

[54] **PRESERVATION OF BEVERAGES WITH POLY(HEXAMETHYLENEBIGUANIDE HYDROCHLORIDE)**

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[22] Filed: **Oct. 5, 1973**

[21] Appl. No.: **403,970**

[52] U.S. Cl. **426/151, 426/227, 426/326, 426/330, 426/335, 424/326**

[51] Int. Cl. **A23I 3/34**

[58] Field of Search **426/151, 227, 326, 329, 426/330, 335; 424/326; 252/401, 405**

[56] **References Cited**

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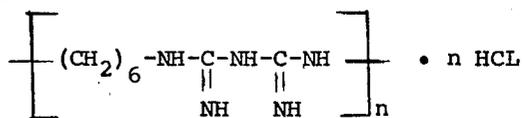
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[57] **ABSTRACT**

Beverages are preserved against undesirable microbial growth by incorporating into the beverage a polymeric biguanide compound of the formula



wherein *n* is such that the average molecular weight lies between 900 and 1,300.

14 Claims, No Drawings

**PRESERVATION OF BEVERAGES WITH
POLY(HEXAMETHYLENEBIGUANIDE
HYDROCHLORIDE)**

This invention relates to the preservation of beverages against undesirable microbial growth to prevent spoilage during storage.

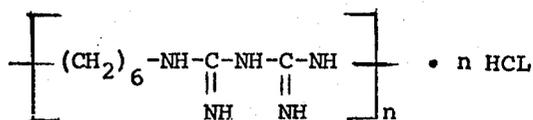
It is necessary in the beverage industry to take steps to make certain that the beverage does not become spoiled due to undesirable microbial growth in the package between the time of packaging and ultimate consumption. In the past this has involved such measures as pasteurization of the packaged article or constant refrigeration of the packaged beverage from the time of packaging until consumption. Each of these measures is, of course, expensive and thus adds to the final cost of the beverage to the consumer. Pasteurization is also attended with the disadvantages that the strength of the beverage container must be greater in order to withstand the high temperatures and pressures involved and that adverse affects on the quality of the beverage often occur.

In recent years other measures have been tried in order to eliminate the need for pasteurization and/or refrigeration. One of these is the microfiltration method whereby harmful microorganisms are filtered out of the beverage immediately prior to its introduction into the final package. This method is attended with difficulty also since the filtration procedure is somewhat expensive and highly sterile conditions must be maintained during the filling operation if it is to be effective.

Another, and more acceptable measure which has been developed is that of incorporating one or more preservative agents into the beverage prior to place it into the final container. The preservative is used in an amount which will prevent the growth of the beverage spoiling microorganisms. An example of this method is disclosed in U.S. Pat. No. 3,175,912 wherein the heptyl or octyl ester of parahydroxybenzoic acid or an alkali metal or alkaline earth metal salt thereof is incorporated into the beverage, e.g., finished unpasteurized beer. While this method, and particularly utilizing these materials, has met with very great success, it is still attended by certain difficulties. The materials to be used are sometimes expensive and it is frequently necessary to use large amounts of the materials. Often the materials are only slightly soluble in the beverage and it is necessary to work at levels which approach the limit of solubility of the material. Extensive research has thus been devoted to locating additional materials which will act as preservative agents in beverages and the use of which will not be attended with the difficulties encountered in the past. The present invention is based upon the discovery of such a material.

The beverages which can be preserved according to this invention are quite numerous and extend to all of those which do not have a chemical incompatibility with the particular material which is utilized. These would include fruit and vegetable juices, both natural and artificial, soft drinks, both carbonated and non-carbonated, as well as light alcoholic beverages such as cider, wines and beers, e.g., lager beer, porter and stout; ale and malt liquor are also intended within the meaning of "beer." In the present description, the emphasis is primarily directed to the preservation of beers, however, it is to be understood that the invention is applicable to a broad range of beverages.

The material which is employed as the preservative agent in the present invention is a polymeric biguanide compound of the formula



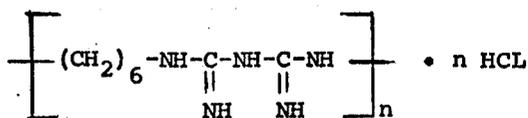
wherein n is such that the average molecular weight lies between 900 and 1,300.

The material may be prepared in the following manner:

Hexamethylene diamine carbonate is formed by passing CO_2 into hexamethylene diamine. This is then reacted at 60°C . with zinc dicyanamide to form a solution of the hexamethylene diamine salt of dicyanamide. This solution is further reacted with hexamethylene diamine hydrochloride at 150°C . to 155°C . to give the product of the above formula. See U.S. Pat. No. 2,643,232 and No. 3,428,576 for more detailed information regarding the product and the method of making it.

The polymeric biguanide material has been described in terms of the hydrochloride salt. It is to be understood, however, that other inorganic acid salts may be employed and for purposes of this invention are deemed to be the equivalents of the hydrochloride. It is also to be understood that the average molecular weight of the product may vary from the specific range given without adversely affecting the results. This material is known to be a biocide and solutions containing the material have previously been marketed as a disinfecting solution for plant equipment used in the brewing, foodstuffs and soft drink industries. In connection with the previously proposed use of the material as a sanitizing agent for brewing plant equipment, very small amounts of the material have been added to beer to determine any adverse effects upon foam, chill stability, and taste due to the possibility of any chance contamination of the beer which might occur if any of the compound were to be employed in filler sanitation. The amounts of the material added were, however, far below the amounts employed in the present invention and also well below the levels necessary to achieve preservation of the beer. To the knowledge of the present inventor, this material has not previously been suggested for incorporation into a beverage for preservation or at a level which would be necessary to achieve effective preservation of the beverage without pasteurization.

In accordance with this invention, the preservation of the beverage is achieved by the incorporation into the finished beverage, e.g., beer, for intimate admixture therewith the polymeric biguanide material:



wherein n is such that the average molecular weight lies between 900 and 1,300. The term "finished beverage" is used herein to refer to beverages which contain all of the necessary additives to make them commercially acceptable products and which have been subjected to any final filtration which may be necessary or desirable. In the case of beer, it refers to beer which has been subjected to polish filtration. The material can, of course, be added at any time during the processing of the bev-

erage so long as it does not interfere with the subsequent processing and so long as the subsequent processing does not interfere with the effectiveness of the material in achieving preservation of the beverage.

The material can be added to the beverage in any convenient form. It is preferred, due to ease of handling, to add it in the form of a stock solution wherein the material is dissolved in solvent which itself has no deleterious effect upon the beverage. In view of the high solubility of the polymeric biguanide material in water, an aqueous solution, e.g., a 20 percent aqueous solution, is particularly suitable. This high water solubility of the material is one of the factors which increases the value of the present invention since the aqueous solutions offer particularly noteworthy ease of handling.

The amount of the polymeric biguanide material employed will vary over a wide range depending upon the type of beverage being preserved and upon the magnitude of preservation to be obtained. In general, the amounts will extend from as low as about 0.4 parts by weight per million parts by volume (ppm) of the beverage employed up to about 50 ppm. The preferred range in beverages generally will be in the range of about 1 to about 10 ppm. In beer, it has been found that the range will generally be from about 0.4 to about 5.0 ppm, with a range of about 1.0 to about 2.0 ppm being preferred. Greater amounts of the material can be employed but are not recommended since they are unnecessary to achieve preservation and since the addition of any foreign material to a consumable beverage should be held as low as possible. One of the outstanding advantages of this invention is that the polymeric biguanide material can be utilized in extremely small amounts to achieve effective preservation of the beverage.

The minimum level of the material necessary to achieve preservation will, of course, be affected by the degree of sanitation achieved in the plant facility wherein the beverage is filled into the final container. It therefore is advisable to maintain the degree of sanitation at the highest level which is economically feasible.

The present invention is applicable to various forms of containers for the beverages. It is contemplated that cartons, bottles, cans, kegs, tank trucks and the like can be employed.

The beverage to be preserved can also be one which contains additives of various types which are used to improve the desirable properties of the beverage. Such additives may for example be color or taste improvers, or, for example, in the case of beer, additives which are used to improve the foaming qualities and/or chill stability properties of the beverage. Another outstanding advantage of the present invention is that the amounts of the polymeric biguanide material which achieve effective preservation are so low as to have a slight, if any, effect upon the properties of the beverage. This is not always the case with other materials which have been previously used to achieve preservation of beverages. Some of these have been found to impart adverse effects upon the quality of the beverage and have necessitated in certain cases the addition of other additives to overcome such adverse effects. It may be found that the polymeric biguanide material can also effectively be utilized in conjunction with other preservative materials. Such combinations of preservatives may be

found to be particularly effective for the preservation of certain particular beverages.

In the instant disclosure, the relationship between parts by weight and parts by volume is the same as that between grams and milliliters. Parts per million (ppm) is uniformly parts by weight of additive per million parts by volume of finished beverage.

The Examples as set forth hereinafter are purely illustrative of the invention and should not be construed as being exhaustive or limitative thereof. In the Examples, beer is utilized as the beverage. It is, however, to be understood that the instant invention is applicable to other beverages as well.

EXAMPLES 1 AND 2

From the regular production line, each of a group of clean, 12 ounce brown beer bottles is filled with cold, unpasteurized finished beer into which no microbiological preservative has been incorporated. The beer is foamed up to expel headspace air and the bottles are capped. Each of these bottles contains 350 ml. of unpasteurized lager beer. These bottles are utilized as the control in the Examples.

Stock Solutions

A series of stock solutions are made up in the following manner:

A required amount of the 20 percent aqueous solution of the polymeric biguanide material is diluted to 200 ml. with distilled water. The amount of the 20 percent water solution is chosen so that the addition of 0.5 ml. of the thus prepared stock solution to 350 ml. of beer yields a solution containing a desired amount of the polymeric biguanide material.

The stock solutions thus prepared are as follows:

Stock Solutions	Amount of Polymeric Biguanide Present When 0.5 ml. of Stock Solution is Added to 350 ml. of Beer (PPM)
1	0.1
2	0.2
3	0.4
4	0.5
5	1.0
6	2.0
7	5.0

EXAMPLE 1

Into each of a group of three clean, 12 ounce brown beer bottles is placed 0.5 ml. of stock solution 2. These bottles are filled from the regular production line with cold, unpasteurized finished beer. The beer is foamed up to expel headspace air and the bottles are capped. Each of these bottles contains 350 ml. of beer together with the stock solution. In the same manner 0.5 ml. of stock solution 3, 5 and 6 are respectively added to groups of three clean, 12 oz. brown beer bottles and the bottles are filled with cold, unpasteurized finished beer. The beer is foamed up to expel headspace air and the bottles are capped. Each of the bottles contains 350 ml. of beer together with the stock solution.

All of the thus prepared bottles together with control bottles are stored at room temperature and periodically (weekly) examined for microbial spoilage. Spoilage is readily observed by a marked amount of sediment in

the beer and by the unpleasant taste and odor produced by microbial growth and metabolism.

In the observations, the amount of sediment is visually observed and estimated on a scale of 1 to 9+. A value of 9+ reflects spoilage of the beer.

The results are set forth in the following Table A:

Table A

Polymeric Biguanide PPM.		SEDIMENT READINGS AFTER:				
		5 Weeks	9 Weeks	14 Weeks	24 Weeks	30 Weeks
Control	0	9+9+9+	9+9+9+	9+9+9+	9+9+9+	9+9+9+
	0.2	2-3-9	9+9+9+	9+9+9+	9+9+9+	9+9+9+
	0.4	1-1-1	1-2-1	1-2-3	3-3-4	3-3-4
	1.0	1-1-1	1-3-2	1-3-3	2-2*-2*	3-4-4
	2.0	1-1-1	3-1-2	2*-2-2	2-2-3	6-4-5

*The slightly lower sediment readings on these bottles compared with the values in the previous readings were due to minor experimental error in the visual estimation method employed.

EXAMPLE 2

Into groups of three clean 12 oz. brown beer bottles is placed 0.5 ml. of each of stock solutions 1, 2, 4, 5, 6 and 7 respectively in the manner of Example 1. These bottles are filled with cold, unpasteurized finished beer from the production line. The beer is foamed up to expel headspace air and the bottles are capped. Each of the bottles contains 350 ml. of beer together with the stock solution.

The bottles together with control bottles are stored at room temperature and periodically (weekly) examined for spoilage in the same manner as Example 1.

The results are set forth in the following Table B:

TABLE B

Polymeric Biguanide PPM.		SEDIMENT READINGS AFTER:			
		7 Weeks	12 Weeks	22 Weeks	28 Weeks
Control	0	9+9+9+	9+9+9+	9+9+9+	9+9+9+
	0.1	9+9+9+	9+9+9+	9+9+9+	9+9+9+
	0.2	9+9+9+	9+9+9+	9+9+9+	9+9+9+
	0.5	9+6-2	9+9+4	9+9+5	9+9+9+
	1.0	1-2-4	3-3-9+(11)	4-4	3*-6
	2.0	1-1-1	2-3-2	4-3-3	3*-2*-3
	5.0	1-3-2	2-3-2	3-3-3	3-4-2*

(11)negative acetic acid bacterial growth; test terminated with respect to this bottle.

*The slightly lower sediment readings on these bottles compared with the values in the previous reading were due to minor experimental error in the visual estimation method employed.

The foregoing Examples demonstrate the extremely good preservative ability of the polymeric biguanide material at very low levels.

EXAMPLES I TO VI

The following Examples demonstrate the chill stability and foam properties of beer containing the polymeric biguanide material as well as other materials for comparative purposes.

Stock Solutions

Various stock solutions are prepared as hereinafter disclosed for use in the following Examples.

Stock Solution No. I

Dissolve 1,680 mg. of n-heptyl parahydroxybenzoate (WS-7) in a sufficient amount of 95 percent ethanol to make 200 ml. of solution. The addition of 0.5 ml. of this stock solution, containing 4.2 mg. of WS-7, to 350 ml. of beer yields a solution containing 12 ppm. of the benzoate.

Stock Solution No. II

Dilute 1.4 ml. of the 20 percent aqueous solution of

the polymeric biguanide to 200 ml. with distilled water. The addition of 0.5 ml. of this stock solution, containing 0.70 mg. of the polymeric biguanide, to 350 ml. of beer yields a solution containing 2 ppm. of the polymeric biguanide.

Stock Solution No. III

- 20 Dissolve 10.5 gms. of Kelcoloid-O (Kelco Co.) a propylene glycol alginate produced in accordance with a process disclosed in U.S. Pat. No. 2,659,695, (hereinafter referred to as KDO), with vigorous agitation in sufficient distilled water to make 1,000 ml. of solution.
- 25 The addition of 2.0 ml. of this stock solution, containing 21 mg. of KDO, to 350 ml. of beer yields a solution containing 60 ppm. of KDO.

In these examples, the following procedures and standards are employed.

- 30 The Determination of Beer Foam Adherence

The procedure is that of Henry L. Ziliotto, John B.

- 50 Bockelmann and William Tirado of The F. & M. Schaefer Brewing Company, Brooklyn, N.Y. Said procedure was presented before the annual meeting of the American Society of Brewing Chemists in 1962. It comprises the creation of foam, the development of foam curtains and the measurement of foam adhering to the glass.

- 55 A. Formation of Foam Curtains

Attemperate beer at least over night at 10°C.

- 60 Use 6 oz. shell glasses that have been numbered on the side near the base and marked with five spots uniformly spaced around the periphery of the bottom, one of the marks being more pronounced than the others. These marks serve as position guides when reading the instrument. Pour the beer into a glass by the method for Foam Life (Ziliotto, H., Bockelmann, John B., "American Society of Brewing Chemists Proceedings," 1954, page 108) to form a head of foam between 20 and 35 millimeters (mm.) in height. When the foam has collapsed (indicated by a change in color of the foam surface or by the beer's starting to show through), encircle the glass at the beer-foam interface with an 8 inch

length of black adhesive tape of $\frac{3}{4}$ inch to 1 inch width. (The foam height at zero time and the rate of foam collapse need not be measured unless it is desired to determine Foam Life at the same time.) Immediately remove every drop of beer in the glass by means of an aspirator, taking care not to disturb the adhering foam. Let the glass dry in air a minimum of ten minutes, and measure the height of the exposed surface between the tape and the top of the glass. Wipe the exterior of the glass with a damp cloth and polish dry.

B. Instrument Readings

The Radiometer described by Thorne, R. S. W., and Beckley, R. F., "Journal of the Institute of Brewing," 64, 38 (1958) is employed for making the readings, but the light intensity is reduced by inserting a variable rheostat in the lamp circuit in a manner that will not affect the operation of the built-in fan or other circuitry. Set the rheostat to give 60 volts for measurement of foam adhesion. Raise again to full current at the termination of the test for future turbidity readings.

On the leading edge of each vane of the centering mechanism of the instrument slip on a 45 mm. length of rubber tubing (6 mm. bore and 2 mm. walls) that has been slit its full length. Fasten a piece of tape over the top end of the tubings so that they will not slip down. When in position, the bottom ends should just clear the top of the support described later. This modification properly centers the glasses despite slight differences in diameter between them and their support.

Do not use water in the cell compartment.

Place a stack of four discs centrally at the bottom of the compartment to act as a support for the glasses. The discs have a diameter just a trifle larger than that of the glasses (59 to 60 mm. diameter for a 55 mm. I.D., 58 mm. O.D., glass) and are painted a dull black. The bottom one is 25 mm. thick while each of the other three is 10 mm. thick.

Invert the glass with foam curtains onto the stack of discs and position the principle mark on the glass toward the light source. Close the cover of the instrument and after turning the current on, momentarily depress the lamp switch to activate the incident light. Note any deflection of the meter needle. Release the lamp switch and adjust the diaphragm dial by an amount estimated to be sufficient to bring the needle to zero. Repeat the illumination and the adjustment a number of times until a dial reading is obtained that is constant within 0.1 unit for a zero needle deflection. Record the reading. Rotate the glass one-fifth turn around its axis, using the marks on the glass for guides, and obtain a new reading as previously obtained. Continue in this manner for five readings. If another level of adhered foam must be read, remove the topmost disc to lower the glass and obtain another five readings around the glass. Proceed thusly for a third level. Use the following table to decide on the number of levels to be measured.

Height of Foam Curtains	Number of Levels to be Measured
0 - 14 mm.	1
15 - 24	2
25 - 36	3

Calculate to the second decimal the average of all readings.

Obtain blank readings on the clean glass without removing the tape, by the procedure followed for the run. When blank readings have once been obtained on a glass at the three planes, use the data for subsequent determinations. Occasionally, recheck the readings to correct for surface changes.

Subtract the average blank reading from that of the run to obtain foam adhesion in the one glass. Report to the first decimal the grand average of six determinations (two pours from each of three replicate bottles, or single pours from six bottles) as the foam adhesion of the beer tested.

Tests have shown that no detectable change in readings of dry foam occur during the three day's standing at room temperature. This finding makes it convenient to postpone reading the glasses in the instrument until all beers of a series have been poured. As a precautionary note, do not cover wet glasses for any reason inasmuch as the entrapped vapors dissolve the foam or change its appearance.

C. Visual readings

Subsequent to the cited publication estimations of the adhering foam have also been obtained visually as follows:

Critically examine the beer glass containing the dry film of foam and estimate the average per cent of the circumferential surface between the tape and the top of the glass that was originally covered by foam bubbles. Express foam adhesion as one-tenth of the percentage figure obtained.

Chill Haze Measurement

The procedure utilized is that of The F. & M. Schaefer Brewing Co., Brooklyn, N.Y. and as the procedure for the determination of foam adherence, does not constitute a part of the instant invention, it is described only to assist in the full appreciation of the data presented.

Chill the upright samples of bottled beer for the specified time in a bath maintained at 0°C.

Using a viewing box, such as Clark turbidimeter (Cargille Scientific Co., New York, New York), determine the turbidity of the sample by visually matching the haze of the supernatant fluid with the suspended haze of standards in bottles of the same shape and color as that containing the sample. The haze standards may be either the Formazin standards of the American Society of Brewing Chemists (A.S.B.C. Proceedings 1957, page 165) or suspensions of insoluble substances that have been visually standardized against the Formazin standards. The Schaefer haze standards have nominal values ranging from 0 to 9, a unit of which is the visual equivalent of 30 Formazin units; i.e., a Schaefer haze of 2 equals 60 Formazin units.

Description of Foam Life Measurement

The foam life measurement is carried out according to the procedure described by Ziliotto and Bockelmann in the American Society of Brewing Chemists Proceedings of 1954, pages 108-210. This method incorporates the pouring of the beer from a 12 oz. bottle into a standard 6 oz. glass in such a way that a foam head of about 25 millimeter height (20 to 35 mm. is acceptable) is formed. The average time, in seconds, required for the collapse of the foam head of twelve pourings is recorded. By direct proportion calculate the life of 25

mm. of original foam and express the average results of twelve calculations as the foam life.

EXAMPLES I TO VI

Example I

Fill each of a group of clean, 12 oz. brown beer bottles with cold, unpasteurized finished beer to which no preservative has been added. Foam up the beer, to expel headspace air and cap. Pasteurize the bottles from this group. Each bottle prepared in this manner contains about 350 ml. of pasteurized lager beer. These bottles are utilized as the control in foam and chill stability tests, the results of which appear in Table I.

Example II

Fill each of a group of clean, 12 oz. brown beer bottles with the same beer as used in Example I. Add, to each filled bottle, 2.0 ml. of stock solution No. III (KDO). Foam up the beer to expel headspace air and cap. Pasteurize the bottles from this group. Each bottle prepared in this manner contains about 350 ml. of pasteurized lager beer and 60 ppm. KDO. The results of foam and chill stability tests on these bottles appear in Table I.

Example III

Place 0.5 ml. of stock solution No. 1 (WS-7) into each of a group of clean 12 oz. brown beer bottles. Fill each of the bottles with the same beer as used in Exam-

and cap. Each bottle prepared in this manner contains about 350 ml. of unpasteurized preservative and KDO treated beer. The concentrations of WS-7 and KDO are about 12 and 60 ppm., respectively. The results of foam and chill stability tests on these bottles appear in Table I.

Example V

Place 0.5 ml. of stock solution No. II (polymeric biguanide) into each of a group of clean 12 oz. brown beer bottles. Fill each of the bottles with the same beer as used in Example I. Foam up the beer to expel the headspace air and cap. Each bottle prepared in this manner contains about 350 ml. of unpasteurized preservative treated beer. The concentration of polymeric biguanide is about 2 ppm. The results of foam and chill stability tests on these bottles appear in Table I.

Example VI

Place 0.5 ml. of stock solution II (polymeric biguanide) into each of a group of clean 12 oz. brown beer bottles. Fill each of the bottles with the same beer as used in Example I. Add to each bottle 2.0 ml. of stock solution No. III (KDO). Foam up the beer to expel the headspace air and cap. Each bottle prepared in this manner contains about 350 ml. of unpasteurized preservative and KDO treated beer. The concentrations of polymeric biguanide and KDO are about 2 and 60 ppm., respectively. The results of foam and chill stability tests as these bottles appear in Table I.

TABLE I

EXAMPLE		CHILL HAZE AFTER					
		3 DAYS	2 WEEKS	4 WEEKS	6 WEEKS	8 WEEKS	12 WEEKS
I	Control	0-0-0-0-0-0	0-0-0-0-0-0	3-3-3-3-3-3	3-3-3-3-3-3	3-3-3-3-4-5	4-4-4-4-4-5
II	KDO 60 ppm	0-0-0-0-0-0	0-0-0-0-0-0	1-1-1-1-1-1	1-1-1-1-1-1	1-1-1-1-1-1	1-1-1-1-1-1
III	WS-7 12 ppm.	0-0-0-0-0-0	0-0-0-0-0-0	3-3-3-3-3-3	3-3-3-4-4-4	4-4-5-4-6-6	4-4-4-6-7-7
IV	KDO 60 ppm. + WS-7 12 ppm.	0-0-0-0-0-0	0-0-0-0-0-0	1-1-1-1-1-0	1-1-1-1-1-1	1-1-1-2-2-*	3-3-3-3-3-*
V	Polymeric biguanide 2 ppm.	0-0-0-0-0-0	0-0-0-0-0-0	0-0-0-0-0-0	1-1-1-1-1-1	1-1-1-1-1-1	3-3-3-3-3-3
VI	KDO 60 ppm. + Polymeric biguanide 2 ppm.	0-0-0-0-0-0	0-0-0-0-0-0	0-0-1-1-1-1	1-1-1-1-1-1	1-1-1-1-1-1	2-2-2-2-2-2

FOAM PROPERTIES

FOAM LIFE (SEC.)		FOAM ADHESION	
FRESH	2 Weeks at 90°F	FRESH	2 Weeks at 90°F
102	94	7.1	6.8
114	102	6.8	7.6
92	86	3.8	3.8
108	111	2.6	0.5
100	100	7.3	6.9
107	108	6.8	6.7

*Bottle opened for inspection.

ple I. Foam up the beer to expel the headspace air and cap. Each bottle prepared in this manner contains about 350 ml. of unpasteurized preservative treated beer. The concentration of WS-7 present is about 12 ppm. The results of foam and chill stability tests on these bottles appear in Table I.

Example IV

Place 0.5 ml. of stock solution No. I (WS-7) into each of a group of clean 12 oz. brown beer bottles. Fill each of the bottles with the same beer as used in Example I. Add to each bottle 2.0 ml. of stock solution No. III (KDO). Foam up the beer to expel the headspace air

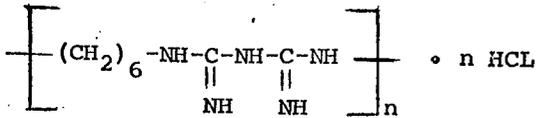
From the foregoing Examples I to VI it is seen that the WS-7 preservative material has an adverse effect on the foam life and foam adherence properties of the beer. These adverse effects on foam life are overcome by incorporating KDO into beer. In contradistinction, the polymeric biguanide material has no adverse effect upon the foam life and foam adherence properties of the beer. The data demonstrate that a commercially acceptable beer can be produced using the polymeric biguanide material alone without the addition of KDO.

The invention has been illustrated above by reference to small quantities of the beverage. It will be apparent to the art skilled that the preservative method

can be scaled up to producing commercial quantities of the beverage. In this respect a stock solution of the polymeric biguanide material is first prepared. This stock solution is then injected into the beverage pipeline at the desired stage of production as the beverage flows through the line. The rate of injection is, of course, correlated to the flow rate of the beverage. The proper proportioning is achieved through means which are per se known. The beverage containing the additive is then filled into the desired container. In a batch operation a desired amount of a stock solution is added to the beverage batch and admixed therewith. The filling operation is then completed to obtain the final packaged beverage article.

We claim:

1. A method of preserving a beverage selected from the group of fruit and vegetable juices, soft drinks and light alcoholic beverages against undesirable microbial growth which comprises incorporating into the beverage prior to packaging the polymeric biguanide material of the formula



wherein n is such that the average molecular weight lies between 900 and 1,300

the amount of the polymeric biguanide incorporated being sufficient to preserve the beverage against undesirable microbial growth.

2. A method according to claim 1 wherein the amount of polymeric biguanide incorporated into the beverage is from 0.4 to about 50 ppm.

3. A method according to claim 2 wherein the amount of polymeric biguanide incorporated into the beverage is from 1 to about 10 ppm.

4. A method according to claim 1 wherein the beverage is a light alcoholic beverage.

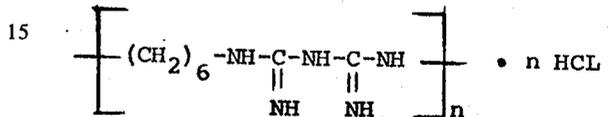
5. A method according to claim 4 wherein the beverage

is a beer.

6. A method according to claim 5 wherein the amount of polymeric biguanide incorporated into the beer is from 0.4 to about 5 ppm.

7. A method according to claim 6 wherein the amount of polymeric biguanide incorporated into the beer is from about 1.0 to about 2.0 ppm.

8. A composition which comprises a beverage selected from the group of fruit and vegetable juices, soft drinks and light alcoholic beverages having incorporated therein the polymeric biguanide material of the formula



wherein n is such that the molecular weight lies between 900 and 1,300

the amount of the polymeric biguanide present being sufficient to preserve the beverage against undesirable microbial growth.

9. A composition according to claim 8 wherein the amount of polymeric biguanide present is from 0.4 to about 50 ppm.

10. A composition according to claim 9 wherein the amount of polymeric biguanide present is from about 1 to about 10 ppm.

11. A composition according to claim 8 wherein the beverage is a light alcoholic beverage.

12. A composition according to claim 11 wherein the beverage is a beer.

13. A composition according to claim 12 wherein the amount of polymeric biguanide present in the beer is from 0.4 to about 5 ppm.

14. A composition according to claim 13 wherein the amount of polymeric biguanide present in the beer is from about 1.0 to about 2.0 ppm.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,860,729 Dated January 14, 1975

Inventor(s) FREDE B. STRANDSKOV and JOHN B. BOCKELMANN

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

On the cover sheet of the patent, in section "[75]", the second inventor's name should read --John B. Bockelmann--.

Signed and Sealed this
Twenty-first Day of March 1978

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

LUTRELLE F. PARKER
Acting Commissioner of Patents and Trademarks