

ORYZA OIL & FAT CHEMICAL CO., LTD.

1 Aza Numata Kitagata Kitagata-cho Ichinomiya-city, Aichi-prefecture 493-8001 JAPAN Phone:+81-586-86-5141 Facsimile:+81-586-86-6191 URL/http://www.oryza.co.jp/ E-mail:info@oryza.co.jp

PRODUCT STANDARD

PRODUCT NAME: JAPANESE BUTTERBUR EXTRACT-PC

(COSMETIC)

This product is extracted with aqueous ethanol from Japanese butterbur, the stem and the leaves of *Petasites japonicus* (Compositae).

<u>Appearance</u> <u>Identification</u> (1)Fukinolic acid Slight yellowish powder with slight characteristic odor.

After a small amount of methanol (HPLC grade) is added to 250mg of this product in a 10mL volume flask, and the flask is treated with ultra-sonic wave for 10 minutes. The solution is filtered through a 0.45 μ m PTFE filter after addition of methanol (HPLC grade) to adequate volume. For preparation of standard solution, methanol (HPLC grade) is added to fukinolic acid, and the concentration is prepared 0.1mg/mL (standard solution). HPLC analysis is performed according to the following conditions for 5 μ L of test solution and standard solution. The peak of fukinolic acid is found in the HPLC chromatogram of test solution.

<HPLC condition>

Column

Mobile phase

 : capcellpak C18 (4.6 mm φ × 250 mm)
: Solvent A = Citric acid solution (citric acid monohydrate 2.1g→1L)
Solvent B = Methanol

Condition of gradient

<u> </u>								
Time(min)	0	19	25	35	50	60	61	70
Mobile	100%	100%	80%A	80%A	60%A	60%A	100%	100%
phase	А	А	20%B	20%B	40%B	40%B	В	В
Flow rate : 1.0mL/min								
Detector	ctor : UV 324 nm							

Column temperature : 30°C

(2)Fukinone

This product (2.0g) in a centrifugation tube is suspended in purified water (10mL), and treated with ultra-sonic wave for 1 minute. Ethyl acetate (10mL) is added to the suspension, and the tube is shaken for 3 minutes. Then the tube is centrifuged (room temperature, 3000 rpm, 10minutes), and an ethyl acetate layer is collected (repeat this procedure three times). The collected ethyl acetate layer is evaporated and is dissolved in 20% acetonitrile (3mL), and is filtered through a Sep-Pak C18 cartridge. Then, the cartridge is washed with 70% acetonitrile (7mL) and is eluted with absolute acetonitrile (5mL). This solution is adjusted to 10mL, and filtered through a 0.45 μ m PTFE filter (test solution). For preparation of standard solution, acetonitrile is added to fukinone, and the concentration is prepared 0.1mg/mL (standard solution). HPLC analysis is performed according to the following conditions for 5 μ L of test solution and



ORYZA OIL & FAT CHEMICAL CO., LTD.

1 Aza Numata Kitagata Kitagata-cho Ichinomiya-city, Aichi-prefecture 493-8001 JAPAN Phone:+81-586-86-5141 Facsimile:+81-586-86-6191 URL/http://www.oryza.co.jp/ E-mail:info@oryza.co.jp

	standard solution. The peak of fukinone is found in the HPLC chromatogram of test solution. $<$ HPLC condition>Column: Shim-pack CLC-ODS (6.0 mm $\phi \times 150$ mm)Mobile phase: acetonitrile : water = 8 : 2Flow rate: 1.0mL/minDetector: UV 254 nmColumn temperature : 30°C				
Total polyphenols	Min. 1.0 %	(Folin-Denis method)			
Loss on Drying	Max. 10.0 %	(Analysis for Hygienic Chemists,			
		1g, 105 °C, 2 hr)			
Purity Test					
(1)Heavy Metals (as Pb)	Max. 10 ppm	(Sodium Sulfide Colorimetric Method)			
(2)Arsenic (as As ₂ O ₃)	Max. 1 ppm	(Standard Methods of Analysis in Food			
		Safety Regulation, The Third Method,			
		Apparatus B)			
<u>Standard Plate Counts</u>	Max. 1×10^2 cfu/g	(Analysis for Hygienic Chemists)			
Moulds and Yeasts	Max. 1×10^2 cfu/g	(Analysis for Hygienic Chemists)			
<u>Coliforms</u>	Negative	(Analysis for Hygienic Chemists)			
<u>Composition</u>	Ingredient	Content (Values are just a guide)			
	Dextrin	80 %			
		Leaf/Stem Extract 20 %_			
	Total	100 %			
<u>Expiry date</u> <u>Storage</u>	2 years from date of manufacturing. Store it in a cool, dry, ventilated area with desiccant. Keep it away from high temperature and sunlight, and store it in a closed container.				

Established Date	June 5, 2004
Revised Date	May 28, 2020
Specification No.	T-028EN