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Alkaline tolerant sulfobetaine amphoteric surfactants.

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GB-A-1 037 647
GB-A-1 419 097
GB-A-2 081 259
US-A-3 280 179

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Description

The present invention concerns an aqueous basic solution of a base, water and betaine according to the introductory portion of claim 1 and a method for making a storage stable aqueous basic solution according to the introductory portion of claim 11.

In a variety of cleaning and other chemical uses, use of strong basic aqueous solutions is required. Since many of these cleaning and other chemical uses also require the basic solution to exhibit surfactancy, a need exists for surface active agents or surfactants which exhibit their surface active properties when contained in strongly basic aqueous solutions.

As an additional property, such surfactants in a strongly basic alkaline solution should display very low to moderate foaming characteristics. Thus, an unusual combination of constraints are placed upon a surfactant in order to satisfy such criteria.

The class of surfactants used in the present invention are soluble in strongly basic alkaline solutions, are storage stable for extended time periods of storage in strongly basic alkaline solutions, and are very low to moderate foamers. Moreover, such surfactants retain their surfactant properties when incorporated into strongly basic aqueous solutions. The surfactants which possess such unique combination of properties are alkyl sulfobetaines wherein the alkyl group ranges from about 6 to 12 carbon atoms in chain length. Another aspect of the present invention is an aqueous solution of the alkyl sulfobetaine and a base, such as, for examples, sodium hydroxide or potassium hydroxide in concentrations ranging up to 30%–50% in concentration.

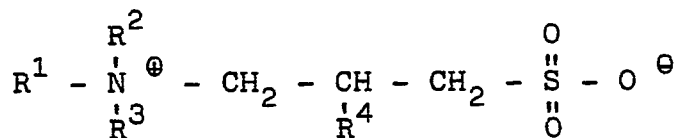
Advantages of the present invention include an amphoteric surfactant which is soluble in water and which is soluble and storage stable in an aqueous basic solution. A further advantage is that the amphoteric surfactant retains its surfactant properties in an aqueous basic solution. Yet another advantage is that the amphoteric surfactant is stable in strongly basic aqueous solutions. These and other advantages will be readily apparent to those skilled in the art based upon the disclosure contained herein.

Sulfobetaines are known in the art and are described in the GB-A 2 081 259, GB-A 1 419 097, GB-A 1 037 647 and US-A 3 280 179.

The aqueous basic solution of the present invention is defined according to the characterized portion of claim 1 and the method of making a storage stable aqueous basic solution of the present invention is defined according to the characterizing portion of claim 11.

The amphoteric sulfobetaine surfactants used in the present invention are unique in their ability to meet diverse criteria required of them in formulating aqueous basic surfactant solutions.

The sulfobetaines used in the present invention can be represented conventionally by the following general structure:



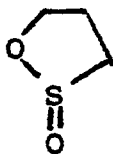
Where R₁ is a C₆–C₁₂ alkyl group;

R₂ and R₃ are a methyl group, a 2-hydroxy ethyl group, or a 2-hydroxy propyl group; and

R₄ is H or OH.

Within the alkyl group range of C₆–C₁₂ the sulfobetaine possesses the requisite degree of water solubility and surfactancy required of it. While various reaction schemes may be envisioned for synthesis of the alkyl sulfobetaines of the present invention, the following two-step reaction scheme currently is favoured where R₄ is OH. The initial step involves the formation of an epichlorhydrin/-bisulfite intermediate. This reaction conveniently is conducted in water in the presence of a base (for example, sodium hydroxide) at relatively moderate reaction temperature (e.g. 49°–93°C (120°–200°F)) and preferably under inert atmosphere.

Following the formation of the epichlorhydrin/bisulfite intermediate, such intermediate is reacted with the appropriate amine for forming the product sulfobetaine. This second reaction step is conducted at reaction temperatures ranging from 37.8°C to 93°C (100° to 200°F). Unreacted material then can be neutralized and/or removed and the pH and percent non-volatile solids of the reaction product adjusted as is necessary, desirable, or convenient in conventional fashion. For sulfobetaine where R₄ is H, a propyl sulfone,



is reacted with the appropriate amine. The resulting alkyl sulfobetaine is soluble in caustic solution at concentrations ranging up to 10% by weight and greater. Moreover, such solubility is present even at elevated levels of potassium hydroxide, sodium hydroxide, or like bases ranging in concentrations of greater than 30%, advantageously 30%–50%, and preferably 40%–50%.

5 A variety of bases may be used in conjunction with the sulfobetaines of the present invention. Such bases include, for example, sodium hydroxide, potassium hydroxide, calcium hydroxide, calcium oxide, sodium metasilicate, tetrapotassium pyrophosphate, sodium tripolyphosphate, trisodium phosphate, potassium silicate, and the like, and even mixtures thereof. As the Example will demonstrate, the alkyl dimethyl hydroxy sulfobetaines user in the present invention are stable in potassium hydroxide and sodium hydroxide solutions ranging up to 40–50 percent concentration.

10 The aqueous basic solutions of sulfobetaines of the present invention find use in a variety of applications. Such applications include, for example, bottle washing compounds, hot vat cleaning compounds, paper pulping, paint strippers, railroad and aircraft cleaners, dairy and food plant cleaners, detergent sanitizers, polymer-based wax strippers, and the like. The excellent stability, surfactancy, and low foaming characteristics of the alkyl dimethyl sulfobetaine caustic solutions make them useful in these and a variety of additional applications.

15 The following Example shows how the present invention can be practiced but should not be construed as limiting. All percentages and proportions are by weight in this application unless otherwise expressly indicated.

20 EXAMPLES

EXAMPLE 1

25 Lauryldimethyl sulfobetaine ($R_4 = OH$) was made by a two-step process described herein. The first step involved the charging of a small Parr reactor with sodium bisulfite (242 g), epichlorohydrin (228 g), deionized water (910 g), and solid sodium hydroxide (2 g). The water and base were mixed and nitrogen sparged to remove dissolved oxygen prior to charging the reactor. The reactor was pressurized to 0.14 MPa (20 psi) with nitrogen and heated to 53°C (125°F) at which point the reaction exothermed to a reaction temperature of 60°–66°C (140°–150°F). The reaction was conducted for one hour and then sampled for determination of free sodium bisulfite. After the one hour reaction time, this analysis showed that the percent free sodium bisulfite was 0.2 percent. The reactor was cooled to 37.8°C (100°F) and the product removed as the reaction was judged to be complete.

30 1310 g of the thus-formed intermediate then was added to a three liter-four necked flask along with 416 g of lauryldimethyl amine. The flask was heated to 66°C–71°C (150°–160°F) and maintained at this temperature while the contents in the flask were stirred. After six hours reaction time, the contents in the flask changed from a milky liquid to a clear liquid. The reaction was continued for a total of 18 hours at which point the reaction was judged to be essentially complete. Sodium hydroxide (18 g, 50% concentration) was added to the flask and the temperature increased to 82°C (180°F) to hydrolyze unreacted epichlorohydrin/bisulfite intermediate. After two hours reaction time, the flask again was sampled and analyzed for percent free in NaCl which proved to be 8.0 percent. The contents of the flask then were cooled to 37.8°C (100°F) and sufficient sulfuric acid (25% concentration) was added to adjust the pH to about 8–8.5. The final analysis of the lauryldimethyl hydroxy sulfobetaine is set forth below:

45	<u>Description</u>	<u>Results</u>
	Appearance at Room Temperature	Crystal Clear Liquid
	Color (Gardner)	1–
50	pH (5% in deionized water)	8.5
	Percent Free Amine (mw = 220)	0.8
	Percent AH (mw = 257)	0.1
55	Percent Solids	51.7
	Percent NaCl	8.0

60 The lauryldimethyl hydroxy sulfobetaine was tested for solubility in aqueous potassium hydroxide solution. Concentrations of potassium hydroxide at 10%, 20%, 30%, 40%, and 50% solutions were formulated at percent solids content of lauryldimethyl hydroxy sulfobetaine of 1%, 3%, 5%, and 10%. The lauryldimethyl hydroxy sulfobetaine was judged to be soluble at all concentrations of sulfobetaine at all concentrations of potassium hydroxide. The lauryldimethyl hydroxy sulfobetaine then was subjected to Ross-

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Miles foam test at 1.0% by weight actives in 72°F distilled water. The following foam heights were measured: initial, 205 mm; and +5 minutes, 26 mm. In 150 ppm hard (Ca) water at 1% concentration, Ross-Miles foam heights were: initial, 200 mm; and +5 minutes, 29 mm. Thus, it will be seen that the lauryldimethyl hydroxy sulfobetaine is low foaming as well as soluble in high concentrations of potassium hydroxide.

Next, the stability of the lauryldimethyl sulfobetaine to alkaline solutions was evaluated. Initial samples of the lauryldimethyl hydroxy sulfobetaine at 1%, 3%, and 5% by weight of a 50% solids solution of the sulfobetaine was established for 40% sodium hydroxide solutions. Surface tension and interfacial tension (against refined mineral oil, Nujol oil) were recorded initially, after one month storage in polyethylene bottles, and after 6 months of storage in polyethylene bottles. Samples for the tension evaluation were prepared by diluting the concentrate to 3% sodium hydroxide in deionized water for taking the measurements. The following results were recorded:

Surface Tension and Interfacial Tension Measurements

Lauryldimethyl Sulfobetaine (% weight)*	m N/m (Dynes/cm)					
	Initial		One Month Storage		Six Month Storage	
	Surface Tension	Interfacial Tension	Surface Tension	Interfacial Tension	Surface Tension	Interfacial Tension
0	55.4	16.5	55.6	16.8	58.0	—
1	40.8	12.4	34.7	12.5	36.2	16.2
3	26.5	9.0	23.5	4.7	27.5	7.2
5	24.0	6.8	22.4	5.1	26.1	6.8

* % by weight sulfobetaine of a 50% solids solution of the sulfobetaine, 40% NaOH, which was stored and then diluted to 3% NaOH for these tests.

The above-tabulated results clearly demonstrate that the lauryldimethyl hydroxy sulfobetaine remains virtually unaffected when stored for time periods of up to six months in concentrated sodium hydroxide solutions. Thus, the lauryldimethyl hydroxy sulfobetaine has been demonstrated to be soluble in concentrated alkaline solutions, storage stable in concentrated alkaline solutions, and low foaming.

EXAMPLE 2

An octyl dimethyl hydroxy sulfobetaine was made in a manner like that described in Example 1. At 5% by weight sulfobetaine, Ross-Miles foam heights in deionized water were: initial, 47 mm; and +5 minutes, 40 mm. In 150 ppm (Ca) hard water, Ross-Miles foam heights were: initial, 43 mm; and +5 minutes, 36 mm. The low foaming properties of this betaine is demonstrated.

Samples of the octyl dimethyl hydroxy sulfobetaine were compounded at 1%, 0.1%, 0.025%, and 0.01% solids in DI Water for tension measurements. The following results were recorded.

Surface Tension and Interfacial Tension Measurements

Octyl Dimethyl Hydroxy Sulfobetaine (% solids)	m N/m (Dynes/cm)	
	Surface Tension	Interfacial Tension
0.01	57.7	30.5
0.025	45.5	19.3
0.1	28.9	6.4
1.0	23.8	2.4

These results clearly demonstrate the excellent surfactancy of the octyl dimethyl hydroxy sulfobetaine.

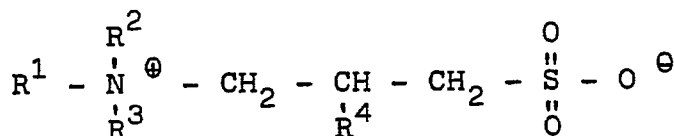
Solubility of the octyl dimethyl hydroxy sulfobetaine to the alkaline solutions was evaluated by dissolving the surfactant into a 50% NaOH solution at active levels of 1, 3 and 5%. The following results were obtained:

Surface Tension and Interfacial Tension Measurements

		m N/m (Dynes/cm)			
5	Octyl Dimethyl Hydroxy Betaine (% weight)	Initial	4 Day Storage		
		Surface Tension	Interfacial Tension	Surface Tension	Interfacial Tension
	1	43.9	19.4	39.5	14.1
	3	34.4	11.9	31.4	9.5
10	5	30.2	10.1	29.6	8.3

Claims

1. An aqueous basic solution of a base, water, and a betaine having the following general structure:



characterized in being improved for storage stability at a calculated pH of greater than 13 and in that R₁ is a C₆-C₁₂ alkyl group; R₂ and R₃ are a methyl group, a 2-hydroxy ethyl group, or a 2-hydroxy propyl group; and R₄ is H or OH; and said base is present in an amount to achieve a calculated pH of greater than 13.

2. The basic solution of claim 1 characterized in that said base is selected from the group consisting of sodium hydroxide, potassium hydroxide, sodium metasilicate, tetrapotassium pyrophosphate, sodium tripolyphosphate, trisodium phosphate, potassium silicate, and mixtures thereof.

3. The basic solution of claim 2 characterized in that said base is selected from potassium hydroxide, sodium hydroxide, or mixtures thereof.

4. The basic solution of claim 1 characterized in that said base is at a concentration of greater than 30% by weight.

5. The basic solution of claim 1 characterized in that R₁ is a C₆-C₁₀ alkyl group.

6. The basic solution of claim 1 characterized in that R₁ is a C₈ alkyl group.

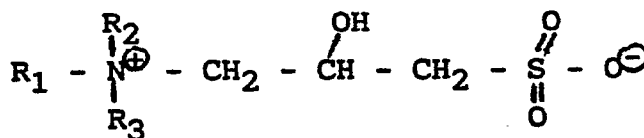
7. The basic solution of claim 1 characterized in that said sulfobetaine ranges in concentration from between about 0.05% and 10% by weight.

8. The basic solution of claim 4 characterized in that said base ranges in concentration from between about 30% and 50% by weight.

9. The basic solution of claim 1 characterized in that R₁ and R₂ are Me.

10. The basic solution of claim 9 characterized in that R₄ is OH.

11. A method for making a storage stable aqueous basic solution characterized in having a calculated pH of greater than 13 of a sulfobetaine of the following general structure:



where R₁ is a C₆-C₁₂ alkyl group,

R₂ and R₃ are CH₃, 2-hydroxy ethyl or 2-hydroxy propyl, comprising

(a) forming an aqueous epichlorohydrin/bisulfite intermediate;

(b) reacting said intermediate and a C₆-C₁₂ alkyl, R₂, R₃ amine in an aqueous reaction mixture; and

(c) adding a base to the thus-formed aqueous solution of said sulfobetaine if not already present therein to achieve said calculated pH of greater than 13.

12. The method of claim 11 characterized in that R₁ is a C₆-C₁₀ alkyl group.

13. The method of claim 11 characterized in that R₁ is a C₈ alkyl group.

14. The method of claim 11 characterized in that said base is selected from the group consisting of sodium hydroxide, potassium hydroxide, sodium metasilicate, tetrapotassium pyrophosphate, sodium tripolyphosphate, trisodium phosphate, potassium silicate, and mixtures thereof.

15. The method of claim 14 characterized in that said base is selected from potassium hydroxide, sodium hydroxide, or mixtures thereof.

16. The method of claim 11 characterized in that the proportion of said sulfobetaine and said aqueous basic solution ranges from between about 0.05 and 10 percent by weight.

17. The method of claim 11 characterized in that said intermediate is formed at a reaction temperature of between 49°C and 93°C (120° and 200°F).

18. The method of claim 11 characterized in that said amine/intermediate reaction is conducted at a temperature of between 37.8°C and 93°C (100° and 200°F).

5 19. The method of claim 11 characterized in that sufficient base is added to result in at least 30% base concentration by weight.

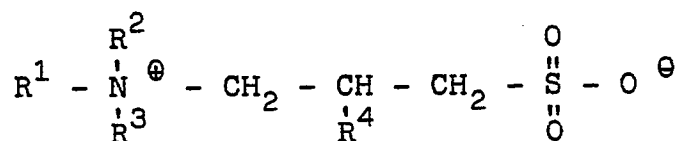
20. The method of claim 19 characterized in that said base concentration is between about 30% and 50% by weight.

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Patentansprüche

1. Wäßrige basische Lösung einer Base, Wasser und eines Betains der allgemeinen Formel

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gekennzeichnet durch eine verbesserte Lagerfähigkeit bei einem berechneten pH-Wert von größer als 13 und dadurch, daß R_1 eine C_6 - C_{12} -Alkylgruppe ist, R_2 und R_3 sind Methylgruppen, eine 2-Hydroxyethylgruppe oder eine 2-Hydroxypropylgruppe und R_4 ist H oder OH, wobei die Base in einer Menge zum Erreichen eines berechneten pH-Wertes von größer als 13 vorliegt.

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2. Basische Lösung nach Anspruch 1, dadurch gekennzeichnet, daß die Base aus der aus Natriumhydroxid, Kaliumhydroxid, Natriummetasilicat, Tetrakaliumpyrophosphat, Natriumtripolyphosphat, Trinatriumphosphat, Kaliumsilicat und Mischungen davon bestehenden Gruppen ausgewählt ist.

3. Basische Lösung nach Anspruch 2, dadurch gekennzeichnet, daß die Base aus Kaliumhydroxid, Natriumhydroxid oder Mischungen davon ausgewählt ist.

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4. Basische Lösung nach Anspruch 1, dadurch gekennzeichnet, daß die Base in einer Konzentration von größer als 30 Gew.-% vorliegt.

5. Basische Lösung nach Anspruch 1, dadurch gekennzeichnet, daß R_1 eine C_6 - C_{10} -Alkylgruppe ist.

6. Basische Lösung nach Anspruch 1, dadurch gekennzeichnet, daß R_1 eine C_8 -Alkylgruppe ist.

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7. Basische Lösung nach Anspruch 1, dadurch gekennzeichnet, daß die Konzentration des Sulfobetains in einem Bereich zwischen etwa 0,05 und 10 Gew.-% liegt.

8. Basische Lösung nach Anspruch 1, dadurch gekennzeichnet, daß die Konzentration der Base in einem Bereich zwischen etwa 30 und 50 Gew.-% liegt.

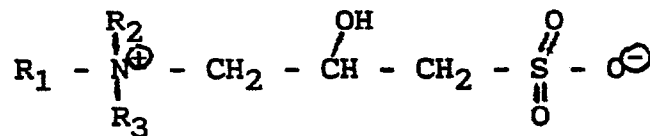
9. Basische Lösung nach Anspruch 1, dadurch gekennzeichnet, daß R_1 und R_2 Me sind.

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10. Basische Lösung nach Anspruch 1, dadurch gekennzeichnet, daß R_4 OH ist.

11. Verfahren zur Herstellung einer wäßrigen basischen Lösung, gekennzeichnet durch einen berechneten pH-Wert von größer als 13 und ein Sulfobetain der allgemeinen Formel

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50 worin R_1 eine C_6 - C_{12} -Alkylgruppe ist, R_2 und R_3 CH_3 , 2-Hydroxyethyl oder 2-Hydroxypropyl sind, umfassend

(a) die Bildung eines wäßrigen Epichlorhydrin/Bisulfitzwischenprodukts,

(b) die Umsetzung des Zwischenprodukts und eines C_6 - C_{12} -Alkyl-, R_2 -, R_3 - Amins in einer wäßrigen Reaktionsmischung und

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(c) das Hinzufügen einer Base zu der so gebildeten wäßrigen Lösung des Sulfobetains, falls darin noch nicht enthalten, zum Erreichen des berechneten pH-Wertes von größer als 13.

12. Verfahren nach Anspruch 11, dadurch gekennzeichnet, daß R_1 eine C_6 - C_{10} -Alkylgruppe ist.

13. Verfahren nach Anspruch 11, dadurch gekennzeichnet, daß R_1 eine C_8 -Alkylgruppe ist.

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14. Verfahren nach Anspruch 11, dadurch gekennzeichnet, daß die Base aus der aus Natriumhydroxid, Kaliumhydroxid, Natriummetasilicat, Tetrakaliumpyrophosphat, Natriumtripolyphosphat, Trinatriumphosphat, Kaliumsilicat und Mischungen davon bestehenden Gruppe ausgewählt ist.

15. Verfahren nach Anspruch 14, dadurch gekennzeichnet, daß die Base aus Kaliumhydroxid, Natriumhydroxid oder Mischungen davon ausgewählt ist.

16. Verfahren nach Anspruch 11, dadurch gekennzeichnet, daß der Anteil des Sulfobetains und der wäßrigen basischen Lösung in einem Bereich zwischen etwa 0,05 und 10 Gew.-% liegt.

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17. Verfahren nach Anspruch 11, dadurch gekennzeichnet, daß das Zwischenprodukt bei einer Reaktionstemperatur zwischen 49°C und 93° (120° und 200°F) gebildet wird.

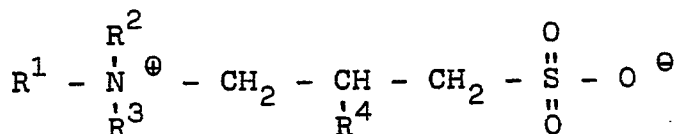
18. Verfahren nach Anspruch 11, dadurch gekennzeichnet, daß die Amin/Zwischenproduktreaktion bei einer Temperatur von zwischen 37,8°C und 93°C (100° und 200°F) durchgeführt wird.

19. Verfahren nach Anspruch 11, dadurch gekennzeichnet, daß ausreichend Base hinzugefügt wird, um eine Basenkonzentration von wenigstens 30 Gew.-% zu erzielen.

20. Verfahren nach Anspruch 19, dadurch gekennzeichnet, daß die Konzentration der Base in einem Bereich zwischen etwa 30 und 50 Gew.-% liegt.

Revendications

1. Solution basique aqueuse d'une base, d'eau et d'une bétaine répondant à la structure générale suivante:



caractérisée en ce qu'elle est améliorée pour la stabilité à la conservation à un pH calculé supérieur à 13 et en ce que R¹ représente un groupe alkyle en C₆-C₁₂; R² et R³ représente chacun un groupe méthyle, un groupe 2-hydroxyéthyle ou un groupe 2-hydroxy-propyle; et R⁴ représente H ou OH, tandis que cette base est présente en une quantité permettant d'obtenir un pH calculé supérieur à 13.

2. Solution basique selon la revendication 1, caractérisée en ce que cette base est choisie parmi le groupe comprenant l'hydroxyde de sodium, l'hydroxyde de potassium, le métasilicate de sodium, le pyrophosphate tétrapotassique, le tripolyphosphate de sodium, le phosphate trisodique, le silicate de potassium et leurs mélanges.

3. Solution basique selon la revendication 2, caractérisée en ce que cette base est choisie parmi l'hydroxyde de potassium, l'hydroxyde de sodium ou leurs mélanges.

4. Solution basique selon la revendication 1, caractérisée en ce que cette base est présente en une concentration supérieure à 30% en poids.

5. Solution basique selon la revendication 1, caractérisée en ce que R¹ est un groupe alkyle en C₆-C₁₀.

6. Solution basique selon la revendication 1, caractérisée en ce que R¹ est un groupe alkyle en C₈.

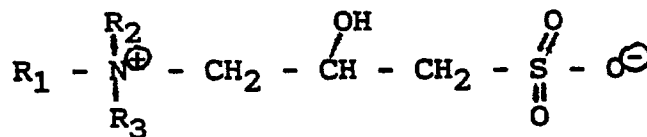
7. Solution basique selon la revendication 1, caractérisée en ce que la concentration de cette sulfobétaine se situe entre environ 0,05 et 10% en poids.

8. Solution basique selon la revendication 4, caractérisée en ce que la concentration de cette base se situe entre environ 30 et 50% en poids.

9. Solution basique selon la revendication 1, caractérisée en ce que R¹ et R² représentent chacun Me.

10. Solution basique selon la revendication 9, caractérisée en ce que R⁴ représente OH.

11. Procédé de préparation d'une solution basique aqueuse stable à la conservation, caractérisé en ce que la solution a un pH calculé de plus de 13 d'une sulfobétaine répondant à la structure générale suivante:



où R¹ représente un groupe alkyle en C₆-C₁₂, R² et R³ représentent chacun CH₃, un groupe 2-hydroxyéthyle ou un groupe 2-hydroxy-propyle, caractérisé en ce qu'il comprend les étapes qui consistent à:

(a) former un produit intermédiaire aqueux d'épichlorhydrine/bisulfite;

(b) faire réagir ce produit intermédiaire et une alkyle(en C₆-C₁₂)amine R², R³ dans un mélange réactionnel aqueux; et

(c) ajouter une base à la solution aqueuse ainsi formée de cette sulfobétaine si tant est qu'elle n'y est déjà pas présente, pour obtenir le pH calculé supérieur à 13.

12. Procédé selon la revendication 11, caractérisé en ce que R¹ représente un groupe alkyle en C₆-C₁₀.

13. Procédé selon la revendication 11, caractérisé en ce que R¹ représente un groupe alkyle en C₈.

14. Procédé selon la revendication 11, caractérisé en ce que cette base est choisie parmi le groupe comprenant l'hydroxyde de sodium, l'hydroxyde de potassium, le métasilicate de sodium, le pyrophos-

phate tétrapotassique, le tripolyphosphate de sodium, le phosphate trisodique, le silicate de potassium et leurs mélanges.

15. Procédé selon la revendication 14, caractérisé en ce que cette base est choisie parmi l'hydroxyde de potassium, l'hydroxyde de sodium ou leurs mélanges.

5 16. Procédé selon la revendication 11, caractérisé en ce que la proportion de la sulfobétaïne et de la solution basique aqueuse se situe entre environ 0,05 et 10% en poids.

17. Procédé selon la revendication 11, caractérisé en ce que le produit intermédiaire est formé à une température réactionnelle se situant entre 49 et 93°C (120 et 200°F).

10 18. Procédé selon la revendication 11, caractérisé en ce que la réaction amine/produit intermédiaire est effectuée à une température se situant entre 37,8°C et 93°C (100 et 200°F).

19. Procédé selon la revendication 11, caractérisé en ce que la base suffisante est ajoutée pour obtenir au moins une concentration de 30% en poids de la base.

20. Procédé selon la revendication 19, caractérisé en ce que la concentration de la base se situe entre environ 30 et 50% en poids.

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