Republic of the Philippines
Department of Health
FOOD AND DRUG ADMINISTRATION

29 JAN 2018

FDA CIRCULAR
No. 2018-003

TO: **ALL COSMETIC MANUFACTURERS, TRADERS, DISTRIBUTORS AND OTHER CONCERNED PARTIES**

SUBJECT: **Updates and Amendments of the ASEAN Cosmetic Directive as Adopted During the 27th ASEAN Cosmetic Committee (ACC) Meeting and Its Related Meetings**

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1. BACKGROUND AND RATIONALE

In 2005, the Department of Health (DOH) – Food and Drug Administration (FDA), then called Bureau of Food and Drugs (BFAD), has adopted and implemented the ASEAN Harmonized Cosmetic Regulatory Scheme and the ASEAN Common Technical Documents through Administrative Orders No. 2005-0015 and 2005-0025, respectively. The harmonization scheme involves the conduct of alignment meetings for the purpose of eliminating trade barriers and enhancing cooperation within the ASEAN Member States (AMS) in ensuring the safety, quality and claimed benefits of cosmetic products.

On 14 to 17 November 2017, the ASEAN Cosmetic Committee (ACC) convened in Bandung, Indonesia for the 27th ACC, 27th ASEAN Cosmetic Scientific Body (ACSB) and 10th ASEAN Cosmetic Testing Laboratories Committee (ACTLC) Meetings. As part of our continuous commitment to provide timely and relevant information on standards, rules, and regulations, the Center for Cosmetics Regulation and Research (CCRR) of the FDA hereby reports the highlights of the aforementioned meetings and presents the updates to the ASEAN Cosmetic Directive (ACD).

2. OBJECTIVE AND SCOPE

This Circular aims to provide the updates and amendments to the ACD as adopted in the 27th ACC meeting and its related meetings which covers cosmetic products made available in the local market. This Circular shall guide establishments that are engaged in the manufacture, importation, exportation, sale, offer for sale, distribution, donation, transfer, and where applicable, the use, testing, promotion, advertising, or sponsorship of cosmetic products.

3. UPDATES AND AMENDMENTS TO THE ACD

3.1. Updates and Amendments to the ACD Ingredient Annexes

The following items are the updates and amendments on cosmetic ingredients and their restrictions as indicated in the ACD Ingredient Annexes. The latest revision of the ACD Ingredient Annexes are posted in the FDA website under the ASEAN Cosmetic Harmonization section.

For easy reference, a table of the new and modified entries as well as the given grace period is provided in **Annex A**.

3.1.1. Oxidative and Non-Oxidative Hair Dyes in Annex III

3.1.1.1. The current entries for both oxidative and non-oxidative hair dyes in Annex III are amended to include the text '***The direction for use "wear suitable gloves" must be included in label or leaflet text***' as "other limitation and requirement" in column E.

3.1.1.2. Effective **01 December 2020**, only products bearing the statement "***wear suitable gloves***" on its label or leaflet can be placed in the market, and non-compliant products must be completely withdrawn from the market.

3.1.2. Hydroquinone (Annex III Ref. No. 14)

3.1.2.1. The superscript referring to footnote 2 "only if the concentration exceeds 0.05 %" under Column E in Annex III Ref. No. 14 (Hydroquinone) is removed.

3.1.3. Benzalkonium Chloride, Bromide and Saccharinate (Annex III Ref. No. 65)

3.1.3.1. The meeting agreed that no change is required to Annex III Ref. No. 65. However, the Philippines noted that products containing **benzalkonium chloride** which functions as an antiseptic agent fall under local drug regulations and are not classified as cosmetics.

3.1.4. Methylisothiazolinone (MI or MIT) in Rinse-Off Cosmetic Products (Annex VI Ref. No. 57)

3.1.4.1. The maximum authorized concentration of methylisothiazolinone in rinse-off cosmetic products is reduced from 0.01% to **0.0015% or 15 parts per million (ppm)**.

3.1.4.2. Effective **01 June 2019**, only compliant cosmetic products can be placed in the market and non-compliant products must be completely withdrawn from the market.

3.1.5. Hydroxyisohexyl 3-Cyclohexene Carboxaldehyde (HICC), 2,6-Dihydroxy-4-methyl-benzaldehyde (Atranol) and 3-Chloro-2,6-Dihydroxy-4-methyl-benzaldehyde (Chloroatranol)

3.1.5.1. New entries for HICC, Atranol and Chloroatranol are added in Annex II.

3.1.5.2. It is noted that since atranol and chloroatranol are present at trace levels in the fragrance ingredients, oakmoss and treemoss, with residue levels

restricted by IFRA quality standards, their inclusion in Annex II would not affect the use of oakmoss and treemoss as the presence of atranol and chloroatranol is technically unavoidable and allowed under ACD Article 4.

3.1.5.3. Effective **23 August 2019**, no cosmetic product with the banned materials can be placed in the market, and effective **23 August 2021**, non-compliant products must be completely withdrawn from the market.

3.1.6. Zinc Oxide (Annex IV Color Index 77947)

3.1.6.1. The current entry for Zinc Oxide in Annex IV (Color Index 77947) is amended to include the condition that the ingredient must "***not be used in applications that may lead to exposure of the end-user's lungs by inhalation***"

3.1.6.2. Effective **01 December 2018**, only compliant products can be placed in the market, and non-compliant products must be completely withdrawn from the market.

3.1.7. Ethyl Tosylamide

3.1.7.1. After a discussion on the scope of the entry "Sulphonamides (sulphanilamide and its derivatives obtained by substitution of one or more H-atoms of the -NH2 groups) and their salts" in ACD Annex II Ref. No. 307, and on the differences in the chemical structure of sulphanilamide and ethyl tosylamide, the Meeting concluded that ethyl tosylamide does not fall within the scope of the aforementioned Annex II entry, and therefore, is still permitted in cosmetic products.

3.2. 1,4-Dioxane

3.2.1. The cosmetic industry is urged to seek to achieve a 1,4-Dioxane trace level of ≤10 ppm through current Good Manufacturing Practices (GMP).

3.2.2. Further discussion of the International Cooperation on Cosmetic Regulation (ICCR) report on acceptable trace level of 1,4-Dioxane is tabled for the 28th ACSB Meeting.

3.3. ASEAN Joint Opinion Statement on Talc

The ASEAN Joint Opinion Statement on the Safety of Talc has been finalized and is undergoing final check to ensure correct reference formatting. Once this is completed, the ASEAN Joint Opinion Statement on Talc will be made available to the public in the ASEAN and FDA websites.

3.4. **ASEAN Cosmetic Method (ACM) 009**

ACM 009 with title "Determination of Salicylic Acid (Beta Hydroxy Acid) in Cosmetic Products by High Performance Liquid Chromatography (HPLC)" has been finalized by the ACTLC and endorsed by the ACC. The new ACM is attached as **Annex B** and is also available for download in the ASEAN Cosmetic Harmonization section of the FDA website.

3.5. Updated List of Cosmetic Accredited Laboratories

The list of accredited testing laboratories in the Philippines may be found in the Department of Trade and Industry – Philippine Accreditation Bureau (DTI-PAB) website (www.pabaccreditation.dti.gov.ph/public/public_test.php) and includes the scope of their accreditation.

3.6. Question and Answer (Q&A) on Specific Provisions of the ACD

The Q&A on the Specific Provisions of the ACD has been finalized and is undergoing final formatting. Once the final formatting is completed, the Q&A document will be made available to the public in the ASEAN and FDA websites.

3.7. Revised Terms of Reference (ToR) of the ACC

The revised ToR of the ACC has been finalized and is to be submitted to the ASEAN Consultative Committee on Standards and Quality (ACCSQ) for its endorsement during the 48th ACCSQ meeting.

4. PENALTY CLAUSE

Establishments engaged in the manufacture, importation, exportation, sale, offer for sale, distribution, donation, transfer, and where applicable, the use, testing, promotion, advertising, or sponsorship of cosmetic products who are found to be operating outside the rules and regulations of FDA shall be subjected to sanctions and penalties as prescribed by Republic Act No. 9711, otherwise knowns as the "Food and Drug Administration Act of 2009."

5. EFFECTIVITY

This Circular shall take effect immediately. Updates and amendments to the ACD Ingredient Annexes (Section 3.1) shall allow a grace period as specified above except for the entries with no specified grace periods.

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Annex A

Updates and Amendments to the ACD Ingredient Annexes

The updated ACD Ingredient Annexes incorporating the new and amended entries adopted during the 27th ACC Meeting and its related events are posted in the FDA website under the ASEAN Cosmetic Harmonization section. The table below representing the new and modified entries of the ACD Ingredient Annexes are shown only for easy reference of the cosmetic industry.

A. ACD Annex II – List of Substances Which Must Not Form Part of the Composition of Cosmetic Products

1. Hydroxyisohexyl 3-Cyclohexene Carboxaldehyde (HICC), 2,6-Dihydroxy-4-methyl-benzaldehyde (Atranol) and 3-Chloro-2,6-Dihydroxy-4-methyl-benzaldehyde (Chloroatranol)

Substances	CAS Number	Ref. No.	Effectivity Date
3- and 4-(4-Hydroxy-4-methylpentyl) cyclohex-3-ene-1-carbaldehyde (HICC)	51414-25-6 31906-04-4	1381	23 August 2019 – For new cosmetic products 23 August 2021 – For cosmetic products already available in the market
2,6-Dihydroxy-4-methyl-benzaldehyde (atranol)	526-37-4	1382	
3-Chloro-2,6-Dihydroxy-4-methyl-benzaldehyde (chloroatranol)	57074-21-2	1383	

B. ACD Annex III - List of Substances Which Cosmetic Products Must Not Contain Except Subject to Restrictions and Conditions Laid Down

1. Oxidative and Non-Oxidative Hair Dyes in Annex III

Other limitations and requirements (Column E)	Effectivity Date
Additional required warning statement that must be printed on the label or leaflet: "**Wear suitable gloves.**"	01 December 2020

2. Hydroquinone

Ref #	Substance	Restrictions			Conditions of use and warning which must be printed on the labels	Effectivity Date
		Field of application and/or use	Maximum authorised concentration in the finished cosmetic product	Other limitations and requirements		
14	Hydroquinone Hydroquinone CAS No 123-31-9	Artificial nail systems	0.02% (after mixing for use)	Professional use only[3]	• For professional use only • Avoid skin contact • Read directions for use carefully	Not Applicable

C. ACD Annex IV - List of Colouring Agents Allowed For Use in Cosmetic Products

1. Zinc Oxide (Color Index 77947)

Colour Index Number	Colour	Field of Application				Other Limitations and Requirements	Effectivity Date
		1	2	3	4		
77947	White	X				Not to be used in applications that may lead to exposure of the end-user's lungs by inhalation.	01 December 2018

D. ACD Annex VI - List of Preservatives Which Cosmetic Products May Contain

1. Methylisothiazolinone

Ref #	Substance	Maximum authorised concentration	Limitations and requirements	Conditions of use and warnings which must be printed on the label	Effectivity Date
57	2-Methyl-2H-isothiazol- 3-one (INCI) Methylisothiazolinone CAS No.2682-20-4	0.0015%	Rinse-off products only	---	01 June 2019

	Title	Revision n°	date	Document No
	DETERMINATION OF SALICYLIC ACID (BETA HYDROXY ACID) IN COSMETIC PRODUCTS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)	0		009

1. SCOPE AND FIELD OF APPLICATION

This test method describes the determination of salicylic acid in cosmetic products (cream).

2. PRINCIPLE

Salicylic Acid is a Beta Hydroxy Acid (BHA). Salicylic acid is used in skin-care products for skin exfoliate and acne care. It is soluble in ethyl alcohol and methyl alcohol which can be separated by HPLC. The concentration of salicylic acid in cosmetic products can be determined from its peak area in sample with the calibration curve of salicylic acid standard.

3. REAGENTS

3.1 General: all reagents used shall be of analytical purity.

3.1.1 salicylic acid, Reference standard

3.1.2 glacial acetic acid, AR grade

3.1.3 methyl alcohol, HPLC grade (MeOH)

3.1.4 Water shall be distilled water (H_2O)

3.2 Standard stock solution

Prepare 0.1 % (w/v) solution of salicylic acid in methyl alcohol.

3.3 Standard calibration solutions

Prepare standard calibration solutions concentration of 2, 6, 10, 20 and 30 µg/mL in mobile phase. Pipette the mix standard solution 20, 60, 100, 200 and 300 µL, respectively with automatic pipette to each 10 mL volumetric flask. Make up to volume with mobile phase and mix well. Label as S1, S2, S3, S4 and S5, respectively.

3.4 Mobile phase: MeOH : 1.5% v/v glacial acetic acid (55 : 45 by volume)

Preparation of mobile phase

Transfer 550 mL of methyl alcohol, 450 mL of 1.5% v/v glacial acetic acid into glass bottle 1000 ml, mix thoroughly upon ultrasonic bath for 5-10 min. (or degas process by unchanged ratio of mobile phase)

4. APPARATUS

Normal laboratory equipment and:

4.1 High Performance Liquid Chromatograph (HPLC) with a photodiode array detector and autosampler

4.2 Analytical column: Hypersil GOLD C18, 5 µm, 4.6 mm x 150 mm (Thermo SCIENTIFIC™) or equivalent.

4.3 Disposable syringe filter 0.45 µm (PVDF or equivalent)

4.4 Ultrasonic bath

4.5 Electronic balance, 0.1 mg (readability)

5. PROCEDURE

5.1 Sample solution preparation:

5.1.1 Weigh accurately 0.25 g of sample (duplicate A and B) into 25 mL volumetric flask.

5.1.2 Dissolve with 10 mL of methyl alcohol by vortexing or sonicate or warm at 60 °C for 2-5 mins as necessary until cream is dispersed.

5.1.3 Let the sample cool down to room temperature, then make up to volume with mobile phase and mix well. Dilute quantitatively and stepwise if necessary, with mobile phase.

5.1.4 Filter sample solution through disposable syringe filter (4.3) into vial with cap.

Note: The amount of the final concentration of sample solution shall be within calibration curve. If necessary, appropriate dilutions may be done as following guide.

5.2 Preparation of spiked sample solution for determination of percent recovery

5.2.1 Accurately weigh sample as in 5.1.1 (duplicate C and D).

5.2.2 Add known amount of standard to the sample, then follow procedure 5.2.1. Calculate the concentration of spiked standard at level 100% or 50% of concentration of SA in sample.

5.2.3 Dissolve spiked sample as in 5.1.1 and dilute quantitatively, and stepwise if necessary, with mobile phase. Filter spiked sample solution through disposable syringe filter (4.3) into vial with cap.

Note: The amount of standard addition to be calculated at level 100% or 50% of concentration of SA in sample and the final concentration shall be within calibration curve as following guide.

5.3 High performance liquid chromatography (HPLC)

5.3.1 Chromatographic conditions

5.3.1.1 Mobile phase: MeOH : 1.5% glacial acetic acid (55 : 45) by volume

5.3.1.2 Flow rate: 1.0 mL/minute

5.3.1.3 Photodiode array detection wavelength: 200 – 350 nm and λ_{max} at 236 or 302 nm

5.3.1.4 Column: Hypersil Gold C18, 5 µm, 150 mm. x 4.6 mm. Id

5.3.1.4 Injection volume: 20 µL

5.3.1.5 Run time: 5 mins (for Standard calibration solutions) 8-15 mins (for sample solutions)

5.4 Sequence of injection to the HPLC system.

Sequentially inject the prepared solution to HPLC and record peak area as follows:

5.4.1 System suitability: Inject standard solution, S1 (3.3) to examine the retention time and triplicate injections to determine standard deviation of peak area, tailing factor, resolution and K prime. The acceptance criteria are as follows

Name	%RSD of peak area (n=3)	Tailing factor	K prime
Salicylic acid	≤ 3	≤ 2.0	≥ 2

5.4.2 Inject S1, S2, S3, S4 and S5 (3.3), respectively for construction of the calibration curve.

5.4.3 Inject S3 (to compare the peak area with the peak area from calibration curve, % RPD should be < 3 %)

5.4.4 Inject mobile phase

5.4.5 Inject sample solution 1A, 1B, 1C, 1D

5.4.6 Inject S3 (to compare the peak area with the peak area from calibration curve, % RPD should be < 3 %)

5.4.7 Inject mobile phase

5.4.8 If there are more samples, inject S3 and mobile phase for each 4-10 injections. % RPD should be < 3 %)

5.4.9 Last vial is S3 to compare the peak area with the peak area from calibration curve, % RPD should be < 3 %)

6. CALCULATION

6.1 Construction of calibration curve between concentration and peak area of standard salicylic acid solutions. From linear regression equation:

$$A_0 = b_1C_0 + b_0$$

Where

b_1 = slope
b_0 = intercept
C_0 = concentration of salicylic acid µg/mL
A_0 = peak area

6.2 Calculation salicylic acid; SA in percentage by mass, using the formula:

$$salicylic\ acid\ (\%w/w) = \frac{conc.of\ SA\ in\ sample\ soln\ \left(\frac{\mu g}{ml}\right) \times\ dilution\ factor}{sample\ weight\ (g) \times\ 1{,}000\ \times\ 1{,}000} \times\ 100$$

$$dilution\ factor = \frac{initial\ volume\ of\ sample\ soln\ (ml)\ \times\ dilution\ volume\ (mL)}{volume\ from\ stock\ sample\ solution\ (mL)}$$

6.3 % Recovery

$$\%\,\text{recovery} = \frac{S - U}{C_{SA}} \times 100$$

Where,

S = concentration of salicylic acid in spiked sample, % w/w
U = concentration of salicylic acid in un-spiked sample, % w/w
C_{SA} = concentration of salicylic acid added, % w/w

6.4 % Relative Percent Different

$$\%\,\text{RPD} = \frac{A_2 - A_1}{\frac{A_1 + A_2}{2}} \times 100$$

Where,
A1 = peak area of S3 in calibration curve
A2 = peak area of S3 when inject interval in sequence of injection

7. REMARKS

7.1 Method validation information

7.1.1 Precision

7.1.1.1 Within day

Parameter	Cream product (%)
Within day (7 replicates): %RSD	0.04

7.1.1.2 Different days (Intermediate precision)

Parameter	Cream product
Between-day (5 days/7 replicates each) : p-value	0.47

7.1.2 Limits

Parameter	Cream product (%w/w)
Limit of Detection (LOD)	0.005
Limit of Quantitation (LOQ)	0.01

7.1.3 Expanded uncertainty

Salicylic acid in	Concentration (%w/w)	Expanded uncertainty at 95% confidence level	
		% w/w	Relative
Cream product	5.00	0.45	0.04

7.2 Chromatogram and spectrum of BHA (salicylic acid)