# Analysis of Sodium Cyclamate on Cork Egg Cake Circulating in Ir Soekarno Sukoharjo Market

# (Analisis Natrium Siklamat pada Kue Telur Gabus yang Beredar di Pasar Ir Soekarno Sukoharjo)

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**Abstract:** Cork egg cake is a traditional cake famous for its sweet taste. Sodium cyclamate is a sweetener that is added to snacks, one of which is cork egg cake. Excess sodium cyclamates can trigger cancer development. The Head of the National Food and Drug Agency of the Republic of Indonesia issued regulation number 4 of 2014 concerning the maximum limit for the use of food additives. The sweetener in the snack food category was 250 mg/kg. This study was conducted to determine the level of sodium cyclamate in cork egg cakes circulating in Ir. Soekarno Sukoharjo market. The type of research was quantitative with a descriptive research design. The method used in this research was qualitative analysis using the precipitation method and quantitative analysis using UV-Vis spectrophotometry. Qualitative analysis showed that samples B and C were positive for sodium cyclamate. The results of the quantitative analysis showed that the average levels of sodium cyclamate in cork egg cakes B and C were 1.02 mg/kg and 1.03 mg/kg. The concentrations of the two samples were still below the limit requirements of BPOM No. 4 in 2014.

Keywords: Cork egg cake, sodium cyclamate, sukoharjo

**Abstrak:** Kue telur gabus merupakan kue tradisional yang terkenal dengan rasa yang manis. Natrium siklamat merupakan bahan pemanis yang ditambahkan dalam makanan ringan salah satunya adalah kue telur gabus. Natrium siklamat yang digunakan secara berlebihan tanpa diimbangi asupan gizi lainnya dapat memicu kanker. Kepala badan pengawas obat dan makanan Republik Indonesia membuat aturan nomor 4 tahun 2014 tentang batas maksimum penggunaan bahan tambahan pangan. Pemanis kategori pangan dalam makanan ringan adalah 250 mg/kg. Penelitian ini dilakukan bertujuan untuk mengetahui kadar natrium siklamat pada kue telur gabus yang Beredar di Pasar Ir Soekarno Sukoharjo. Jenis penelitian ini adalah kuantitatif dengan rancangan penelitian deskriptif. Metode yang digunakan dalam metode spektrofotometri UV-Vis. Hasil analisa kualitatif menunjukkan sampel B dan C positif natrium siklamat. Kemudian hasil analisa kuantitatif menunjukkan rata rata kadar natrium siklamat dalam kue telur gabus B dan C yaitu 1,02 mg/kg dan 1,03 mg/kg. Kadar kedua sampel tersebut masih berada dibawah batas persyaratan Perka BPOM Nomor 4 Tahun 2014.

Kata kunci: Kue telur gabus, natrium siklamat, sukoharjo

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### **INTRODUCTION**

FOOD is anything that comes from biological sources of an agricultural, plantation, forestry, fishery, livestock, aquatic, and water products, both processed and unprocessed, which is intended as food and drink for human consumption, including food additives, food raw materials, and raw materials. other materials used in the process of preparing, processing, or making food and beverages<sup>(1)</sup>.

Food that has been processed and ready to be eaten, while snacks are food that has been processed in such a way that it can be directly served to consumers and this activity can be carried out at the place of business or outside the place of business<sup>(2,3)</sup>. And in general, producers use food additives which include sweeteners, preservatives, dyes, emulsifiers, bleaches, stabilizers, flavors, and aromas, as well as antioxidants<sup>(4)</sup>.

In Indonesia, the use of food additives, both types, and amounts, is regulated in the Regulation of the Minister of Health of the Republic of Indonesia Number 033 of 2012 concerning food additives<sup>(5)</sup>. Food Additives are additives that may or may not have nutritional value, which are intentionally added to food for technological purposes in the manufacture, processing, treatment, packing, packaging, storage, or transportation of food to produce or affect the properties of the food either directly or indirectly. indirect. One of the most popular BTP (food additives) among the public is artificial sweeteners, which are often added to food or beverages to give a sweet taste when consumed<sup>(6)</sup>.

At first, artificial sweeteners were produced for commercial purposes to meet the availability of food and beverage products for people with diabetes (diabetes) or people who need low-calorie foods. According to the Regulation of the Minister of Health of the Republic of Indonesia Number 033 of 2012 concerning food additives, artificial sweeteners are food additives that can cause a sweet taste in food, which have no or almost no nutritional value. One of the allowed artificial sweeteners is cyclamate<sup>(7)</sup>.

Sodium cyclamate is often used in food and beverage products to give it a sweet taste. The sweet taste of nation cyclamate does not produce energy in the body and is easily soluble in water with a sweetness intensity of  $\pm 30$  times the sweetness of sucrose or cane sugar. In the food industry, sodium cyclamate is used as a non-nutritive sweetener as a substitute for sucrose or cane sugar. Sodium cyclamate is heat resistant, so it is often used in foods that are processed at high temperatures<sup>(8)</sup>. Sodium cyclamate was first discovered accidentally by Michael Sveda in 1937. Sodium cyclamate is approved for use in food and beverages in more than 100 countries around the world, including Canada, Australia, Mexico, and Indonesia<sup>(9)</sup>. In addition, according to the annual report of the POM Agency, a test has been carried out on snack foods taken from elementary schools in 30 cities in Indonesia, the results are that from 3925 samples, 421 samples containing sodium cyclamate exceeded the predetermined threshold. This excess artificial sweetener is feared to harm health<sup>(10)</sup>.

According to the Food and Drug Supervisory Agency of the Republic of Indonesia No. 4 of 2014 concerning the Maximum Limit for the Use of Sweetener Food Additives, the maximum limit of Sodium Cyclamate in the food category in snacks is 250 mg/kg<sup>(11)</sup>. Consuming sodium cyclamate in excessive doses and without being balanced with other nutritional intakes can cause sore throats, coughs, migraines and headaches, memory loss, confusion, insomnia, irritation, asthma, hypertension, diarrhea, stomach pain, allergies, impotence, and disorders. sexual intercourse, baldness, brain cancer, bladder cancer<sup>(12,13)</sup>.

The cork egg cake is a traditional Indonesian cake in the form of small, long puffed, golden yellow and colorful, smooth texture, not broken, some have a savory taste and some have a sweet taste. This cork egg cake is famous for its sweet taste and is usually sold in kilos and has a fairly affordable price until now the cork egg cake is still popular and favored by the general public. In Indonesia, cork egg cake is usually served on certain celebrations and holidays such as Eid<sup>(14)</sup>. The cork egg cake is one of the cakes sold at Ir. Soekarno Sukoharjo Market with many enthusiasts, but until now there is no information regarding the content of food additives, especially sodium cyclamate, whether it is following the requirements or not. Based on this background, research was conducted on the analysis of sodium cyclamate levels in cork egg cakes circulating in the Ir. Soekarno Sukoharjo market. This study was conducted to determine the sodium cyclamate content in cork egg cake.

# MATERIALS AND METHODS

**MATERIALS.** The materials used in this study were cork egg cake samples, sodium cyclamate raw, aqua dest, HCl 10% p.a.,  $BaCl_2$  10% p.a.,  $NaNO_2$  10% p.a., ethyl acetate 96% p.a., cyclohexane p.a.,  $H_2SO_4$  30% p.a., 1% hypochlorite, concentrated  $H_2SO_4$  p.a., NaOH 10N p.a.

**Tools.** The tools used in this study were UV-Vis spectrophotometer, analytical balance, 10mL volumetric flask, 50mL volumetric flask, 100mLvolumetric flask, micropipette, stirring rod, test tube, cup, test tube clamp, glass funnel, separating funnel, beaker 50 mL, volumetric pipette, water bath, filter paper, Erlenmeyer flask, cuvette, watch glass, mortar.

**METHOD.** This research is descriptive research with a total sampling technique. Ir. Soekarno market is one of the traditional markets in Sukoharjo, between cake shops in Ir. Soekarno, only 3 shops sell cork egg cake, so the samples used in this study were 3 samples. Sample preparation was carried out with a sample of cork egg cake first mashed using a mortar and dissolved with distilled water and then homogenized, then filtered with filter paper. The filtrate solution was then used for qualitative and quantitative analysis.

**Qualitative Analysis of Cyclamate using Precipitation Method**<sup>(15)</sup>. Pipette 100 mL of each sample solution into an Erlenmeyer flask and add distilled water to the mark. Then filtered with 15cm x 15cm Whatman paper, then 10 mL of 10% HCl solution was added, and 10 mL of 10% BaCl2 solution was left for 30 minutes. Filtered using Whatman paper measuring 15cm x 15cm, 10mL of 10% NaNO2 solution was added again in an acid chamber. Heated on a hotplate or water bath at a temperature of around 125-130°C. The results obtained about 20-30 minutes after heating are if there is a white precipitate, it means the sample contains sodium cyclamate.

Quantitative Analysis of Cyclamate Levels using the UV-Vis spectrophotometric method<sup>(16)</sup>. Test solution is prepared by weighing a sample of 10 grams of sample dissolved with aquadest then taken as much as 50 mL and put in a separating funnel to I, add 2.5 mL of concentrated sulphuric acid. Cool, after cooling adds 50 mL of ethyl acetate, shake for 2 minutes. Separate the ethyl acetate layer and take 40 mL of the clear part, then put it into the second separating funnel. Shaken 3 times each time with 15 mL of distilled water, shaken and separated by layers of water. put into a separator funnel III, add 1 mL of 10 N NaOH, 5 mL of cyclohexane, and shake for 1 minute. Shake and separate the aqueous layer then put into the separator funnel to IV, add 2.5 mL of 30% sulphuric acid, 5 mL of cyclohexane, 5 mL of hydrochloride solution containing 1% free chlorine, shake again for 2 minutes. The cyclohexane layer will be greenish-yellow, if it is colorless, about 5 mL of hydrochloride solution is added. The water layer was removed, the cyclohexane layer was added to 25 mL of distilled water, shaken and separated, the bottom layer was taken and put into a 10 mL volumetric flask.

Blank solution is prepared by pipetting 50 mL of distilled water into the first separator funnel and add 2.5 mL of concentrated sulphuric acid. Cool, after cooling adds 50 mL of ethyl acetate, shake for 2 minutes. Separate the ethyl acetate layer and take 40 mL of the clear part, then put it into the second separating funnel. Shaken 3 times each time with 15 mL of distilled water, shaken and separated by layers of water. put into a separator funnel III, add 1 mL of 10 N NaOH, 5 mL of cyclohexane, and shake for 1 minute. Shake and separate the aqueous layer then put into the separator funnel to IV, add 2.5 mL of 30% sulphuric acid, 5 mL of cyclohexane, 5 mL of hydrochloride solution containing 1% free chlorine, shake again for 2 minutes. The cyclohexane layer will be greenish-yellow, if it is colorless, about 5 mL of hydrochloride solution is added. The water layer was removed, the cyclohexane layer was added to 25 mL of distilled water, shaken and separated, the bottom layer was taken and put into a 10 mL volumetric flask.

Determination of wavelength is conducted as follows: Weighing 100 mg of standard sodium cyclamate was put in a 10 mL volumetric flask and added with distilled water up to the limit mark so that the concentration of the standard mother liquor was obtained at 10,000 ppm. The standard mother liquor was diluted to a concentration of 1400 ppm by pipetting 1.4 mL into a 10 mL volumetric flask and added with distilled water up to the mark. The standard mother liquor is put in a separating funnel to I, add 2.5 mL of concentrated sulphuric acid. Cool, after cooling adds 50 mL of ethyl acetate, shake for 2 minutes. Separate the ethyl acetate layer and take 40 mL of the clear part, then put it into the second separating funnel. Shaken 3 times each time with 15 mL of distilled water, shaken and separated by layers of water. put into a separator funnel III, add 1 mL of 10 N NaOH, 5 mL of cyclohexane, and shake for 1 minute. Shake and separate the aqueous layer then put into the separator funnel to IV, add 2.5 mL of 30% sulphuric acid, 5 mL of cyclohexane, 5 mL of hydrochloride solution containing 1% free chlorine, shake again for 2 minutes. The cyclohexane layer will be greenish-yellow, if it is colorless, about 5 mL of hydrochloride solution is added. The water layer was removed, the cyclohexane layer was added 25 mL of distilled water, shaken, and separated, the bottom layer was read for absorbance at a wavelength of 200-400 nm.

Calibration curve is prepared from five 10 mL volumetric flasks were each filled with a standard solution of 10,000 ppm with a concentration of 1000; 1200; 1400; 1600; 1800 ppm was diluted with distilled water to the mark and treated the same as the test

solution, starting from the standard, it was put into the first separating funnel, then 1 mL of 10 N NaOH, 5 mL of cyclohexane were added and shaken for 1 minute. The water layer was separated and put into a separatory funnel II, added 2.5 mL of 30% sulfuric acid, 5 mL of cyclohexane, 5 mL of hypochlorite solution, and shaken for 2 minutes. The cyclohexane layer will be greenish-yellow, if it is colorless, about 5 mL of sodium hypochlorite is added. The water layer was removed, the cyclohexane layer was added to 25 mL of distilled water and then shaken, then separated and the bottom layer was taken.

The bottom layer obtained from the test solution is measured for absorbance at the maximum wavelength that has been obtained. The data obtained from the results of research in the laboratory were analyzed descriptively in the form of tables and graphs.

## **RESULTS AND DISSCUSION**

The sample of cork egg cake obtained from the Ir. Soekarno there are 3 samples, namely sample A, sample B, sample C. The samples obtained were then identified qualitatively with a sodium cyclamate test. **Table 1. Results of qualitative analysis of sodium** cyclamate precipitation method.

Sample	Standard	Results	Description		
Raw	White	A white	Positive (+)		
Sodium	precipitate	precipitate			
Cyclamate		is formed			
Α	White	No white	Negative (-)		
	precipitate	precipitate			
		formed			
В	White	A white	Positive (+)		
	precipitate	precipitate is formed			
С	White	A white	Positive (+)		
	precipitate	precipitate is formed			

Based on Table 1. above, it can be seen that the 2 samples tested (B and C) were positive for sodium cyclamate which was marked by the formation of a white precipitate.

Positive samples containing artificial sweetener sodium cyclamate, then analyzed quantitatively using UV-Visible spectrophotometry. The absorbance value of the cyclamate standard solution quantitatively can be seen in the following table.

 Table 2. Absorbance value of sodium cyclamate standard solution maximum wavelength of 288nm.

Concentration	Absorbance	
1000 ppm	0,23	
1200 ppm	0,31	
1400 ppm	0,41	
1600 ppm	0,53	
1800 ppm	0,62	

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The absorbance value of the sodium cyclamate standard solution above can be used to determine the linearity test with the correlation (r) in the linear regression equation y = ax + b. The graph of the calibration curve and the linear regression equation can be seen in Figure 1.

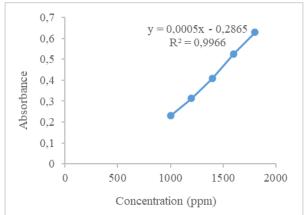


Figure 1. Sodium cyclamate standard solution calibration curve.

Table 3. Sodium cyclamate levels in cork egg cake based on uv-visible spectrophotometer.

on uv-visible spectrophotometer.					
Sample	Replicat ion	Absor- bance	Content (mg/kg)	Average content (mg/kg)	
В	1 2 3	0,22 0,22 0,22	1,02 1,02 1,02	1,02	
С	1 2 3	0,22 0,23 0,23 0,23	1,02 1,03 1,04 1,04	1,03	

The absorbance obtained from each sample was then entered into a linear regression equation (y = 0.0005x - 0.2865) to obtain the sample concentration (x). The average level of sodium cyclamate in the sample is 1.019 mg/kg, respectively, for sample B and 1.033 mg/kg for sample A.

In everyday life, the use of food additives in food and beverages is often found, one of which is a sweetener. Sweeteners are often added to food or beverage products to enhance taste and aroma. The sweetener that is often added to food and beverage products is sodium cyclamate.

This research was conducted to determine the level of sodium cyclamate present in the cork egg cake, samples were taken from different stores whose sources came from their production. The samples used were given sample codes A, B, and C. Before determining the levels, a qualitative test was carried out with a precipitation method that could identify the presence of sodium cyclamate in each sample. The precipitation method is one of the basic methods used to test sodium cyclamate as a food additive according to SNI 01-2893-1992. The principle of the presence of sodium cyclamate in the sample is by precipitation or is called a precipitation reaction<sup>(4)</sup>. A reaction can be said to be a precipitation reaction if the reaction produces a precipitate. Precipitation is done by adding barium chloride in an acid atmosphere and then adding sodium nitrite to form a barium sulfate precipitate. For the precipitation test, positive control was made as a comparison which was used to determine the chemical reaction in the sample if it was detected to contain sodium cyclamate<sup>(17,18)</sup>.

Pure sodium cyclamate is used as a comparison solution or positive control which aims to compare the results of testing positive samples containing sodium cyclamate. In making a positive control solution, 10% HCl was added which aims to create a strongly acidic atmosphere so that the reaction can take place quickly, then 10% NaCl was added and then left for 30 minutes, 10% BaCl, as a precipitation agent which seeks to facilitate the analysis of sodium cyclamate additives in the solution. The sample can then be continued with the addition of 10% NaNO, and heating produces a very soft white precipitate of BaSO, which indicates positive sodium cyclamate. In addition, 10% NaNO<sub>2</sub> acts as a reagent to form a white precipitate of BaSO<sub>4</sub> which indicates the presence of sodium cyclamate<sup>(4,19)</sup>.

Qualitative test with the deposition method on each sample given the same treatment, starting with dissolving the sample with 100 mL of distilled water, then adding 10 mL of 10% HCl so that the solution is in an acidic state so that the reaction can react easily and added 10 mL of 10% BaCl, which serves to precipitate impurities in the solution and then left for 30 minutes so that the chemical reaction process occurs between the sample and BaCl, in an acid atmosphere. After being left for 30 minutes filtered and added 10 mL of 10% NaNO, which aims to break the sulfate bond in sodium cyclamate. When the sulfate bond has been broken, the Ba<sup>2+</sup> ion will react with the sulfate ion and produce a precipitate of barium sulfate (BaSO<sub>4</sub>) so that the nitrogen gas produced from the reaction can be identified with a pungent odor when the test process is then heated for 20-30 minutes which aims to separate the precipitate. of liquid. then allowed to stand until there is a white precipitate which indicates the presence of cyclamate. The barium sulfate precipitate obtained is the same as the amount of cyclamate present, because in the mechanism the sodium cyclamate reacts the same as the barium sulfate obtained, in other words, 1 mole of cyclamate is equal to 1 mole of barium sulfate<sup>(18,20)</sup>.

Based on the results of the precipitation test on the three samples A, B, and C, positive results were obtained to form a white precipitate, namely samples B and C. After qualitative testing was carried out, quantitative analysis was then carried out on samples that were positive for sodium cyclamate which aimed to determine the amount of concentration in the sample.

To test the concentration in the sample, it begins by making a blank solution which is extracted using a separating funnel. In making a blank solution, it functions as a correction factor for the solvent and reagents used, in the blank measurement the absorbance value obtained must be zero because what is measured is the absorption of the solvent and reagents. Next, make a standard solution of sodium cyclamate for the determination of the maximum wavelength, Aims to determine the maximum absorption area produced in the form of absorbance values. The standard sodium cyclamate was dissolved in aqua dest with a concentration of 10,000 ppm then diluted to a concentration of 1400 ppm then the standard solution was put in the first separating funnel and concentrated sulfuric acid was added. In the addition of concentrated sulfuric acid which serves to acidify the sample, cool it after it has cooled, add ethyl acetate to extract sodium cyclamate in the sample which is then shaken for 2 minutes, then the bottom layer is removed, the ethyl acetate layer is put in a second separating funnel and shaken three times, each time. with 15 mL of distilled water to dissolve and extract sodium cyclamate, then shaken and separated. The water layer is accommodated and put in the third separatory funnel and NaOH solution is added to provide an alkaline atmosphere to the sodium cyclamate, add the cyclohexane solution, shaken for 1 minute to extract the sodium cyclamate, shake and separate the water layer, and then put in the fourth separating funnel. A 30% sulfuric acid solution, a cyclohexane solution, and a hypochlorite solution containing 1% free chlorine were added to remove the water present in the solution and shaken again for 2 minutes. Separate on the top layer will be greenishyellow if colorless if added again a hypochlorite solution containing 1% free chlorine. The water layer was removed, aquadest was added to the cyclohexane layer, then shaken and separated the bottom layer to read the absorbance at a wavelength of 200-400 nm. Based on the scan results, the maximum wavelength is 288 nm at an absorbance of 0.319 because that wavelength is the highest. These results are still in the range of the optimum absorption area for sodium cyclamate, which is 220-300 nm<sup>(19)</sup>.

Sodium cyclamate calibration curve was obtained by measuring the absorbance of standard solutions with concentrations of 1000 ppm, 1200 ppm, 1400 ppm, 1800 ppm at a wavelength of 288 nm. In the process of making the calibration curve, they were given the same treatment by determining the wavelength and making the test solution by extracting using a separating funnel from each concentration series. From this measurement, the regression equation is obtained, namely y = 0.0005x - 0.2865with a correlation coefficient value of  $r^2 = 0.9966$ indicating the linearity of the standard curve value, the closer the correlation coefficient is to 1, the more linear the equation is then used to calculate the sample concentration.

Furthermore, the determination of sodium cyclamate levels in the sample was carried out three times with the same treatment in the manufacturing process, namely by extracting the sample using a separating funnel and then measuring it at a maximum wavelength of 288 nm. Then the concentration of sodium cyclamate in the sample can be calculated using a calibration curve with the regression equation y = bx + a. Based on the measurement results, samples B and C samples analyzed still meet the requirements for cyclamate levels listed in the Regulation of the Head of the Food and Drug Supervisory Agency No. 4 of 2014 concerning the maximum limit for the use of sweetener additives, which is 250 mg/kg. The levels obtained from the results of the study for sample B were 1.019 mg/kg while sample C was 1.033 mg/kg. The results of these levels are still below the threshold determined by the Regulation of the Head of BPOM No. 4 the Year  $2014^{(11)}$ .

Another study conducted by Hadiana<sup>(8)</sup> showed that from 2 samples consisting of cakes, cakes, and ice cubes, one of them was suspected to contain sodium cyclamate, which was 218.75 mg/kg, while the ice cubes were negative for sodium cyclamate. Although the levels do not exceed the limit and are still safe for consumption, artificial sweeteners, sodium cyclamate, need to be wary of because in excessive doses it can cause side effects that can harm human health. If the consumption of sodium cyclamate in excessive doses and without balanced with another nutritional intake can cause sore throat, cough, migraine and headache, memory loss, confusion, insomnia, irritation, asthma, hypertension, diarrhea, stomach pain, allergies, impotence, and sexual disorders, baldness, brain cancer, bladder cancer<sup>(9,12)</sup>.

## CONCLUSION

Based on the results of the research that has been carried out, it can be concluded: The results of the analysis of sodium cyclamate levels from three samples of cork egg cake there were 2 positives for sodium cyclamate, namely in sample B and sample C. For sample B 1,02 mg/kg and sample C 1,03 mg/kg. Sodium cyclamate levels in the analyzed samples of cork egg cake still meet the requirements set by the Regulation of the Head of the Food and Drug Supervisory Agency No. 4 of 2014 concerning the limit for the use of food additives sodium cyclamate, which is 250 mg/kg.

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