# CHAPTER I

# INTRODUCTION

## 1.1 Background

Food safety requires extra attention because it plays a significant role in the body's growth and development. Snacks or nibbles are one of the meals that are frequently becoming a source of food poisoning, as they are generally the results of industrial production that neglects the quality of their processed products.

Beverages are one of the primary needs for humans that must be fulfilled daily. In doing so, manufacturers are competing to create more varied beverage products. This encourages consumers to be more careful in choosing beverage products that are nutritious, good in quality and safe for consumption.

Recently, the presence of energy drink products has increased with a variety of trademarks being offered. The increasing number of energy drink production shows an increasing interest in energy drink consumption in the community.

Energy drinks are beverages that are classified as supplements. This drink is believed to be beneficial as an energy booster and restorer. They contain a source of energy from sugar. Sugar added to energy drinks can be in the form of pure sugar or artificial sweeteners. Besides sweeteners, energy drinks also contain caffeine, guarana, vitamin B, taurine, carnitine, ginkgo biloba, ginseng, ephedrine, keratin, and inositol (Pertiwi, R. 2017).

Energy drinks often use artificial sweeteners instead of sugar. The use of artificial sweeteners is widely used by manufacturers because of the high level of sweetness and economical prices, making it possible for the producers to be more interested in using these artificial sweeteners. In the food industry, there are several kinds of artificial sweeteners, such as saccharin, cyclamate, and aspartame. The use of artificial sweeteners need to be watched out for and should not exceed the limits set by the government. Cyclamate is one of the artificial sweeteners reportedly to be often misused; the usage exceeds the limit in Indonesia.

The use of cyclamate that exceeds the maximum limit can cause health problems. In the short term it can cause very common symptoms such as dizziness, nausea, vomiting, diarrhea, or defecation issues. The effect of long-term use of cyclamate still raises controversy regarding its safety aspect, which is potentially carcinogenic when converted to cyclohexylamine in the digestive tract. Repeated exposure to high doses of these compounds can cause liver and kidney damage.

Cyclamate as a artificial sweetener is generally no longer used in several countries because its degradation products are carcinogenic. However, in Indonesia the use of cyclamate as a artificial sweetener in food is still allowed. According to the Decree of the Head of BPOM No. HK.00.05.1.4547 of 2004, concerning the requirements for the use of artificial sweetener food additives in food products, the limit on the use of cyclamate is determined based on the food category, for the food supplement category it must not exceed 1.25 g/kg of material.

Several test methods on cyclamate that are often used include qualitative analysis using the FeCl3 test, qualitative analysis using the Phenol-Sulfuric Acid test, qualitative analysis using the resorcinol – sulfuric acid test, qualitative analysis using the sodium nitrite test, qualitative analysis using Thin Layer Chromatography (TLC), quantitative analysis using the titration method, quantitative analysis using the colorimetric method, quantitative analysis using the Nitrimetric method, quantitative analysis using the Gravimetric method, quantitative analysis using the general method for non-alcoholic beverages, quantitative analysis using the Phenol-H2SO4 Colorimetric method, UV Spectrophotometry Method and Chromatography Method High Performance Liquid (HPLC) (Depkes RI, 2014).

Based on the above, it is necessary to conduct research to analyze the artificial sweetener Na cyclamate in energy drink products on the market.

### 1.2. Research Questions

Based on the introduction above, the research question in this study is whether the energy drinks circulating in the Medan city market contain cyclamate?

### 1.3. Research Objectives

Based on the formulation of the problem above, this study aims to determine the content of cyclamate in energy drinks circulating in the Medan city market.

### 1.4 Research Benefits

As a scientific reference for the Government, especially the Health Service, to determine the content of cyclamate in energy drinks on the market.

# CHAPTER II

# LITERATURE REVIEW

## 2.1 Food Additives

Food additives in general are materials that are not usually consumed as meal and are usually not a typical component of food, may have or do not have nutritional value, which are intentionally added to food for technological purposes in the manufacture, processing, preparation, treatment, packaging, and storage (Apriyana, 2009). In general, food additives can be divided into two major groups, namely as follows

If we look at its origin, food additives may come from natural sources such as lecithin, citric acid, and so on. This material can also be synthesized from chemicals that have similar properties to β-carotene and ascorbic acid. In general, synthetic materials have advantages, i.e., being more concentrated, more stable, and cheaper, but there are also disadvantages, i.e., process imperfections often occur so that they contain substances that are harmful to health, and sometimes are carcinogenic which can stimulate cancer in animals or human. The usage of food additives can only be justified if they are intended to achieve their respective purposes of use in processing, are not used to conceal the use of improper or non-compliant ingredients, are not used to conceal work methods that are contrary to good food processing method, and are not used to conceal food spoilage.

The use of food additives should be in doses below a predetermined threshold. There are 2 types of food additives, namely GRAS (Generally Recognized as Safe), this substance is safe and does not have a toxic effect, such as sugar (glucose). While the other type, namely ADI (Acceptable Daily Intake), this type is always set daily intake limit to maintain / protect the health of consumers (Cahyadi, 2009).

## 2.2 Artificial Sweeteners

### 2.2.1. Definition of Artificial Sweeteners

Artificial sweeteners are substances that can produce a sweet taste or can aid sharpen the acceptance of the sweet taste, while the calories they produce are much lower than sugar. Generally, artificial sweeteners have different chemical structures from the polyhydric structure of natural sugars. Although many artificial sweeteners have been found, only a few are allowed to be used in foodstuffs. Initially, Na- salt and Ca-cyclamate, which are thirty times as sweet as sucrose, were used as sweeteners. In Indonesia, the use of cyclamate is still permitted, but the result of cyclamate metabolism, namely cyclohexylamine, is a carcinogenic compound, the disposal of cyclohexylamine through urine can stimulate the growth of bladder tumors in rats (Darlina, 2009).

Artificial sweeteners that are now widely used in food and beverages are Ca- salt or Na-saccharin. The use of saccharin depends on the intensity of the desired sweetness. At high concentrations, saccharin will cause a bitter taste. The sweetness of saccharin is four hundred times greater than the sweetness of a 10% sucrose solution (Winarno, 1997).

Sweetener is a food substance that is low in energy, 180 times sweeter than sucrose, which is called aspartame (phenylalanyl-aspartic acid dipeptide). Initially, this dipeptide might cause brain tumors in mice and other laboratory animals, but this is not supported by other observations. The FDA estimates that these sweeteners do not significantly improve the mental retardation seen with undiagnosed phenylketonurics even though consumption is increased to 3 mg phenylalanine/kg/day. As a dipeptide consisting of amino acids normally its consumption in large quantities (in the form of protein) is not expected because the sweetener has been proven to be harmful to humans and animals. As a substitute for saccharin, aspartame has one disadvantage because it loses its sweet taste (due to hydrolysis) when exposed to water or heat, therefore it cannot be used properly in the preparation of a food product (Linder, 1992).

### 2.2.2. Types of Artificial Sweeteners

#### 1. Saccharin

When it was first discovered, saccharin was used as an antiseptic and preservative, but since 1900 it has been used as a sweetener. Saccharin with the formula C7H5NO3S and a molecular weight of 183.18 is synthesized from toluene usually available as the sodium salt. Other names for saccharin are 2,3-dihydro-3-oxobenzylsulfonasol, benzosulfimide or o-sulfobenzene. The intensity of the sweet taste of saccharin sodium salt is quite high, which is approximately 200-700 times sweeter than 10% sucrose (Cahyadi, 2006).

Besides the sweet taste, saccharin also has a bitter taste due to the low purity of the synthesis process. Saccharin is widely used as a sugar substitute because it has stable, non-carcinogenic properties, low calorific value, and relatively cheap price, besides that saccharin is widely used to replace sucrose for people with diabetes mellitus or low-calorie foodstuffs. The use of saccharin is usually mixed with other sweeteners such as cyclamate or aspartame. It is meant to mask the unpleasant taste of saccharin and amplify the sweetness. For example, the combination of saccharin and cyclamate with a ratio of 1:3 is the best mixture as a sweetener that resembles sugar in beverages. Food and beverage products that use saccharin include soft drinks, candies, jams, salad dressings, low-calorie gelatin, and other processed products without sugar. In addition, saccharin is used as an additive in oral health products such as toothpaste and mouthwash (Cahyadi. 2006).

#### 2. Cyclamate

Cyclamate was first discovered by Michael Sveda in 1937. Cyclamate has been added to food and beverages since 1950 (Cahyadi, 2008). In contrast to saccharin's sweetness which has a bitter after-taste , cyclamate only tastes sweet without being followed by a bitter taste. Cyclamate has the formula C6H11NHSO3Na, generally in the form of calcium, potassium, and sodium cyclamate salts. Cyclamate salt is in the form of white crystals, odorless, colorless, and easily soluble in water and ethanol, its sweetness intensity is ± 30 times the sweetness of sucrose. The combination of the use of cyclamate with saccharin is synergistic, and compatible with flavors and as a preservative (Indrie and Qanytah, 2009).

The physical properties of cyclamate are heat resistant, so it is often used in foods that are processed at high temperatures, such as canned food. (Yusuf and Nisman, 2013). Cyclamate has a calorific value of 0 kcal/g or equivalent to 0 kg/g and the ADI for cyclamate is 0mg/kg - 11mg/kg body weight, the maximum limit for the use of cyclamate is based on the jelly, jam, and marmalade food categories. namely 1000 mg/kg (SNI 01.6993.2004).

In calculating the ADI value, the standard weight for the age group is used based on the FAO – WHO standard in the Handbook on Human Nutrition Requirements, namely: - The standard weight of children (0-9 years) is 17 kg - The standard weight of adolescent boys - male (10-19 years old) is 42 kg - The standard weight for adolescent girls (10-19 years) is 41 kg - The standard weight for adult males (20-60 years) is 55 kg - The standard weight for female adults (20-60 years) is 47 kg (Sediaoetomo, 2008).

Despite its use is permitted and has been limited, the use of cyclamate is reported to be frequently abused and its use exceeds the permitted limit. BPOM research in November - December 2002 has shown that cyclamate consumption has reached 240% Acceptable Daily Intake (ADI) (BPOM, 2004). The results of research conducted on energy drinks sold in the city of Medan showed that from the 15 samples examined, 6 samples used cyclamate, with the lowest level of 1.77 g/kg and the highest level of 2.91 g/kg almost approaching the maximum level of cyclamate that is 3 g/kg according to the Regulation of Minister of Health (Permenkes) No.722/Menkes/IX/88 (Sinamo, 2004). The results of a similar study were also carried out on local syrups or national products in traditional markets in Medan City. The cyclamate content in syrup was 129.8 mg/kg, while a ready-to-eat syrup was 18.8 mg/kg (Simatupang, 2009). The results of this second study indicate that the level of use is still far below the threshold used, which is 500mg/kg according to SNI 01-6993-2004 concerning requirements for the use of sweeteners.

The results of other studies were also carried out on carrying herbs at the Grobogan Market, Semarang from the analysis obtained by the seller adding cyclamate to some of the *jamu* (Indonesian herbs beverages) sold. Of the 23 types of positive herbs containing cyclamate, out of a total of 32 types, 16 *jamu* exceeded the threshold, and 7 types of *jamu* were below the threshold (Lestari, 2011). This result is in line with the impact that will be felt on health if it is continuously consumed. Similar research was also conducted on the *jamu* carrying turmeric acid in Jakarta. Quantitative analysis was carried out using paper chromatography and high working liquid chromatography (HPLC), from the results obtained from the 5 samples examined that did not contain artificial sweeteners saccharin and aspartame but all positive samples contained cyclamate (Yusuf and Nisman, 2013).

Based on the studies that have been carried out, it shows that there is still a lot of use of artificial sweeteners in the form of cyclamate in food and beverages, but the levels used are still below the threshold level that has been set, which is 300 mg/kg according to the Minister of Health of the Republic of Indonesia No.722/Menkes/per/ IX/88 while according to WHO itself the safe consumption of cyclamate (ADI) is 11 mg/kg body weight. According to the Ministry of Health of the Republic of Indonesia, its use is only allowed for diabetic patients or people who need low-calorie foods. But in fact, the use of cyclamate is increasingly widespread in various

circles and various products. This is because the cyclmate price is much cheaper, producing a sweet taste without a bitter taste, and thirty times sweeter than sugar (Putra, 2011).

**3. Aspartame**

Aspartame is a methyl ester dipeptide compound, namely L-aspartyl-L-alanine methylester with the formula C14H16N2O5, which has a sweetness of 100-200 times that of sucrose. Aspartame, known by the trade name equal, is one of the food additives that has gone through various in-depth and thorough tests and is safe for people with diabetes mellitus. Since 198,1 it has been allowed to be marketed. In its use in soft drinks, aspartame is less profitable because storage for a long time will result in a decrease in sweetness. In addition, aspartame is not heat-resistant, so it is not suitable for use in foodstuffs that are processed through heating.

Aspartame is composed of amino acids so that the body will experience metabolism similar to processing other amino acids in general. For people with heredity associated with mental retardation (phenyl ketone urea / PKU), it is forbidden to take aspartame because of the presence of phenylalanine which cannot be metabolized by the disease. Excess phenylalanine in the body of PKU sufferers is thought to cause damage to the brain and ultimately lead to mental disability. Referring to the amino acids that make up aspartame, aspartame is not a non-calorie sweetener because like protein, aspartame is metabolized into its constituent amino acids and has an energy of 4 kcal/g. However, because in its use 100 g of sucrose can be replaced with 1 g of aspartame, it can be said that aspartame is a non-calorie sweetener. In addition to safety factors, the use of aspartame for people who suffer from an inherited disease known as phenylketonuria needs special attention. An estimated 1 in 15,000 people have this disorder. People suffering from phenylketonuria are unable to metabolize phenylalanine, one way to treat it is by limiting the intake of phenylalanine, not eliminating it, because phenylalanine is an essential amino acid that is essential for life. Excessive amounts of phenylalanine in patients with phenylketonuria can cause mental retardation because phenyl pyruvic acid formed from phenylalanine will accumulate in the brain (Cahyadi, 2006).

**2.3 Cyclamate**

**2.3.1 Physicochemical Properties**

Cyclamate was first discovered accidentally by Michael Sveda in 1937. Since 1950 cyclamate has been added to food and beverages. Unlike saccharin, cyclamate has a sweet taste without the unpleasant aftertaste. It is easily soluble in water and its sweetness intensity is ±30 times the sweetness of sucrose. Cyclamate is usually available as the sodium salt of cyclamic acid with the molecular formula C6H11NHSO3Na. The combination with saccharin is synergistic. JECFA set the acceptable daily intake (ADI) for cyclamate at 11 mg/kg bw/day. The use of essence syrup is not more than 1000 mg/kg (BSN. 2004).

**2.3.2. Usage**

The main uses of cyclamate include soft drinks, fruit juice drinks, syrups, processed fruit, chewing gum, jellies, jams, and gelatin-based toppings (Surya. 2014).

### 2.3.3 Side Effects

The results of the research that have been carried out have implied that cyclamate is a bladder carcinogen in rats, so cyclamate is removed from the "Generally Safe" list and finally banned from use in food and beverages in the United States (Hunt, 2011).

### 2.3.4 Impact of Cyclamate on Health

Even if its use is permitted and is safe for consumption in small amounts, it will still pose a health hazard within certain limits. The use of this cyclamate is shown for people with diabetes mellitus and people whose diet really helps their health. However, when cyclamate is consumed by normal people, it will cause a problem for their health. Older research shows that cyclamate can cause atrophy, namely the occurrence of testicular shrinkage and chromosome damage, research conducted by experts from the Academy of Science in 1985 reported that cyclamate and its derivatives (cyclohexylamine) are also suspected of being tumor promoters (Cahyadi, 2009).

Several studies have shown that the addition of 10% sodium cyclamate can stimulate the occurrence of bladder tumors (Frank, 1995 in Putra, 2011). The results of human studies in 3 people who had taken cyclamate at a dose of 40-57 mg/kg body weight regularly for 18 months caused tumor growth. Cyclamate undergoes several processes in the human body. The absorption of cyclamate in the body is relatively slow, i.e., ± 6-8 hours. Cyclamate is not completely absorbed through the small intestine, partly out (excretion) with feces about 18-36%. This indicates that cyclamate is not absorbed in the intestine (Sinamo, 2004). Consumption of cyclamate in excessive doses causes bladder cancer, in addition it will cause lung, liver and spleen tumors (Indrie et.al, 2011). In addition, cyclamate causes many health problems including tremors, migraines, and headaches, memory loss, insomnia, irritation, asthma, hypertension, stomach pain, allergies, impotence and sexual disorders, brain cancer (Indriasri, 2009).

# CHAPTER III

# RESEARCH METHOD

## 3.1 Location and Period of Research

This research was conducted in LAB KESDA research laboratory on February-March 2019.

## 3.2 Required Tools

measuring cup, erlemeyer, measuring flask, evaporating dish, pipette, dropper, test tube, beaker glass, spatel, funnel, microscope, stirring rod, analytical balance, object glass, vial, spiritus lamp, platinum wire, filter paper, paper parchment, pycnometer, separating funnel and a set of UV-Vis spectrophotometers.

## 3.3. Required Materials

The ingredients used are energized with various trademarks, raw materials for comparison of sweeteners cyclamate, aquadest, Hydrochloric Acid (Merck), Barium Chloride (Merck), Sodium Nitrite (Merck), Ether (Brataco), Sodium Hydroxide (Merck), Iron (III) Chloride (Merck), resorcinol (Merck), Zinc uranyl acetate (Merck), Acetic Acid (Brataco), Sulfuric Acid (Merck), Chloroform (Brataco), P-Benzoquinone (Brataco), Absolute Ethanol (Merck).

## 3.3 Samples

Sampling was carried out by taking samples of the Medan City Super Market purposively, without comparing the samples taken with samples that were not taken. The samples in this study were energy drinks with various brands.

## 3.4 Quantitative Analysis

### 3.4.1 Artificial Sweetener Extraction

To 50 mL of sample add 10 mL of concentrated hydrochloric acid, shake homogeneously, extract with 25 mL of ether 3 times, wash the ether layer with 5 mL of distilled water, then evaporate on a water bath to form a residue.

### 3.4.2 Color Reaction

Add 5 mL of concentrated hydrochloric acid to 10 mg of extract, then add 2 mL of 10% barium chloride, leave for 30 minutes, filter with filter paper and then add 10 mL of 10% sodium nitrite, then a white precipitate occurs, which indicates the presence of cyclamate. Dissolve approximately 100 mg of the substance in 5 mL of a 10% sodium hydroxide solution. Evaporate until dry slowly melt the rest over the flame until there is no more ammonia, let it cool. Dissolve in 20 mL of water. The solution with hydrochloric acid and filter is neutralized, then add 1 drop of iron (III) chloride into the filtrate. Purple color indicates the presence of saccharin. Mix 20 mg of substance with 40 mg of resorcinol, add 10 drops of sulfuric acid, heat over low heat until dark green, cool, add aquadest and excess sodium hydroxide. Green, fluorescent liquid.

### 3.5 Quantitative Analysis

1. Weigh carefully 50 g of snacks plus 50 ml of distilled water and stir until

late.

2. Added 10 ml of concentrated HCL then extracted with 25 ml of diethyl ether as much as

three times.

3. Collected the extraction results, washed with 20 ml of distilled water, discarded distilled water (aquadest), solution

The ether is collected and then evaporated.

4. The residue is dissolved with 3 ml of acetone then added 2 ml of aquadest.

5. Titrate with 0.05 N NaOH solution with BTB indicator until there is a change in

color from yellow to blue.

The sample content obtained from the titration is made in the following tabulation:

# CHAPTER V

# CONCLUSION AND SUGGESTIONS

## 5.1 Conclusion

There are energy drinks circulating in the Medan city market that contain cyclamate, namely Proman samples with an average sodium cyclamate content of 10.926 mg/kg, however, the dose is still below the maximum threshold that is still permitted based on the requirements of BPOM Number HK.00.05.5.1.4547 2004 is not more than 1.25 g/kg material.

## 5.2 Suggestions

It is suggested to other researchers to test the content of other harmful ingredients in energy drinks circulating in the city of Medan.