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Cocoa Butter

DEFINITION

Cocoa Butter is the fat obtained from the seed of *Theobroma cacao* L. (Fam. Sterculiaceae).

SPECIFIC TESTS

• **Fatty Acid Composition**

Sample solution: Place 100–150 mg of Cocoa Butter in a 50-mL round-bottom flask, and add 4 mL of 0.5 N sodium hydroxide solution, prepared in methanol. Add a few boiling chips to the flask, connect the round-bottom flask to a condenser, and boil the mixture under total reflux until the fat globules go into solution. Add 5.0 mL of a 2.0 M borontrifluoride in methanol solution to the boiling mixture via the condenser, and continue boiling for 2 min. Add 2–5 mL of chromatographic *n*-heptane to the boiling mixture via the condenser, and boil for another min. Remove the flask from the source of heat, and remove the reflux condenser. Add saturated sodium chloride solution, and swirl the flask gently. Add more of the saturated sodium chloride solution to bring the liquid level into the neck of the round-bottom flask. Transfer 1 mL of the organic layer into a glass-stoppered test tube, add some anhydrous sodium sulfate to remove the last traces of water, and filter. Use the filtrate.

System suitability solution: 1 mg/mL each of methyl stearate and methyl oleate, in *n*-heptane

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 0.25-mm × 15-m fused-silica capillary; 0.25-µm stationary phase G19 coating

Temperature

Detector: 250°

Injection port: 250°

Column: See *Table 1*.

[Note—The components of interest elute during the temperature program. The final hold at a temperature of 240° serves only to facilitate elution of higher boiling components.]

Table 1

Initial Temperature (°)	Temperature Ramp (°/min)	Final Temperature (°)	Hold Time at Final Temperature (min)
180	10	240	5

Carrier gas: Helium

Linear velocity: 48 cm/s

Injection size: 0.1 µL

Injection type: Split

Split ratio: 60:1

System suitability

Sample: *System suitability solution*

[Note—The relative retention times for stearate and oleate are about 0.97 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 1.5 between the stearate and oleate peaks

Relative standard deviation: NMT 5.0%

Analysis

Sample: *Sample solution*

Measure the areas for the peaks of the methyl esters of the fatty acids. [NOTE—The relative retention times for palmitate, stearate, oleate, linoleate, linolenate (if present), and arachidate are about 1.0, 1.55, 1.60, 1.72, 1.89, and 2.30, respectively.]

Calculate the percentage of each fatty acid methyl ester in the portion of Cocoa Butter taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = response of each peak

r_T = sum of the responses of all of the peaks

Acceptance criteria: See *Table 2*.

Table 2

Fatty Acid Methyl Ester	Relative Retention Time	Percentage (%)
Palmitate	1.0	23–30
Stearate	1.55	31–37
Oleate	1.60	31–38
Linoleate	1.72	1.6–4.8
Linolenate (if present)	1.89	0–1.5
Arachidate	2.30	0–1.5

• **MELTING RANGE**

Analysis: Melt the material to be tested at a temperature of 50°–60°. Place 50 g of the melted material in a beaker, and cool in a water bath at 25°. Stir continuously until it assumes a pasty consistency, taking care to avoid the inclusion of air bubbles. Place the beaker in a water bath maintained at a temperature of 32°–33°. Continue stirring until the specimen reaches the temperature of the water bath and changes to a liquid cream (about 30 min). Pour the contents into another beaker, and allow it to solidify at room temperature for at least 2 h. Press one side of a U-shaped capillary tube, 1.5 mm in diameter and 80 mm in length with a distance of 10 mm between both capillaries, into the solidified specimen. Using a very fine metal rod, push the column down to 10 mm before the bend of the U-tube. Then attach the other arm of the U-tube to a precision thermometer (having 0.1° graduations) by suitable means, with the U-tube bend at the level of the thermometer bulb. Insert the thermometer into a water bath so that the upper edge of the material is at least 20 mm below the surface, and heat as directed under *Melting Range or Temperature ⟨741⟩, Class I* except, within 5° of the expected melting temperature, regulate the rate of the temperature rise so that it does not exceed 0.2°/min.

Acceptance criteria: The slip point (temperature at which the column visibly flows toward the bend in the tube) is 30°–34°. The clear melting point (clarity via magnifying glass) is 31°–35°.

• **REFRACTIVE INDEX ⟨831⟩:** 1.454–1.459 at 40°

• **FATS AND FIXED OILS,** *Free Fatty Acids ⟨401⟩*: The free fatty acids in 10.0 g of it require for neutralization NMT 5.0 mL of 0.10 N sodium hydroxide (1.4% as oleic acid).

• **FATS AND FIXED OILS,** *Iodine Value ⟨401⟩*: 33–42

• **FATS AND FIXED OILS,** *Saponification Value ⟨401⟩*: 188–198

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in well-closed containers.

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

Topic/Question	Contact	Expert Committee
COCOA BUTTER	Documentary Standards Support	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

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