filter of 0.45-µm or finer pore size.

Standard solution 6: 0.5 mg/mL of [USP Coix Seed Oil Extract RS](#) in [methanol](#)

Sample solution: Accurately transfer about 500 mg of Coix Seed Powder into a suitable flask and add 35 mL of [methanol](#). Sonicate for 30 min and filter under vacuum. Rinse the original flask and the residue left in the flask with 5 mL of [methanol](#), and wash the residue and paper on the filter with the rinsing. Repeat the rinse and wash procedures one more time, and transfer the filtrate from the vacuum flask to a 50-mL volumetric flask. Wash the vacuum flask twice with about 2.5 mL of [methanol](#) each time, transfer the washings into the volumetric

flask, dilute with [methanol](#) to volume, and mix. Before injection, pass through a suitable membrane filter of 0.45-µm or finer pore size, and discard the first portion of the filtrate. [NOTE—Dried filter and vacuum flask should be used.]

Chromatographic system

(See [Chromatography \(621\)](#), [System Suitability](#).)

- Mode:** LC
- Detector:** Evaporative light-scattering. [NOTE—The parameters should be adjusted to achieve the best signal-to-noise ratio, according to manufacturer's recommendations.]
- Column:** 3.0-mm × 10-cm; 2.7-µm packing L7
- Column temperature:** 20°
- Flow rate:** 0.3 mL/min
- Injection volume:** 5 µL

System suitability

- Samples:** *Standard stock solution* and *Standard solutions 1–6*.
- Resolution:** NLT 1.5 between the peak of 1,2-dilinoleoyl-3-olein and the small peak following it, *Standard solution 6*
- Tailing factor:** NMT 1.5 for the triolein peak, *Standard stock solution*
- Relative standard deviation:** NMT 5.0% for the triolein peak in repeated injections, *Standard stock solution*
- Correlation coefficient:** NLT 0.995 for the regression line as determined in *Analysis*, *Standard solutions 1–5*
- Signal-to-noise ratio:** NLT 15 for the triolein peak, *Standard solution 1* (0.0125 mg/mL)
- Chromatogram similarity:** The chromatogram of *Standard solution 6* is similar to the reference chromatogram provided with the lot of [USP Coix Seed Oil Extract RS](#) being used.

Analysis

- Samples:** *Standard solutions 1–6* and *Sample solution*
- Using the chromatograms of *Standard solutions 5–6* and the reference chromatogram provided with the lot of [USP Coix Seed Oil Extract RS](#) being used, identify the retention times of the peaks of the relevant analytes in the *Sample solution*. [NOTE—See [Table 1](#) for the approximate relative retention times.]

Table 1

Analyte	Approximate Relative Retention Time
Trilinolein	0.61
1,2-Dilinoeoyl-3-palmitin	0.67
1,2-Dilinoeoyl-3-olein	0.71
1-Palmitoyl-2-oleoyl-3-linolein	0.79
1,2-Dioleoyl-3-linolein	0.84
1,2-Dioleoyl-3-palmitin	0.94
Triolein	1.00

Plot the logarithms of peak responses versus the logarithms of concentrations, in mg/mL, of triolein from *Standard solutions 1–5*. Using a least-squares analysis, establish a regression line or determine a linear regression equation.

Determine the concentration (C), in mg/mL, of the relevant analytes in the *Sample solution* by using the regression line or linear regression equation.

Separately calculate the percentages of trilinolein, 1,2-dilinoeoyl-3-palmitin, 1,2-dilinoeoyl-3-olein, 1-palmitoyl-2-oleoyl-3-linolein, 1,2-dioleoyl-3-linolein, 1,2-dioleoyl-3-palmitin, and triolein in the portion of Coix Seed Powder taken:

Result = C × (V/W) × 100

- C = concentration of relevant analyte in the *Sample solution* as determined above (mg/mL)
- V = volume of the *Sample solution* (mL)
- W = weight of Coix Seed Powder taken to prepare the *Sample solution* (mg)