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Estimation shelf life on hand sanitizer that utilizes waru leaf (*Hibiscus tiliaceus*) waste using the accelerated method with arrhenius model

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Abstract. Reprocessing organic waste such as leaves and fruit peels can produce natural hand sanitizers with novelty or useful findings. Waru leaf hand sanitizer is a new innovation that uses natural ingredients and low alcohol. Natural extracts in products are still susceptible to damage, so an estimated shelf life is needed. Estimated shelf life is related to product quality, both producers and consumers really need it. Estimating the shelf life of an item is one way to find out how long the product can be stored. By using the ASLT (Accelerated Shelf Life Testing) method with the Arrhenius model, this study aims to determine how long the waru leaf hand sanitizer gel can last. For 18 days, the waru leaf hand sanitizer was stored at storage temperatures of 30°C, 35°C, and 40°C. Direct observation of the object of research (pH, texture, aroma, color, and absorbency) was used to obtain research data. Waru leaf hand sanitizer has 108.48 days (3.17 months) shelf life at 30°C, according to the results.

1. Introduction

Determining shelf life is an important thing to do regarding the quality of a product. Shelf-life information is needed by producers and consumers because it is closely related to the quality of the product. Hand sanitizer is one of the products that need to estimate shelf life, especially natural hand sanitizers. Natural hand sanitizers have constituent components in the form of natural ingredients. One form of natural hand sanitizer product innovation is waru leaf gel hand sanitizer. The choice of gel-shaped hand sanitizer is due to having high viscosity and adhesion so that it does not flow easily on the surface of the skin, is easily evenly distributed when applied, and leaves a film-like layer at the time of use. Therefore, the use of this type of gel is able to maintain hand hygiene longer.

Waru gel hand sanitizer consists of components of waru leaf extract and lime peel extract which have natural antimicrobial compounds. This hand sanitizer can be said to be a further processing product from the utilization of waru leaf and lime peel waste. The waru plant is a common shade plant on the roadside and beach. This plant has wide leaves and a tree height of about 6-7 meters. Utilization of waru plants is rarely done. Usually, waru leaves are just left to fall and become organic waste. Even though waru leaves have chemical components that have many benefits. Waru leaves have various bioactive components, such as phenolics, oleic acid, octadecanoic acid, hexadecanoic acid methyl ester, and so on which act as natural antioxidants and antimicrobials [1].



In addition to the waru plant, this hand sanitizer also uses lime peel. The utilization of citrus more on the pulp and juice causes a large amount of lime peel waste. In fact, lime peel waste still has bioactive compounds that have potential as natural fragrances, antioxidants, and antimicrobial ingredients. Lime peel contains ascorbic acid and flavonoids which acts as an antioxidant, alkaloids, terpenoids (essential oils), and saponins that act as antibacterials [2].

The use of waru leaf gel hand sanitizer is still vulnerable to damage considering that most of the components of this product are made from natural ingredients. Meanwhile, based on [3], waru leaf extract on gel hand sanitizer with a content of 25% was able to inhibit *Staphylococcus aureus* bacteria with an inhibitory score of 16.18 mm, dispersion power of 6.40 cm, pH 6.10 and no irritation to 10 panelists. Therefore, making hand sanitizers by reusing waste waru leaves and lime peels requires further research regarding estimating the shelf life of hand sanitizers as a way to ensure the safety and suitability of the product when it reaches consumers. The process of determining shelf life in hand sanitizer research can be done using the ASLT (Accelerated Shelf-Life Testing) method of the Arrhenius model. This method uses accelerated temperatures to speed up reactions that cause deterioration in the product. The choice of the ASLT method with the Arrhenius model is also due to this method being able to be done in a short time with good accuracy [4]. With the use of the Arrhenius model ASLT method, it is expected that the shelf life of the waru leaf gel hand sanitizer can be known.

2. Materials and methods

The ingredients used in making gel hand sanitizer are waru leaf extract (*Hibiscus tilaceus L.*) 25%, lime peel extract (*Citrus aurantifolia Swingle*) 25%, aquades 45 ml, CMC-Na (Food Grade) 0,5gram, glycerin 1 ml, and alcohol (Onemed, 70% and 96%). The tools used in making gel hand sanitizer are measuring cups, digital scales, pH meters, spatulas, ovens, grinders, sealers, centrifuges, and packaging aluminum foil bags with a thickness of 80 μm delkchoice brand.

The study was conducted using descriptive methods. The three storage temperatures for Waru leaf gel hand sanitizer are 30°C, 35°C, and 40°C with two repetitions. Furthermore, hand sanitizer storage is carried out with 7 storage points at 0, 3, 6, 9, 12, 15, and 18 days. After that, predetermined daily sample testing of pH, irritation test, and descriptive organoleptic test using sensory parameters, such as texture, scent, color, and absorption rate, is carried out. The test result data will be used to determine the shelf life with the Arrhenius model ASLT method using Microsoft Excel 2019 software.

2.1. Determination of shelf life

The quality value data in each parameter is then averaged, then plotted into a graph of the relationship between storage duration and quality value in each parameter. Based on this graph, a regression equation is obtained at each temperature with the form $y = kx + b$, where the y axis (ordinate) is a quality parameter, the x axis (abscissa) is the storage time (days), the b value is the initial product quality value and the k value is the % quality change rate per day (slope (k)).

In addition, there will also be a value of R² (correlation coefficient), where if the value of R² is greater and closer to 1 it will clarify the changes that occur. Next, graphing is carried out, for the value of ln k is plotted as the y-axis (ordinate) and 1/T is plotted as the x-axis (abscissa). The result of the graph of the relationship ln k with 1/T will produce a slope value in the form of a value of $-E_a/R$ in the Arrhenius equation and the intercept in the form of a value of k₀ is then entered into the formula [5]:

$$k = k_0 \cdot \exp(E_a/RT) \quad (1)$$

where, k: the rate constant of deterioration, k₀: constant (frequency factor independent of temperature), E_a: activation energy (cal/mol), R: ideal gas constant (1.986 cal/mol K), T: absolute temperature (K = °C+273).

After calculations are carried out until we get the k value for each temperature, then the shelf life of the product is further calculated. The calculation of shelf life with the Arrhenius model can use the equations of order 0 and order 1 [6]. The following is the description of the formula for each order:

$$t = (A_0 - A_t)/k \tag{2}$$

$$t = (\ln A_0 / \ln A_t) / k \tag{3}$$

where, t: storage time (days), A_0 : initial quality value, A_t : critical (final) quality value, K: rate of deterioration (% per day)

3. Results and discussion

Organic waste such as leaves and fruit peels are generally just thrown away, so that they often become pollution and pollute the environment. In fact, the reuse of organic waste such as waru leaves and lime peels can produce hand sanitizers that actively kill staphylococcus aureus and eschericia coli bacteria. This is reinforced by looking at the active components in waru leaves, such as saponins, flavonoids, polyphenols, and tannins [7]. These ingredients work as antimicrobial agents against Salmonella partyphi, Staphylococcus aureus, and Staphylococcus epidermis amon other Gram-positive and Gram-negative bacteria. In addition, waru leaf extract was able to reduce Eschericia coli contamination with a percentage reduction of 76.12% [8]. Waru leaf extract is the main component used in making waru leaf gel hand sanitizer.

This hand sanitizer is composed of waru leaf extract and lime peel extract as active substances. The filtrate produced from lime peel extract is solid green and has a sharp ethanol and lime scent. This is because ethanol solvents can dissolve pigments in the form of green color from lime peels. Furthermore, the resulting waru leaf extract is solid brown with a characteristic waru odor. The brown color of waru leaf extract is caused by the drying process so that the green color of chlorophyll in the leaves is oxidized [9]. The concentration of waru leaf extract can be reduced by centrifugation so that dissolved particles in the extract can be removed. Waru leaf gel hand sanitizer is then stored using delkchoice brand aluminum foil packaging. Aluminum foil packaging was chosen as the primary packaging material for gel hand sanitizer products because it has a good chemical composition, is resistant to fats and oils, does not cause chemical reactions to products, has good strength, and hygenis is not easy to make the growth of bacteria and mold [10].

Waru leaf gel hand sanitizer packaged using aluminum foil packaging changes in quality during storage at temperatures of 30°C, 35°C, and 40°C. Quality changes that occur include sensory changes (texture, scent, color, and absorption rate), as well as changes in the degree of acidity (pH). Even so, the stored gel preparation has not experienced signs of damage. Signs of damage to the gel hand sanitizer visually based on the appearance of black spots and the appearance of mold on the preparation.

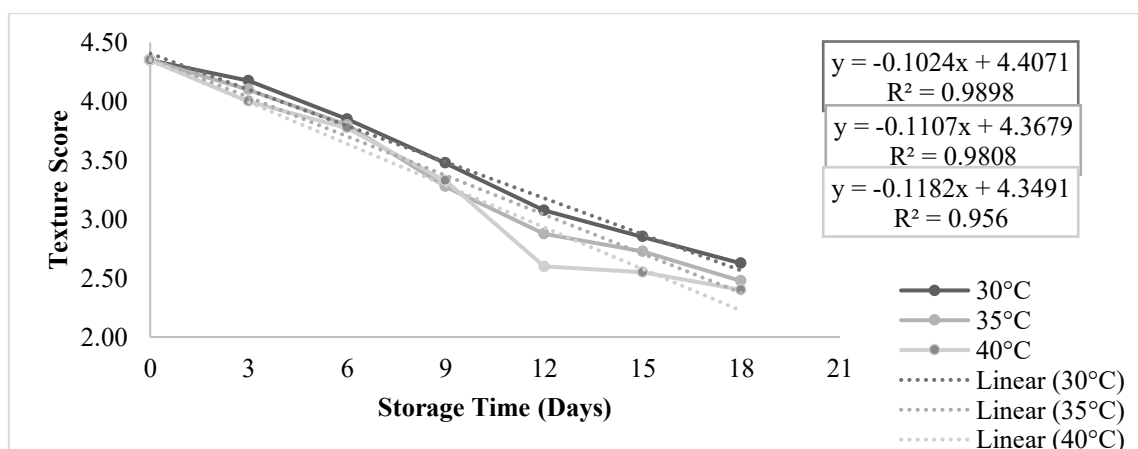


Figure 1. Graph of the relationship between storage time and the texture value of waru leaf gel hand sanitizer in aluminum foil packaging at temperatures of 30°C, 35°C, and 40°C.

During storage, the hand sanitizer texture decreases (Figure 1). According to the texture of gel hand sanitizer is influenced by the addition of CMC-Na. The use of CMC-Na on the day of manufacture forms a good consistency. CMC-Na as a gelling agent when water is added, Na^+ from the bonds of CMC-Na compounds are released and replaced with H^+ ions to form CMCH which increases viscosity well [11]. The decrease in the texture of waru leaf gel hand sanitizer is in accordance with the research, where the cause of the decrease in gel viscosity or dilution is due to temperature and storage time which is getting longer and further reducing viscosity [12]. Due to air-induced oxidation, which occurs when CMC breaks down the carboxyl group, oxygen molecules in the air can damage the colloidal dispersion system of CMC during storage, and that is why its viscosity is reduced. In addition, changes in the texture of the gel hand sanitizer can also occur due to the process of syneresis. Syneresis is the process by which water is released from the gel as it starts to wrinkle and squeeze out the trapped liquid from within [13].

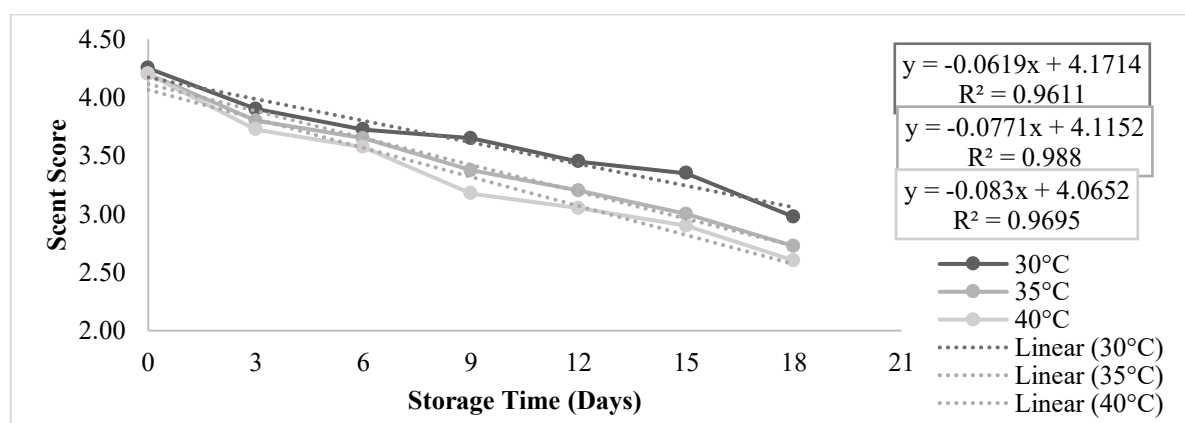


Figure 2. Graph of the relationship between storage time and the scent value of waru leaf gel hand sanitizer in aluminum foil packaging at temperatures of 30°C, 35°C, and 40°C.

During storage, the scent of hand sanitizer decreases (Figure 2). The very distinctive scent of the ingredients at the beginning of hand sanitizer storage comes from lime peel extract which has a distinctive fresh scent. The scent of gel hand sanitizer is influenced by the strong consistency of the gel. When the viscosity of the gel gets higher, many scents will be trapped in the gel [14]. The relationship between storage time and storage temperature with the scent of waru leaf gel hand sanitizer shows that the scent decreases.

The decrease becomes a slight or missing citrus smell and the smell of alcohol is slightly pungent and very pungent. This is because compounds from essential oils are volatile and are derivative compounds from the terpene group which tend to be unstable [15]. In addition, the pungent scent of alcohol obtained is suspected of hydrolysis of ester compounds derived from waru leaf extract and lime peel extract added to the preparation. Hydrolysis of ester compounds that occur will form new compounds in the form of carboxylic acids and alcohols in preparations [16]

During storage, the color of the hand sanitizer decreases (Figure 3). The color of gel hand sanitizer waru leaf extract is formed from a mixture of color waru leaf extract and lime peel extract. The lime peel extract obtained is liquid with a dense green color. Meanwhile, the waru leaf extract obtained is in the form of a viscous liquid with a brown color. The relationship between storage duration and storage temperature with the color of the gel hand sanitizer preparation of waru leaf extract shows that the color of the preparation will look more intense brown and slightly cloudy.

The results of this study support the hypothesis that the presence of tannin components in the extract causes the preparation to appear slightly more turbid during storage [17]. In addition, the higher turbidity value of the product could be due to the higher storage temperature. The formation of a solid brown color in preparations is also thought to be influenced by the enzyme polyphenol oxidase with oxygen which causes oxidation [18].

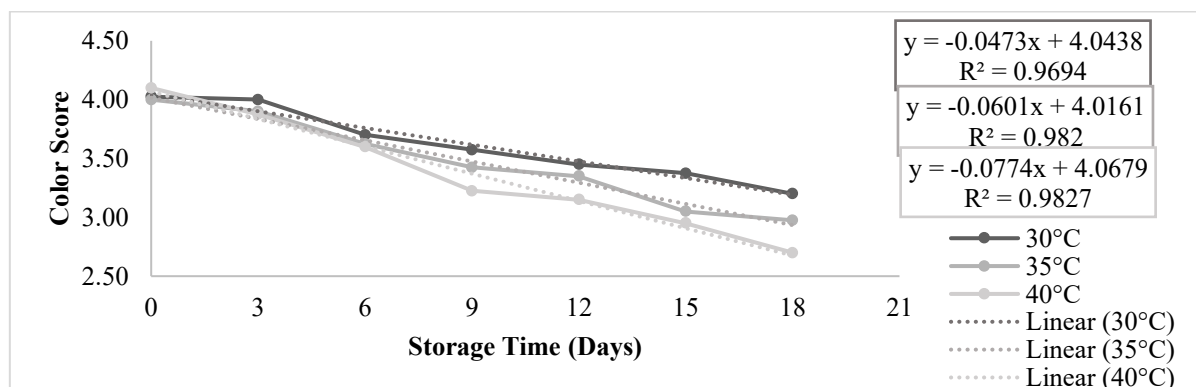


Figure 3. Graph of the relationship between storage time and the color value of waru leaf gel hand sanitizer in aluminum foil packaging at temperatures of 30°C, 35°C, and 40°C.

The longer storage, can make the enzyme is able to convert phenol compounds into quinones and polymerize into brown melanoidin pigments. The presence of phenol compounds in waru leaf extract and lime peel extract added to hand sanitizers can cause enzymatic reactions continuously and the resulting color is increasingly dark brown. Even so, the intense brown color obtained is able to form a defense against pathogens.

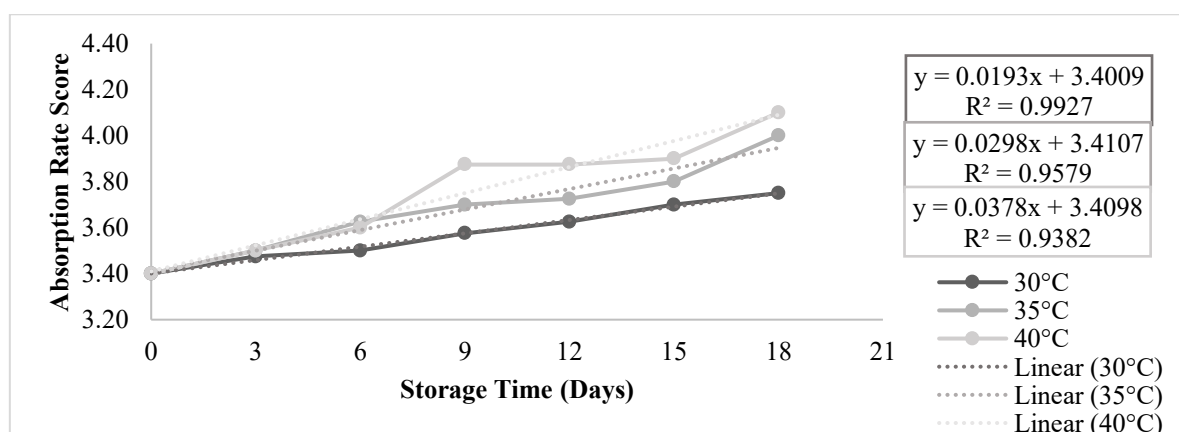


Figure 4. Graph of the relationship between storage time and the absorption rate of waru leaf gel hand sanitizer in aluminum foil packaging at temperatures of 30°C, 35°C, and 40°C.

During storage, the absorption rate of hand sanitizer increases (Figure 4). Absorption rate testing was carried out to determine the level of acceptance of panelists on the ability of waru leaf gel hand sanitizer to absorb on the skin. Each panelist has a different drying time speed because each panelist's hands have different humidity, some are moist and dry. At room temperature 30°C there is a less drastic increase in spreading ability. However, at 35°C and 40°C there is a drastic increase in spreading ability. This is because at high temperatures the polymer bond in the gel will break and cause the preparation to be thinner and easier to spread [19].

The relationship between storage time and absorption shows that the longer the storage period, the increase in absorption and spreadability. Absorbency is the ability of the preparation to absorb on the skin. Meanwhile, dispersion is the ability of the preparation to spread on the skin. Absorbency and dispersion are interrelated. Viscosity is related to the dispersion of a preparation. The thicker the gel produced, there will be a decrease in dispersion while the thinner the gel produced, there will be an increase in dispersion. The greater the dispersion of a preparation, the faster it will penetrate or absorb on the skin [20]. The resulting preparation meets the expected specifications, which is easy to pour and

drip on the palm. In addition, after dripping on the palm of the hand, it will immediately dissolve at body temperature.

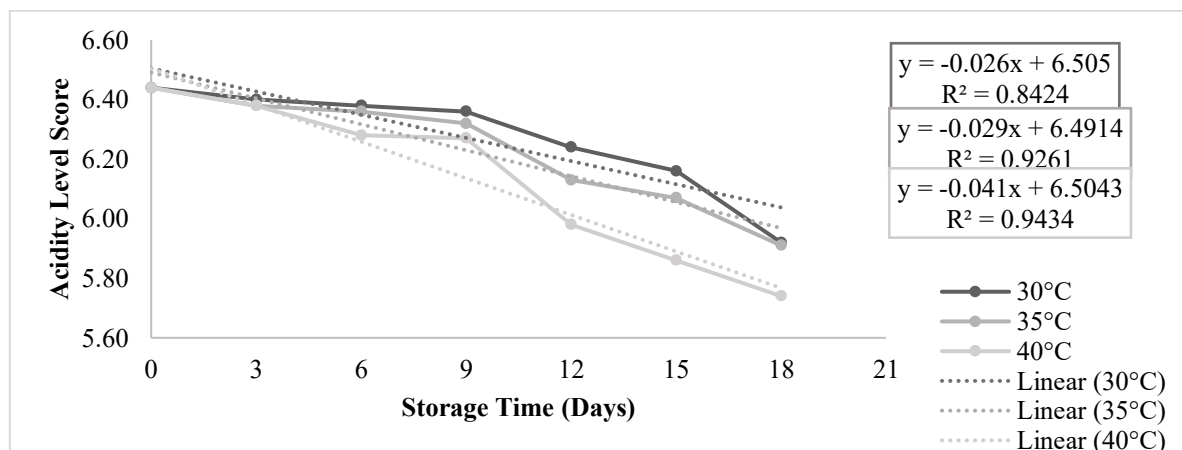


Figure 5. Graph of the relationship between storage time and pH (acidity level) of waru leaf gel hand sanitizer in aluminum foil packaging at temperatures of 30°C, 35°C, and 40°C.

During storage, the pH of hand sanitizer decreases (Figure 5). pH testing is one of the physical indicators to determine the stability of a substance during the storage period [21]. The pH value of waru leaf gel hand sanitizer decreased as shown by the relationship between temperature and storage time. Even so, the pH reduction is not significant and is still in accordance with the pH range of hand sanitizers based on SNI 06-2588-1992 around 4.5-8.0. The results of this study are an accordance that during storage, pH decreases due to a decrease in the binding power of CMC. This decrease in binding power is due to the decomposition of CMC-Na in the carboxylate group, thereby increasing the acidity of the preparation [22]. In addition, a decrease in pH can be caused by evaporation of the extract components and the acid content in lime peel extract also affects the pH value of the preparation.

After obtaining information on the use of reaction orders and key parameters, it can be calculated the shelf life at each temperature. The determination of the waru leaf gel hand sanitizer's shelf life while it is kept in aluminum foil packaging at 30° C, 35° C, and 40° C can be seen in Table 1.

Table 1. Shelf life of waru leaf gel hand sanitizer based on order 1 on quality deterioration parameters at various storage temperatures (30°C, 35°C, dan 40°C).

Temperature	Shelf Life of Waru Leaf Gel Hand Sanitizer				
	pH (Day)	Absorption Rate (Day)	Scent (Day)	Texture (Day)	Color (Day)
30°C	264	156	37	56	108
35°C	209	113	31	51	81
40°C	166	83	26	46	62

After that, there is also irritation testing to determine the safety level of the product when used by panelists. Based on the results of the irritation test, hand sanitizer gel waru leaf extract dripped on the palms of 20 panelists for 5 minutes showed negative results from the observed reaction parameters or did not cause skin irritation. Therefore, it can be concluded that waru leaf gel hand sanitizer is safe to use on the skin.

4. Conclusion

Recycling organic waste in the form of waru leaves and lime peels can produce natural hand sanitizers with novelty or useful findings. The discovery is able to provide added value and reduce organic waste into useful products. In previous studies, it has been known that this hand sanitizer is able to provide

inhibition against staphylococcus aureus bacteria by 16.18 mm with a large inhibition. The use of this natural hand sanitizer is able to compete with other synthetic hand sanitizers.

Shelf-life estimation was then carried out to ensure the safety and durability of this natural product within a certain period of time. The shelf life of waru leaf gel hand sanitizer was discovered by research employing the Accelerated Shelf-Life Testing (ASLT) method of the Arrhenius model. This method determined color characteristics and one-order reactions. The average correlation coefficient at first order (R²) of 0.9591, which is higher than the zero-order R² of 0.9587, indicates the first-order determination is used for calculation. Determination of the color parameter as a key parameter is based on the largest correlation value (R²) compared to other quality parameters. The color parameter at first order has an average slope (k) of -0.0178; an average intercept (b) of 14031; and an average correlation coefficient of 0.9812. Waru leaf hand sanitizer has 108.48 days (3.17 months) shelf life at 30°C. The temperature used in the results of determining the shelf life adjusts to the room temperature in Indonesia (30°C).

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