Purification of Sodium Isethionate


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12 Claims. (Cl. 260—513)

This invention is concerned with a process for the purification of sodium isethionate. In particular, it is concerned with a process for treating sodium isethionate so that when the materials is subsequently used in the synthesis of a detergent, the resulting detergent will not have an undesirable odor.

Sodium isethionate is generally prepared commercially by the reaction of ethylene oxide and sodium bisulfite. U.S. Patent 2,810,747 discloses a method of carrying out the reaction so as to minimize impurities by careful selection of reaction conditions. Sulfite and bisulfite impurities can be removed readily from sodium isethionate prepared by methods other than that of the patent. For example, they may be removed by the following washing process: a 5% aqueous solution of the material at atmospheric pressure in a heated sigma blade mixer, producing a dry powder containing no reducing material detectable by iodine titration. Alternatively, the sodium isethionate solution is treated with a low level (less than 1%) of oxidizing agents such as sodium hypochlorite and then evaporated to dryness. Again, iodine titration shows no reducing impurities to be present.

The avoidance or removal of sulfite and bisulfite impurities, however, does not prevent the sodium isethionate material from producing an objectionable odor when it is converted to a detergent. On the contrary, even though the sodium isethionate has been prepared or treated so that it contains no detectable sulfite or bisulfite, when that sodium isethionate is converted to a detergent, the detergent may have a disagreeable odor. The absence of sulfite or bisulfite in a finished sodium isethionate powder of itself is not sufficient to assure that the detergent prepared therefrom will possess a clean odor.

According to the present invention, odor causing impurities are removed from sodium isethionate by treating the material with certain oxidizing agents. Not all oxidizing agents bring about the desired result. Good results are obtained with sodium hypochlorite, sodium chlorite, calcium hypochlorite, potassium persulfate and potassium monopersulfate. Most surprisingly, however, hydrogen peroxide does not yield good results, and neither does sodium perborate or sodium nitrate. Sodium nitrate has the additional disadvantage of producing a darkening of the treated material.

The sodium isethionate is first treated to remove whatever sulfite or bisulfite impurities are present, and is then subjected to treatment with an oxidizing agent according to the present invention. The minimum level of oxidizing agent required varies somewhat depending upon the particular oxidizing agent used and upon the level of odor causing impurities which are present in the sodium isethionate. The lowest level of oxidizing agent solutions which have given very good, clean odors in the final detergent are as follows, the percentages referring to the weight of treated sodium isethionate:

<table>
<thead>
<tr>
<th>Oxidizing Agent</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hypochlorite</td>
<td>0.24</td>
</tr>
<tr>
<td>Sodium chlorite</td>
<td>0.1</td>
</tr>
<tr>
<td>Potassium persulfate</td>
<td>1.0</td>
</tr>
<tr>
<td>Potassium monopersulfate</td>
<td>0.5</td>
</tr>
</tbody>
</table>

Useful improvements in final detergent odors were observed at the following levels:

<table>
<thead>
<tr>
<th>Oxidant</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hypochlorite</td>
<td>0.12</td>
</tr>
<tr>
<td>Sodium chlorite</td>
<td>0.05</td>
</tr>
<tr>
<td>Calcium hypochlorite</td>
<td>0.12</td>
</tr>
<tr>
<td>Potassium persulfate</td>
<td>0.42</td>
</tr>
<tr>
<td>Potassium monopersulfate</td>
<td>0.25</td>
</tr>
</tbody>
</table>

The above results were obtained using commercial sodium isethionate which analyzed approximately 0.02% reducing material calculated as sodium sulfite. For sodium isethionate containing higher levels of reducing impurities, higher levels of oxidizing agents are required. For example, in the case of sodium isethionate to which 0.4% sodium sulfite was deliberately added, sodium chloride was required in an amount sufficient to oxidize all the added sodium sulfite plus 0.2% excess. When the original level of reducing materials in the sodium isethionate is unknown, the amount of oxidizing agent required to purify the material can be determined by first analyzing the sample for reducing materials, for example, by means of an iodine titration.

In general, there is no advantage in using any of the oxidants of the present invention in an amount over 1%. Use of excessive amounts of oxidant, besides being uneconomical, has the disadvantages of causing a destruction of the isethionate and a darkening of color.

Of the oxidizing agents useful in the present process, sodium hypochlorite is preferred. Sodium isethionate treated according to the present invention with sodium hypochlorite may be converted into acyl isethionate detergents which have a very clean odor and which retain such a clean odor for a longer time than is the case when other oxidizing agents have been used.

The following examples are given solely for purposes of illustration and are not to be considered as limiting the invention to these embodiments. Many modifications will be apparent to those skilled in the art without departing from the spirit or scope of the invention.

Example 1

Preparation of Acyl Isethionate Detergents

All acyl isethionate samples discussed in the examples below were prepared in pilot plant equipment according to the following procedure: 10.7 lbs. of sodium isethionate was heated to 108° C. in a 10 gal. jacketed, stainless steel, sigma blade mixer using 30 lb. steam. 14.85 lbs. of coconut fatty acid chloride (prepared from iodine value = 5 coconut fatty acids and phosphorous trichloride) was then added and the mixture was heated until the reaction (evolution of HCl) ensued and the temperature rose to 130° C. The steam was then shut off and mixing was continued until the temperature fell to 90° C. The pH (10% solution in distilled water at 35° C.) of the resulting powdery reaction product was then adjusted to 7.2 by slowly adding approximately 180 grams of 50% sodium hydroxide. After mixing for an additional ten minutes, the batch was cooled to 40° C. by passing tap water through the mixer jacket. The batch was then discharged and sampled for an odor evaluation. The overall time cycle for each preparation was about one hour.

The odor evaluation was carried out on a water slurry of the acyl isethionate detergent samples (about one teaspoon of each) in a clean odor-free disposable paper cup. The slurry was prepared and smelled by the tester in a special odor testing room to avoid interference from for-
eign odors. The results were credited according to the following system: A rating of “1” was assigned to a sample which had a satisfactory clean odor. A rating of “2” meant that the sample had an acceptable odor. A rating of “3” was given to samples which had an odor level too strong to be masked by perfume.

In Examples 2 through 9 cited below the same lots of fatty acid chloride and sodium isethionate were used in order to obtain a comparison of the effectiveness of the various oxidizing agents.

**EXAMPLE 2**

Acyl isethionate detergent was prepared as described in Example 1 above from regular commercial sodium isethionate. The odor of the resulting detergent was given the rating of “3,” and the material had a smell of burnt rubber.

**EXAMPLE 3**

Acyl isethionate detergent was prepared as described in Example 1 from commercial sodium isethionate which had first been dissolved in water to form a 42% aqueous solution and then evaporated to dryness before reacting with the acid chloride. The odor of the resultant detergent was still given the rating of “3” and the material still had a burnt rubber odor. The experiment shows that in the absence of treatment with a suitable oxidizing agent according to the present invention, simple evaporation of an aqueous solution of sodium isethionate, even though it is sufficient to remove the sulfite and bisulfite impurities, does not lead to a clean or improved odor in the ultimate detergent.

**EXAMPLE 4**

*Use of Sodium Hypochlorite*

240 grams of 5.25% sodium hypochlorite solution was added to a solution of 12 lbs. of sodium isethionate in 16.6 lbs. of water; i.e., of 0.23% NaOCl basis sodium isethionate. The resulting solution was then evaporated to dryness at atmospheric pressure in a 10 gal. sigma blade, stainless steel mixer using 40 lb. steam on the jacket. 10.7 lbs. of the resulting dry sodium isethionate was then converted to acyl isethionate detergent according to the procedure described in Example 1. The odor of the acyl isethionate detergent was noticeably clean and rated “1.”

**EXAMPLE 5**

*Use of Sodium Hypochlorite*

The procedure of Example 4 was repeated, but 1% NaOCl (basis sodium isethionate) was used. The odor of the resulting detergent was noticeably clean and rated “1.”

**EXAMPLE 6**

*Use of Sodium Chlorite*

Eight grams of sodium chlorite (100%) was dissolved in 148 grams of water containing 3 grams of sodium hydroxide. This solution was then added to 12 lbs. of sodium isethionate dissolved in 16.6 lbs. of water; i.e., 0.15% NaClO₂ basis sodium isethionate. After the resulting solution was evaporated to dryness, the sodium isethionate was converted to acyl isethionate detergent as described in Example 1 above. The odor of the acyl isethionate detergent was noticeably clean and rated “1.”

**EXAMPLE 7**

*Use of Calcium Hypochlorite*

40.8 g. of 30.2% active calcium hypochlorite was added to a solution of 12 lbs. of sodium isethionate in 16.6 lbs. of water; i.e., 0.23% Ca (OCl)₂ basis sodium isethionate. After the resulting solution was evaporated to dryness, the sodium isethionate was converted to acyl isethionate detergent as described in Example 1. The odor of the acyl isethionate detergent was moderately improved (“2” rating) compared to the control.

**EXAMPLE 8**

*Use of Potassium Persulfate*

54.5 grams of potassium persulfate (100%) was added to a solution of 12 lbs. of sodium isethionate in 16.6 lbs. of water; i.e., 1.0% K₂S₂O₅ basis sodium isethionate. Approximately 48 grams of 50% sodium hydroxide was added to this solution during the subsequent evaporation step in order to maintain the pH between 8 and 9. The dried sodium isethionate was then converted into acyl isethionate detergent according to the procedure described in Example 1. The odor of the acyl isethionate detergent was noticeably clean and rated “1.”

**EXAMPLE 9**

*Use of Potassium Monopersulfate*

59 grams of 44.6% active potassium monopersulfate (trademark Oxone) was dissolved in a solution of 12 lbs. of sodium isethionate in 16.6 lbs. of water; i.e., 0.5% KH₂O₂ basis sodium isethionate. Thirty grams of 50% sodium hydroxide was added during the subsequent evaporation step in order to maintain the pH between 8 and 9. The dried sodium isethionate was then converted into acyl isethionate detergent according to the procedure described in Example 1. The odor of the acyl isethionate detergent was noticeably clean and rated “1.”

**EXAMPLE 10**

*Use of Potassium Monopersulfate*

123 grams of 44.0% active potassium monopersulfate (trademark Oxone) was dissolved in a solution of 12 lbs. of sodium isethionate in 16.6 lbs. of water; i.e., 1.0% KH₂O₂ basis sodium isethionate. Fifty-seven grams of 50% sodium hydroxide was added during the subsequent evaporation step in order to maintain the pH between 8 and 9. The dried sodium isethionate was then converted into acyl isethionate detergent according to the procedure described in Example 1. The odor of the acyl isethionate detergent was noticeably clean and rated “1,” as compared with a “2” rating and a burnt characteristic for the product made from the same lot of sodium isethionate, but which was not treated in accordance with the invention.

**EXAMPLE 11**

*Use of Potassium Monopersulfate*

30.0 grams of 44.0% active potassium monopersulfate (trademark Oxone) was dissolved in a solution of 8.0 grams of sodium hydroxide in 240 grams of water. This solution (i.e., 0.25% KH₂O₂ basis sodium isethionate) was then added to a solution of 11.6 lbs. of sodium isethionate in 16 lbs. of water. The combined solutions were evaporated to dryness. The dried sodium isethionate was then converted to acyl isethionate detergent according to the procedure described in Example 1. The odor of the acyl isethionate detergent was moderately improved and rated “2” with a slight burnt characteristic, as compared with a “3” rating and a strong burnt rubber characteristic for the product made from the same lot of sodium isethionate, but which was not treated in accordance with the invention.

What is claimed is:

1. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising removing whatever sulfite and bisulfite impurities are present, and treating the sodium isethionate with an oxidizing agent selected from the group consisting of sodium hypochlorite, sodium chlorite, calcium hypochlorite, potassium persulfate and potassium monopersulfate, each of said oxidizing agents, when selected, being present in the following minimum percent-
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ages by weight of sodium isethionate: sodium hypochlorite—0.12%, sodium chloride—0.05%, calcium hypochlorite—0.12%, potassium persulfate—0.42%, and potassium monopersulfate—0.52%, said oxidizing agent being present in a maximum amount of about 1%.

2. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating the sodium isethionate with an oxidizing agent selected from the group consisting of sodium hypochlorite, sodium chloride, calcium hypochlorite, potassium persulfate and potassium monopersulfate, each of said oxidizing agents, when selected, being present in the following minimum percentages by weight of sodium isethionate: sodium hypochlorite—0.12%, sodium chloride—0.05%, calcium hypochlorite—0.12%, potassium persulfate—0.42% and potassium monopersulfate—0.52%, said oxidizing agent being present in a maximum amount of about 1%.

3. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating the sodium isethionate with sodium hypochlorite in an amount sufficient to oxidize whatever sulfite and bisulfite impurities are present, and with at least an excess of 0.24% based on the weight of the sodium isethionate, said sodium hypochlorite being present in a maximum amount of about 1%.

4. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating the sodium isethionate with sodium hypochlorite in an amount sufficient to oxidize whatever sulfite and bisulfite impurities are present, and with at least an excess of 0.24% based on the weight of the sodium isethionate, said sodium hypochlorite being present in a maximum amount of about 1%.

5. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating sodium isethionate which is substantially free of sulfite and bisulfite impurities with sodium hypochlorite in an amount at least 0.12% based on the weight of the sodium isethionate, said sodium hypochlorite being present in a maximum amount of about 1%.

6. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating sodium isethionate which is substantially free of sulfite and bisulfite impurities with sodium hypochlorite in an amount at least 0.24% based on the weight of the sodium isethionate, said sodium hypochlorite being present in a maximum amount of about 1%.

7. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating the sodium isethionate with sodium chlorite in an amount sufficient to oxidize whatever sulfite and bisulfite impurities are present, and with at least an excess of 0.5% based on the weight of the sodium isethionate, said sodium chlorite being present in a maximum amount of about 1%.

8. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating the sodium isethionate with sodium hypochlorite in an amount sufficient to oxidize whatever sulfite and bisulfite impurities are present, and with at least an excess of 0.12% based on the weight of the sodium isethionate, said sodium hypochlorite being present in a maximum amount of about 1%.

9. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating the sodium isethionate with sodium hypochlorite in an amount sufficient to oxidize whatever sulfite and bisulfite impurities are present, and with at least an excess of 0.42% based on the weight of the sodium isethionate, said sodium hypochlorite being present in a maximum amount of about 1%.

10. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating the sodium isethionate with sodium hypochlorite in an amount sufficient to oxidize whatever sulfite and bisulfite impurities are present, and with at least an excess of 0.5% based on the weight of the sodium isethionate, said sodium hypochlorite being present in a maximum amount of about 1%.

11. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating the sodium isethionate which is substantially free of sulfite and bisulfite impurities with sodium hypochlorite in an amount at least 0.5% based on the weight of the sodium isethionate, said sodium hypochlorite being present in a maximum amount of about 1%.

12. A process for removing odor causing impurities from sodium isethionate which has been prepared by the reaction of ethylene oxide and sodium bisulfite, said process comprising treating sodium isethionate which is substantially free of sulfite and bisulfite impurities with sodium monopersulfate in an amount at least 0.25% based on the weight of the sodium isethionate, said sodium monopersulfate being present in a maximum amount of about 1%.

References Cited in the file of this patent

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