

## PHYSICAL STABILITY OF HAND WASHING SOAP PREPARATIONS LIME ESSENTIAL OIL (*Citrus aurantifolia*)

Definingsih Yuliasuti<sup>1\*</sup>, Dessy Ratna Sari<sup>1</sup>, Nina Melinda Erzaputri<sup>1</sup>

Department of Pharmacy, Health Polytechnic of Surakarta, Klaten, Indonesia

\*e-mail : [defie.farmasi17@gmail.com](mailto:defie.farmasi17@gmail.com)

### ABSTRACT

The human hand's palm is highly susceptible to the spread of bacteria and microorganisms, which can lead to infection. Pharmaceutical preparations that are able to kill germs and bacteria are soaps. Lime (*Citrus aurantifolia*) is a type of plant that contains essential oils that have antibacterial activity and can be used as an active ingredient in soap formulations. The purpose of this study was to determine the effect of storage cycles on the results of the physical stability test of hand soap preparations formulated with lime essential oils using the cycling test method. This test method was carried out for 6 cycles at temperatures of 4°C and 40°C for 24 hours each by testing several parameters of the physical stability of hand soap preparations, including organoleptic, homogeneity, pH, viscosity, and foam height. The results of the organoleptic stability of the soap preparation were in the form of a thick, yellow liquid and had a distinctive lime odour. The soap preparation was homogeneous during 6 cycles of storage, with an average pH of 5.98, a viscosity of 524-578 cPs, and an average foam height of 52 mm. The results of data analysis in the stability test using one-way ANOVA showed no significant difference between storage cycles, which means that the soap preparation was stable during the storage cycle.

**Keywords:** lime, physical, soap, stability

### INTRODUCTION

The skin is an organ that covers the entire human body. Another function of the skin is to protect the internal organs from physical harm such as friction, pulling, and heat from the sun. One part of the human body covered by skin is the hands, which are a highly conducive medium for the spread of bacteria and microorganisms. If the skin on the hands is injured, it is very easy for germs, bacteria, or microorganisms to enter the body. These bacteria or microorganisms can infect other body tissues, triggering infection (Rasyadi et al., 2019).

Skin infections can be caused not only by bacteria but also by open skin due to wounds. Another factor that can lead to infection is the transfer of microorganisms from unclean hands. One of the simplest ways to prevent infection and the spread of germs is to maintain skin hygiene, one of which is by washing hands with soap. This is the most effective way to suppress the spread of microorganisms on the skin (Rumlus et al., 2022).

Hand soap is a pharmaceutical preparation containing active antibacterial ingredients that function to inhibit bacterial growth and treat various diseases caused by bacteria (Lestari et al., 2020). Liquid hand soap is often used because it has several advantages compared to solid soap. Liquid soap is considered more hygienic, practical, and economical in its use. One natural ingredient that can be used as an active ingredient and has antibacterial activity in hand soap formulations is lime. Based on the results of research conducted by (Rahmawati et al., 2021), it is stated that flavonoids are abundant in lime peel. The flavonoid content found

in lime essential oil can inhibit bacterial growth and does not damage skin tissue (Triyani et al., 2021).

Physical stability tests on soap preparations are essential to ensure that the preparations have met the established standards and requirements. Stability tests aim to determine parameter criteria during storage. Physical instability in liquid soap is characterized by the formation of coalescence and other physical changes (Sativareza et al., 2021). Physical stability tests on liquid soap preparations include organoleptic tests, homogeneity tests, pH tests, viscosity tests, and foam height tests. The stability test aims to determine the stability value of a hand soap preparation in a short time, which can be carried out using the cycling test method. The cycling test method, or accelerated stability test method, aims to determine the stability of a produced preparation, whether it remains stable or experiences changes in physical quality during a predetermined storage period at a certain temperature (Rusmin, 2020).

According to the research results (Sulistyowati et al., 2019) the soap quality test on the solid soap formulation of lime with stevia leaves has met the soap requirements. Research related to the preparation of hand washing soap with essential oil formulations in lime (*Citrus aurantifolia*) has been conducted and showed that the hand soap was thick, had a distinctive fresh lime odour, and was yellow in colour (Yulastuti et al, 2026). Further research on the physical stability test of lime essential oil hand soap formulations is needed to determine the stability of the formulation. Therefore, researchers are interested in conducting research on the physical stability test of lime essential oil hand soap formulations.

**METHOD**

This type of research is a laboratory experimental study. Experimental research is a research method that functions to test the relationship between one variable and another and to examine the existence of a cause-and-effect relationship. This study aimed to determine whether differences between cycles influence the physical stability of the preparation (Sahir, 2022).

**Tools and Materials**

The tools used in this research are: stirring rod, porcelain cup, Erlenmeyer flask (pyrex), stamper, beaker (iwaki), analytical balance (ohaus), thermometer, measuring cup (pyrex), pH paper (triwarnaleather), oven, refrigerator, pipette, test tube rack, spatula, test tube (pyrex), ruler, timer, brookfiled viscometer, glass object (slides).

The materials used in this study include active substances in the form of lime essential oil and other additional ingredients including: glycerin (technical), sodium lauryl sulfate (technical), sodium chloride (technical), betaine (technical), HEC (technical), methyl paraben (technical), propyl paraben (technical), dye (technical), aquadest (technical).

**Work Procedures**

**a. Making Lime Essential Oil Hand Soap**

The first step in making lime essential oil hand soap is to prepare all the tools and ingredients and weigh each ingredient according to the measurements used in the hand soap formulation in table 3.2.

**Table 3.2 Formula**

Ingredients	Utility	Concentration (%)
Lime essential oil	Active ingredient	4
Glycerin	Humactant	8
Sodium lauryl sulfate	Surfactant	1
Sodium chloride	pH stabilizer	3
Betain	Foam booster	10

Hydroxyethyl Cellulose (HEC)	Thickener	1
Methylparaben	Preservative	0,18
Propylparaben	Preservative	0,02
Dye	Dye	Q.s
Water	Solvent	50 ml

The next process is to dissolve sodium lauryl sulfate in distilled water until dissolved. Then NaCl is added little by little and stirred until homogeneous, the soap solution is added with methyl paraben and propyl paraben and stirred until homogeneous, the addition of propyl and methyl paraben functions as a preservative that increases the antimicrobial activity of the soap preparation (Zulfa, 2020). Hydroxyethyl Cellulose (HEC) is first dissolved with glycerin before being added to the beaker glass, after which betaine is added and stirred until homogeneous. Lime essential oil is put in the beaker glass then stirred until homogeneous, and dye is added to taste. Then let the soap solution sit for a while, before being put into the container.

### **b. Physical stability test of liquid hand soap with lime essential oil**

The stability test was conducted using the cycling test method, which involves alternating accelerated storage times. The stability of the preparation was assessed by assessing organoleptic properties, homogeneity, pH, and foam height. The storage time was accelerated to six cycles over 12 days, with the preparation stored alternately at 4°C and 40°C. Each cycle was replicated three times (Usman & Baharuddin, 2023).

#### **1) Organoleptic Test**

Organoleptic testing includes testing the color, odor, taste, and consistency of the liquid soap preparation. Organoleptic testing is performed by observing the physical appearance of the liquid soap preparation using the five senses (Yusriyani, K.A, et al., 2022).

#### **2) Homogeneity Test**

The homogeneity test is conducted using a glass slide. A 0.5 ml sample of the hand soap preparation to be tested is taken, placed on the slide, and observed. A hand soap preparation is considered homogeneous if there are no coarse grains on the surface of the slide (Syamsu et al., 2022).

#### **3) pH Test**

The pH test is performed by weighing 1 ml of the preparation and diluting it with 10 ml of distilled water. The pH test is then performed using a pH meter that has been calibrated with a buffer solution. The electrode is dipped into the sample and the pH indicator is waited until it stabilizes and shows a constant reading (Lestari et al., 2021).

#### **4) Viscosity Test**

The viscosity test is performed using a Brookfield viscometer. 10 ml of the sample is placed in a container, then the spindle is inserted into the sample up to the mark. The motor is then turned on and left until the reading on the viscometer scale stabilizes (Mahayuni et al., 2023).

#### **5) Foam Height Test**

The foam test is performed by weighing 1 ml of the soap preparation, placing it in a test tube containing 10 ml of distilled water, and shaking for 20 seconds. Measure the initial foam height using a ruler, then measure the final foam height again after 5 minutes (Rinaldi et al., 2021).

### c. Data Analysis

The results of the physical stability test of liquid hand soap using lime (*Citrus aurantifolia*) essential oil were presented using bivariate analysis. Bivariate analysis is used to identify two variables suspected to have a relationship (Notoatmodjo, 2019). The variables used in this study consisted of independent and dependent variables. The independent variable in this study was the storage cycle of the stability test. Meanwhile, the dependent variable was the results of the soap stability test.

The test was conducted to determine whether the stability value of the preparation during storage from day 0 to 12. The stability test results were obtained using the Shapiro-Wilk normality test, and were considered normal if a significance value of  $>0.05$  was obtained. The analysis continued with a homogeneity test using the test of homogeneity of variance. The data were considered homogeneous if a significance value of  $>0.05$  was obtained. If the data obtained were normal and homogeneous, the next analysis used the One-Way ANOVA statistical test, and was considered significant if a value of  $>0.05$  was obtained. If the data obtained is not significant, data analysis can be carried out using Kruskal Waills (Muhid, 2019).

## RESULTS AND DISCUSSION

Lime peel contains essential oils, also known as essential oils. Lime essential oil is a pale yellow liquid with a distinctive lime odor and evaporates easily at high temperatures. Compounds in lime essential oil include limonene,  $\beta$ -pinene, linalool, and linalyl acetate. According to the certificate of analysis for lime essential oil as an active ingredient, lime essential oil contains alpha-pinene, beta-pinene, benzene, limonene, alpha-terpinolene, linalool,  $\alpha$ -citral, neryl acetate, and beta-bisabolene. Limonene is the compound with the highest concentration among the others (Sary *et al.*, 2020).

Limonene provides a distinctive, pungent aroma that can be used as a natural fragrance for hand soap (Ulandari *et al.*, 2022). Limonene has pharmacological effects such as antioxidant, antiviral, and antibacterial properties. In addition, high limonene levels can improve the quality of lime essential oil (Mubarok *et al.*, 2023).

This study created a handwashing soap preparation using lime essential oil (*Citrus aurantifolia*) as the active ingredient, which contains antioxidants and antibacterials that can kill bacteria on the skin of the hands. The soap preparation begins by weighing all the ingredients in the formula. Sodium lauryl sulfate, used as a surfactant in soap making, is placed in a beaker glass and dissolved in distilled water until dissolved. Then, NaCl is added little by little and stirred until homogeneous. The addition of NaCl is used as a pH stabilizer and can affect the soap's viscosity (Arrazi *et al.*, 2021). Methylparaben and propylparaben are added to the soap solution and stirred until homogeneous. The addition of propyl and methylparaben functions as preservatives that increase the antimicrobial activity of the soap preparation (Zulfa, 2020).

The addition of Hydroxyethyl Cellulose (HEC) to the formula is the result of a modification of the formula, so that the resulting soap has a thick and clear liquid form, Hydroxyethyl Cellulose (HEC) is first dissolved in glycerin to prevent clumping when added to the beaker glass. The next step is to add betaine as a foam booster or foaming agent to the soap preparation, this is done because during the soap-making experiment, the soap foam did not meet the foam height requirements so the addition of a foam booster is necessary (Fanani *et al.*, 2021). Lime essential oil is added to the beaker glass then stirred until homogeneous, and added with sufficient coloring. Then let the soap solution sit for a while, before being poured into the container.

This study tested the physical stability of a hand soap preparation containing *Citrus aurantifolia* essential oil and determined the effect of storage cycles. The soap was made with *Citrus aurantifolia* essential oil as the active ingredient, and its physical stability was tested. The physical stability test was conducted using the cycling test method. The cycling test method refers to previous studies. Furthermore, the cycling test aims to assess the stability of preparations consisting of a two-phase mixture, namely a water phase and an oil phase, such as soap. Parameters tested in the physical stability test include organoleptic testing, homogeneity testing, pH testing, viscosity testing, and foam height testing (Puspita et al., 2023).

Organoleptic testing of soap preparations was conducted to determine and observe changes in the shape, odor, and color of the soap preparation during storage. Organoleptic testing was conducted by describing the shape, odor, and color of the soap preparation. The standards set by SNI, the organoleptic test standards for liquid soap, include a thick and homogeneous form, and a distinctive odor and color (Yusriyani, A, et al., 2022). The results of the organoleptic stability test can be seen in Table 4.1.

**Table 4.1 Results of the Physical Organoleptic Stability Test of Lime (*Citrus aurantifolia*) Essential Oil Hand Soap**

Cycle	Parameter		
	Odor	Color	Shape
0	Typical lime	Yellow	Thick liquid
1	Typical lime	Yellow	Thick liquid
2	Typical lime	Yellow	Thick liquid
3	Typical lime	Yellow	Thick liquid
4	Typical lime	Yellow	Thick liquid
5	Typical lime	Yellow	Thick liquid
6	Typical lime	Yellow	Thick liquid

Table 4.1 shows the results of organoleptic physical stability testing of the soap preparation in cycle 0, which showed a liquid form, a distinctive lime odor, and a yellow color. Stability tests in cycles 1, 2, 3, 4, 5, and 6 showed no changes in the stability test parameters for form, odor, and color. The stability of the parameters in the soap form is due to the addition of glycerin to the formula, which is able to absorb water from the outside, resulting in an increased water content in the soap preparation, which will affect the form of the soap preparation (Febrianti, 2018).

The organoleptic stability results for odor and color in the soap, which have a distinctive lime odor and a yellow color, are due to the presence of limonene compounds in the addition of lime essential oil active ingredients in the soap formulation. Limonene compounds can provide a distinctive lime odor to the soap preparation, and the addition of dyes to the soap formula provides a more attractive color (Rusli et al., 2018). The stability of the soap's odor and color parameters is due to the addition of lime essential oil, which contains antioxidants that can function to prevent oxidation, which can cause the soap preparation to smell rancid and change color (Adiwibowo, 2020).

The results of the physical stability test on lime (*Citrus aurantifolia*) essential oil hand soap, conducted for 12 days or 6 cycles at 40°C and 4°C, showed no changes in shape, odor, or color during each storage cycle. This indicates that between cycles 0 and 6, the color, odor, and form were stable, with a yellow color, a distinctive lime odor, and a liquid form. The results of the organoleptic test are shown in Table 4.1.

The homogeneity stability test was conducted to detect any unequally distributed particles. The results of the stability test for each storage cycle of the lime (*Citrus aurantifolia*) essential oil hand soap preparation are shown in Table 4.2.

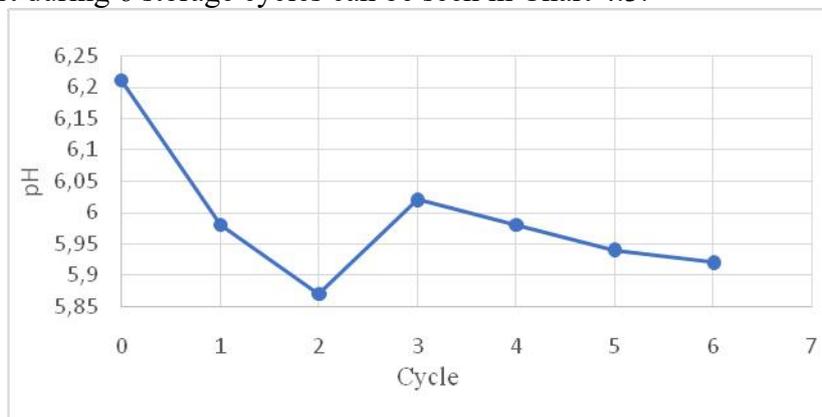
**Table 4.2 Results of the Physical Stability Test for the Homogeneity of Lime (*Citrus aurantifolia*) Essential Oil Hand Soap**

Cycle	Parameter
0	Homogeneous
1	Homogeneous
2	Homogeneous
3	Homogeneous
4	Homogeneous
5	Homogeneous
6	Homogeneous

The results of the physical stability test for the homogeneity of the soap preparation aim to determine whether all ingredients in the hand soap formula, such as active ingredients and additives, are well mixed (Mewar et al., 2023). The physical stability test for homogeneity is conducted by applying the soap preparation to a glass slide.

Based on Table 4.2, the results of the physical stability test for the homogeneity of the soap preparation in cycle 0 and each storage cycle, from cycles 1 to 6, show homogeneity, as indicated by the absence of particles on the glass slide. This study aligns with the requirements of the physical stability test for homogeneity, which states that the absence of small particles on the glass slide indicates a homogeneous composition of the soap preparation, making it safe for use (Indriaty et al., 2019). The stability test for the lime essential oil soap preparation concluded that the storage cycles did not affect the homogeneity of the soap preparation.

A physical stability test of the degree of acidity (pH) can be conducted to determine the acid or base level of the lime essential oil hand soap preparation. The results of the physical pH stability test during 6 storage cycles can be seen in Chart 4.3.



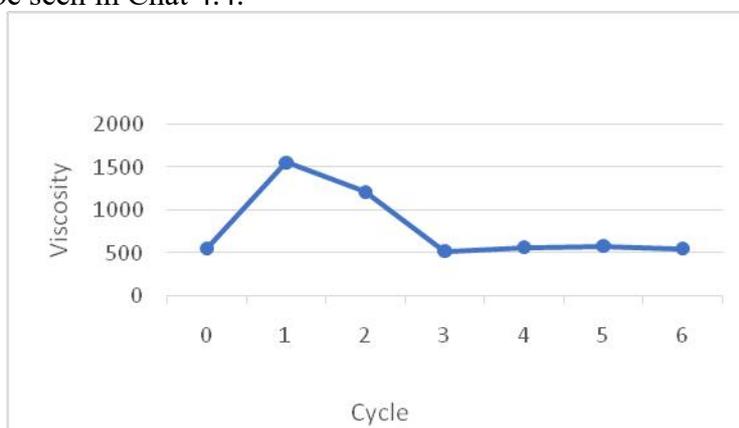
**Chat 4.3 Results of the Physical Stability Test for the Acidity Degree (pH) of Lime (*Citrus aurantifolia*) Essential Oil Hand Soap**

The pH stability test, or acidity level, is one of the requirements for soap quality. Because soap comes into direct contact with human skin, the pH value must be safe and meet established requirements (Kurniawati, 2022). The standard pH requirement for liquid soap preparations is between 4 and 10 (SNI-2588), ensuring the soap preparation is safe for use on the skin and does not cause irritation. The results of the pH stability test showed that all data met the requirements. However, after analyzing the data using a One-Way ANOVA test with a 95% confidence level, a significant effect was found between storage cycles.

The pH change began in the first storage cycle and continued into the second storage cycle. The decrease in pH can be caused by environmental factors such as temperature and humidity. The decrease in pH is influenced by temperature changes during stability testing. In

the third storage cycle, the pH value increased, which can cause skin irritation and dehydration. This increase in pH can be caused by the hydrolysis process between a weak acid and a strong base when the temperature rises. During the storage cycle, temperature changes accelerate the hydrolysis reaction, resulting in an increase in the pH of the soap preparation (Cahyaningsih et al., 2016). The pH of soap, which can irritate the skin, is set between 10-12 and can be controlled by adding acid to lower the pH value. However, the increase in pH in the third cycle is still within the established pH range, making it safe for application to the skin (Kristanti, 2021). In the fourth, fifth, and sixth storage cycles, the pH value decreases again. This decrease in pH is not only caused by temperature but also by the influence of the soap preparation's contact with air humidity during the storage process, where CO<sub>2</sub> gas reacts with the water in the preparation to form acid (Yustisi et al., 2023). According to Simatupang (2020), the decrease in pH can be caused by the duration of stirring. Increases and decreases in pH indicate a reaction or damage to the components of the liquid soap preparation, both active ingredients and additives (Muluuchah et al., 2021).

The physical viscosity stability test aims to determine the viscosity level of hand soap preparations during the storage cycle. The results of the physical stability test for the soap preparations can be seen in Chart 4.4.



**Table 4.4 Physical Stability Test Results for the Viscosity of Lime Essential Oil Hand Soap**

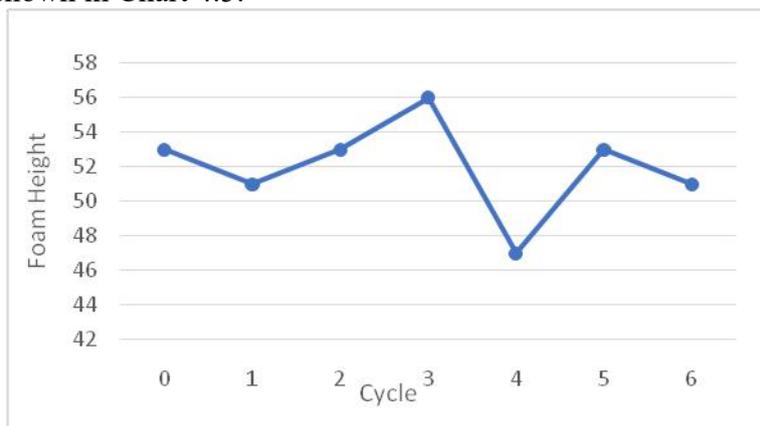
Chart 4.4 shows the results of the physical stability test for the soap preparation, which shows that all test data met the requirements. However, after analyzing the data using a One-Way ANOVA test with a 95% confidence level, there was a significant influence on the viscosity test results between storage cycles. The viscosity requirement for liquid soap, according to general standards, is 400-4000 cPs ((SNI 06-4085-1996).

The increase and decrease in viscosity values during storage are influenced by various factors. According to research conducted by Yanuarto et al. (2021), the increase in viscosity values during storage cycles 1, 4, and 5 is related to particle size during storage. The presence of interparticles that combine to reduce the surface area causes the particles to become larger and the surface area to decrease, thus increasing the soap's viscosity. Furthermore, soap viscosity can be caused by the addition of NaCl to the soap formula. The amount of electrolytes or salts added to soap can increase the viscosity, so adding NaCl to the soap base can thicken the soap by salting out the surfactant (Arrazi et al., 2021).

The decrease in viscosity that occurs during storage is due to the presence of glycerin in the soap preparation. The glycerin contained in the soap formula influences the decrease in viscosity because glycerin is hygroscopic, meaning it has the ability to absorb water vapor from the outside, thus The water content in soap preparations increases, causing the viscosity value of the soap to decrease (Zahra et al., 2019). In addition, the decrease in viscosity value is due to an increase in temperature during the storage process. An increase in temperature during the storage process will increase the distance between atoms, so that the interatomic

forces will decrease, which will cause a gap between atoms so that the viscosity value decreases (Yustisi et al., 2023). Another factor that can influence the decrease in viscosity value is the water content in the soap preparation; the higher the water content in the soap, the lower the viscosity value of the hand washing soap will be (Simatupang & Zalukhu, 2020). Another factor that influences instability in viscosity values can be caused by the treatment time span of each preparation, after storage at a temperature of 40°C.

Foam height testing was performed using a test tube using the cylinder shake method. The foam height stability test was conducted to determine foam stability, measured by the foam height in the test tube over a specified time period, and to measure the surfactant's ability to produce foam (Fikriana et al., 2023). The results of the physical stability test for foam height are shown in Chart 4.5.



**Chart 4.5 Physical Stability Results of Foam Height of Lime (*Citrus aurantifolia*) Essential Oil Hand Soap Preparation**

The results of the physical stability test for foam height of the soap preparation are shown in Chart 4.5. The foam height test for liquid soap has a requirement of 13-220 mm(SNI 06-4085-1996). The results table shows that all test data met the requirements. After analyzing the data using a One-Way ANOVA test with a 95% confidence level, it was found that there was no significant effect on the foam height test results between storage cycles, but the foam height stability test did increase.

Foam height stability is strongly influenced by the concentration and viscosity of the liquid soap preparation. Another factor influencing foam height is the size of the soap particles. The larger and more numerous the particles in the soap preparation, the foam height tends to decrease (Lestari et al., 2021). The foam height of the lime essential oil hand soap preparation had varying results, with increases and decreases in foam height due to the manual shaking process carried out by the researcher, resulting in unstable foam height results (Yustisi et al., 2023). Furthermore, the addition of betaine to the soap formulation affected the foam height (Fanani et al., 2021). The amount of foam in soap is also caused by the high surfactant concentration. SLS is a surfactant that functions as a surfactant in the formulation of lime essential oil hand soap (Handayani et al., 2020).

## CONCLUSION

The lime essential oil hand soap preparation exhibited good physical stability and met the required range for each physical quality parameter during the storage cycle. The soap preparation was a thick, yellow liquid with a distinctive lime odour. It maintained good homogeneity throughout the storage cycle. It had a pH range of 5.87-6.21, a viscosity range of 553-1555 cPs, and a foam height of 47-56 mm.

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