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Synthesis and characterization of Taurine

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Abstract: Have been obtained 2-aminoethanesulfonic acid (taurine) from ethanolamine, sulfuric acid and sodium sulfite during the synthesis in laboratory condition. The process involves two steps of reactions, the first was esterification of ethanolamine with sulfuric acid to produce the intermediate product of 2-aminoethyl ester which than was extended to the second step by sulfonation with sodium sulfite to produce 2-aminoethanesulfonic acid. Resulting product was analyzed using ^{1}H-NMR, IR, FAB-MS analysis and examined purity characterizations of the synthesized products.

Keywords: 2-aminoethanesulfonic acid, esterification, sulfonation, ^{1}H-NMR, IR, FAB-MS

INTRODUCTION

The term energy drink refers to a beverage that contains caffeine in combination with other ingredients such as taurine, guarana, B vitamins, and that claims to provide its consumers with extra energy [1]. Energy drinks, include monster energy drink, market taurine as a substance that enhances the entry of glucose in to muscles - which improves endurance because the body uses the glucose in times of stress. Taurine along with caffeine may actually cause a “crash” effect after consumption [2].

Taurine (2-aminoethanesulfonic acid) is a conditionally - essential amino acid which is not utilized in protein synthesis, but rather is found free or in simple peptides. First discovered as a component of ox bile in 1827, it was not until 1975 that the significance of taurine in human nutrition was identified, when it was discovered that formula-red, pre-term infants were not able to sustain normal plasma or urinary taurine levels [2]. Taurine is found in large amounts in the brain, retina, heart and blood cells called platelets. The best food sources are meat and fish. Taurine is involved in a number of physiological processes including bile acid conjugation, osmoregulation, and detoxification of xenobiotics, cell membrane stabilization, modulation of cellular calcium flux, and modulation ofneuronal excitability. One of the imported materials is 2-aminoethanesulfonic acid which is as amino acid composed of protein which is useful in metabolism process. It is also needed as nutrition for brain in growth period, to stimulate a better condition of hearth and eye [3]. Taurine derived from amino acid which has a simple molecular structure. Many procedures to synthesized taurine mostly use two steps of reaction. Ethylene chloride reacts with sodium sulfite to produce 2-chloroethylsulfonic acid after refluxing for 72 hours and then it is reacted with ammonia to produce 75% of taurine. Reaction of ethanolamine and tionyl-chloride produces 2-chloroethylamine (80%) and then sodium bisulfate is added to produce 85% of taurine. From the tree procedures mentioned above, the second produces low yield while the first and third procedures involve starting materials which are difficult to obtain and carcinogenic [4, 5].

Based on literature study, we are conducting a research experiment to prepare 2-aminoethansulfonic acid in a laboratory scale. Raw materials of ethanolamine, sulfuric acid and sodium sulfite were used. Better yield was expected to be obtained and production cost would be lower.

EXPERIMENTAL

General: Chemicals used in this study were technical grade ethanolamine, sulfuric acid and sodium sulfite. Most reagents were obtained from commercial sources (Sigma-Aldrich, Tsetsuuh trade CO., Ltd-MGL) and used without further purification. The equipment used for preparation of taurine consisted of tree neck glass reactor, funnel, condenser, thermometer and hotplate with stirrer. All spectrometer determinations were made in the laboratory at Inje University, in South Korea. ^{1}H NMR spectra were obtained in D_2O on a spectrometer (LC-NMR, VNMRS 500; VARIAN, Palo Alto, CA, USA) at room temperature with tetramethylsilane (TMS) as an internal standard. FTIR spectra were recorded using KBr pellets on 8201 fourier transform infrared spectrophotometer (640-IR; VARIAN, Palo Alto, CA, USA). Molecular weights were determined on a high resolution FAB mass spectrometer (FAB-MS, JMS 700; Jeol, Akishima Tokyo, Japan).

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All reactions were performed in flame-dried or oven dried glassware under room atmosphere.

Synthesis: Preparation of 2-aminoethyl ester (1): Sulfuric acid (105g, 80%) was loaded into glass reactor equipped with a stirred and reflux condenser. Ethanolamine (61g, 10.0mol) was slowly added with stirring and water-cooling. The reactor was placed in an oil bath. The reflux condenser was replaced by an ordinary one. The heater was turned on. The intermediate product was produced and washed by using ethanol to remove the excess of sulfuric acid. Then, it was filtered and dried, give white powder product.

Synthesis of 2aminoethansulfonic acid (taurine) (2): Sodium sulfite (6.7g, 0.5mol) was dissolved with heating in water (30 ml) and then transferred to the reactor containing **1**. The mixture was refluxed for 20h. After that the condensate was distilled for 2-3 h, after which gradually with anhydrous sodium sulfate (14g, 1mol) with stirring and cooling to 50-60^{0}C.

Isolation of 2-aminoethansulfonic acid: The resulting suspension was treated gradually with aqueous ammonia (15ml, 25%). The contents of the reactor were stirred for 2-3 h with cooling to 10-15^{0}C.

The precipitate was filtered off and treated again with aqueous ammonia (15 ml). The ammonia extracts were combined and heated. Gaseous ammonia was distilled at first and absorbed by cold water. Condensate distilled next. The distillation was continued until product started to crystallize from the solution. The mixture was cooled. The resulting precipitate was separated to afford crude product that was recrystallized from water. After that that dried in air at 90-100^{0}C produced pure white powder taurine (compound **2**).

Quantitative determination of 2-aminoethansulfonic acid: The resulting product **2** determined qualitative and quantitative composition of impurities.

RESULTS AND DISCUSSION

The method for preparing of taurine using monoethanolamine, sulfuric acid, and sodium sulfite as starting materials is of great interest from the viewpoint of the technology and the process for preparing includes three main steps.

1. Preparation of 2-aminoethylsulfuric acid (2-aminoethyl ester, compound **1**) by esterification of monoethaolamine with sulfuric acid:

$$NH_2CH_2CH_2OH + H_2SO_4 \xrightarrow{®} NH_2CH_2CH_2SO_4H + H_2O \quad (1)$$

2. Synthesis of taurine by sulfonation of (compound **2**) whit sodium sulfite:

$$NH_2CH_2CH_2SO_4H + Na_2SO_3 \xrightarrow{®} NH_2CH_2CH_2SO_3H + Na_2SO_4 \quad (2)$$

3. Isolation of taurine from the mixture of side products.

The sulfate ester (compound 1) prepared in the first process step is the etherification monoethanolamine and sulfuric acid. Molar ratio of reactants between ethanolamine and sulfuric acid was varied 1:1.5, time period of reaction 5 h was the optimum molar ratio for etherification reaction. Water should be removed from the reaction mixture at reduced pressure and temperatures of 130-135^{0}C using vigorous stirring of the mixture. The intermediate product of 2-aminoethyl ester was produced and washed by using ethanol to remove the excess of sulfuric acid.

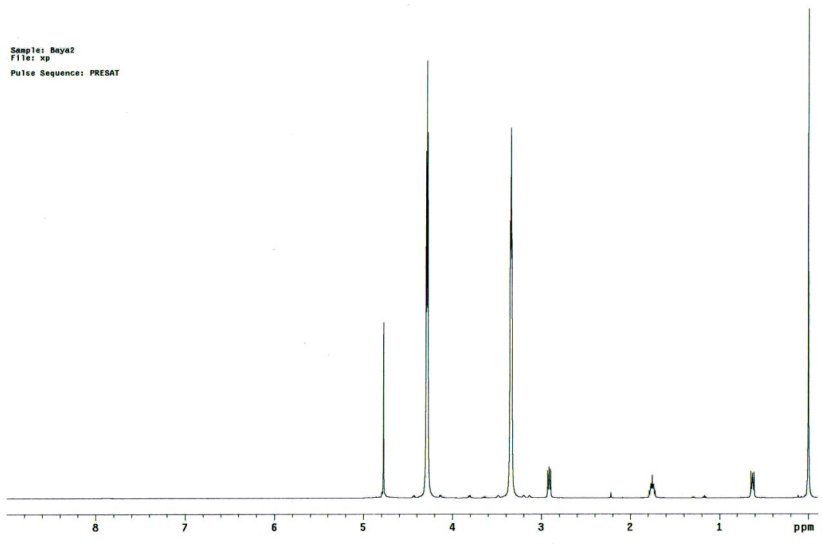

Fig. 1. ^{1}H NMR spectrum of 2-aminoethyl ester

The second reaction, sulfonation of 2-aminoethyl ester (**2**) with sodium sulfite was refluxed 20 h. The main products of performing this step with prolonged heating in aqueous are compound **2** and sodium sulfate. Taurine was isolated from the ammonia solution by removing gaseous ammonia from it by heating. The resulting product contained a small amount of sodium sulfate impurity. Therefore recrystallized with cold water, and obtained a pure product.

Intermediate and final compounds were analyzed with ^{1}H NMR, FTIR and FAB Mass spectrum. ^{1}H NMR spectrum showed that 2-aminoethansulfonic acid (taurine) has been produced in reaction which was indicated by methylene ($HOCH_2$-) - and (NH_2CH_2-) peaks at 3.439 ppm and 3.281 ppm, respectively (Fig.2). The peak was changed, the peak of reactant ethanolamine ($HOCH_2$-) was at 3.355 ppm and (NH_2CH_2-) was at 2.910 ppm (Fig.1). It can be noted that the time period was sufficient for the second reaction.

^{1}H NMR spectrum of the reactant, intermediate product and taurine product showed that conversion of first and second reactions were 100%, its mean that all of reactant was converted to each product. Figure 1 for ^{1}H NMR spectrum of compound 1 showed that the peak was 2-aminoethylester's peak, its mean that all of ethanolamine converted to 2-aminoethyl ester as an intermediate product.

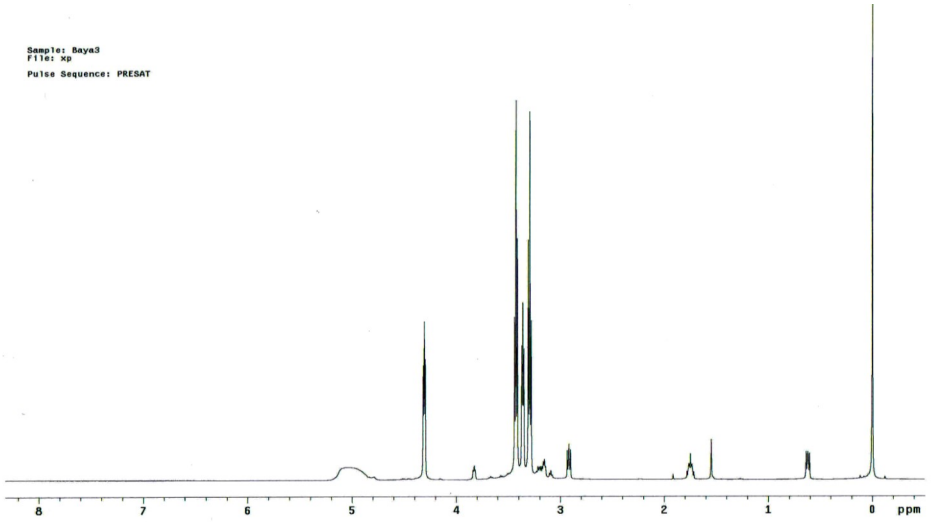

Fig. 2. ^{1}H NMR spectrum of 2-aminoethansulfonic acid

In other method of preparation of taurine can be purified and recrystallized using HCl and methanol [4]. In our case we purified using aqueous ammonia and recrystallized from cold water and to give pure product with high yield.

Analysis of the intermediate product and purified taurine by using FAB-MS showed the molecular of 142.04 and 126.98 respectively. In literature the molecular weight of compound **1** and compound **2** were 141.1414 and 125.1244 (Figure 3.). Due to high sulfate intent in intermediate product, purification of the compound **1** was necessary by washing with ethanol.

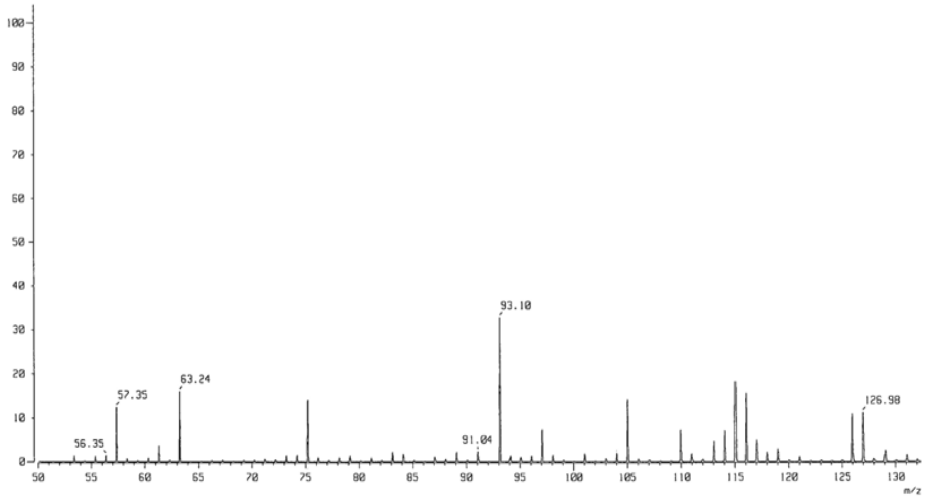

Fig. 3. FAB-MS of 2-aminoethansulfonic acid (taurine)

Determined the infrared absorption spectrum of 2 as directed in the potassium bromide and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers (Figure 4.). Furthermore 619.15 cm^{-1} more defined sulfonic acid (619.15 cm^{-1}), and observed the asymmetric C-H stretch of the methylene group (2965.4 cm^{-1}).

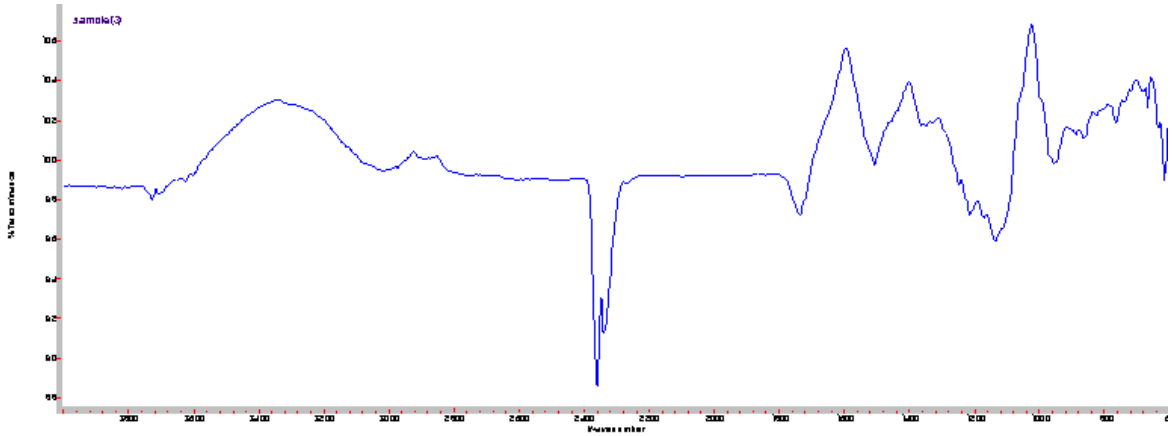

Fig. 4. IR of 2-aminoethansulfonic acid

In case of health safety the usage of taurine for energy drinks, those must need to determine qualitative and quantitative composition of impurities. Therefore we have been several studies that have strictly examined the effects of microbial characteristics and content of heavy metals and chloride, sulfate in the final product.

The results of bacterial testing are given in Table 1. Table 1 shows the results of microbial characteristics of taurine. Result was show that not detected any bacteria in final product. Chloride, sulfate and ammonium have been analyzed in the final product. The results of bacterial testing are given in the table 1.

Table 1. Bacterial characteristics of taurine

Simple	Salmonella	Salmonella	E.coli	S.aureus	B.cereus
Compound 1	-	-	-	-	-
Compound 2	-	-	-	-	-

Table 2. Characterization of taurine

Name of compound	**Characterizations**				
	Chloride (as Cl)	Sulphate (as SO_4)	Ammonium salt	Heave metal (Pb)	Heavy metal (As)
Taurine (2-aminoethansulfonic acid)	0.094%	0.089%	0.018%	7 ppm	1 ppm

Results of the table 2 were compared with standard results. The standard as shown, chloride not more than 0.011%, sulfate not more than 0.01%, ammonium not more than 0.02%, heavy metals, Pb not more than 10 ppm and As not more than 2 ppm, respectively.

CONCLUSIONS

2-aminoethylsulfonic acid (taurine) as one of the source of energy drinks can be prepared in laboratory scale from ethanolamine, sulfuric acid and sodium sulfite as raw materials with 2 steps of reaction. For the first time this work demonstrates results on the laboratory levels. Based on the results, it can be given good possibility to obtain taurine in Mongolia and as a good starting material for energy drink.

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