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AUSTRALIA  
Patents Act 1990

654508

PATENT REQUEST: STANDARD PATENT

We, COLGATE-PALMOLIVE COMPANY, being the person(s) identified below as the Applicant, request the grant of a standard patent to the person identified below as the Nominated Person, for an invention described in the accompanying complete specification.

Full application details follow.

Applicant: COLGATE-PALMOLIVE COMPANY  
a Delaware corporation

Address: 300 Park Avenue New York New York 10022 United States of America

Nominated Person: COLGATE-PALMOLIVE COMPANY  
a Delaware corporation

Address: 300 Park Avenue New York New York 10022 United States of America

Invention Title: "CONCENTRATED LIQUID DETERGENT COMPOSITION CONTAINING ALKYL BENZENE SULFONATE AND MAGNESIUM"

Name(s) of Actual Inventor(s): GARY J. JAKUBICKI; DIRK WARSCHESKI

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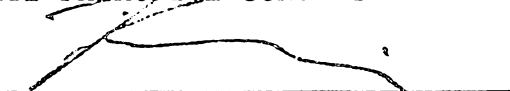
BASIC CONVENTION APPLICATION(S) DETAILS

<u>Application No</u>	<u>Country</u>	<u>Country Code</u>	<u>Date of Application</u>
616496	United States of America	US	21 November 1990

We are not an eligible person described in Section 33 - 36 of the Act.

Dated this 7 day of November 1991

COLGATE-PALMOLIVE COMPANY

By:   
Registered Patent Attorney

REPRINT OF RECEIPT  
8025521 07/11/91

NOTICE OF ENTITLEMENT  
(To be filed before acceptance)

I/We Robert C. Sullivan - Vice President and Chief Patent Counsel  
of Colgate-Palmolive Company of 300 Park Avenue New York  
New York 10022 United States of America

being the \*applicant/\* authorised by the applicant in respect of an application for  
a patent for an invention entitled CONCENTRATED LIQUID DETERGENT  
COMPOSITION CONTAINING ALKYL BENZENE SULFONATE AND MAGNESIUM  
filed under Application No. \_\_\_\_\_, state the following:-

Part 1 - Must be completed for all applications.

The person(s) nominated for the grant of the patent:

\*is/\*are the actual inventor(s)

or

has, for the following reasons, gained entitlement from the actual inventor(s):  
Colgate-Palmolive, ~~XXXX~~ Company is the assignee of the invention  
from the actual inventor(s)

Part 2 - Must be completed if the application is a Convention application.

The person(s) nominated for the grant of the patent \*is/\*are:

the applicant(s) of the basic application(s) listed on the patent request form

or

entitled to rely on the basic application(s) listed on the patent request form  
by reason of the following: Company  
Colgate-Palmolive, ~~XXXX~~ is the assignee of the basic  
application(s) from the actual inventor(s)

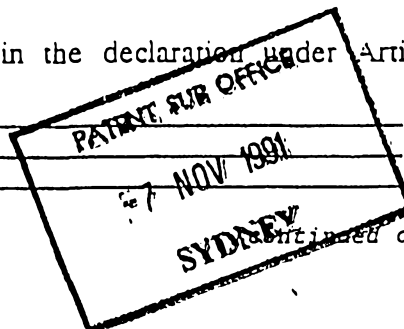
~~Part 3 - Must be completed if the application was made under the PCT and  
claims priority.~~

~~The person(s) nominated for the grant of the patent \*is/\*are:~~

~~the applicant(s) of the application(s) listed in the declaration under Article 8  
of the PCT~~

~~or~~

~~entitled to rely on the application(s) listed in the declaration under Article 8  
of the PCT by reason of the following:-~~

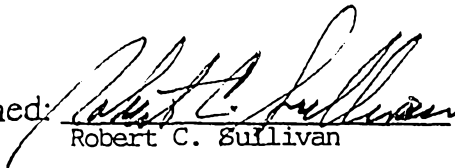


Part 4 - Must be completed if the application is a Convention application, or the application was made under the PCT and the applicant made a declaration under Article 8 of the PCT in respect of the basic application.

Except as stated in the next paragraph, the basic application(s) ~~listed on the patent request form/referred to in the declaration under Article 8 of the PCT~~ is/are the application(s) first made in a Convention country in respect of the invention.

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Signed:

  
Robert C. Sullivan

Date:

OCT 9 1991

Status: Vice President and Chief Patent Counsel



**(12) PATENT ABRIDGMENT (11) Document No. AU-B-87706/91**  
**(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 654508**

(54) Title  
**CONCENTRATED LIQUID DETERGENT COMPOSITION CONTAINING ALKYL BENZENE SULFONATE AND MAGNESIUM**

(51)<sup>5</sup> International Patent Classification(s)  

C11D 001/65	C11D 001/12	C11D 001/14	C11D 001/22
C11D 001/29	C11D 001/46	C11D 003/04	C11D 003/20
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(21) Application No. : **87706/91** (22) Application Date : **07.11.91**

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(57) Claim

1. A liquid dishwashing detergent composition providing stable foaming characteristics and which is especially effective in removing greasy soils, said composition comprising:

- (A) from 20 to 70% by weight of a surfactant system comprising
  - (a) from 15 to 35% by weight of the magnesium salt of a C<sub>10</sub>-C<sub>18</sub> alkylbenzene sulfonic acid anionic surfactant;
  - (b) from 1 to 5% by weight of a water-soluble magnesium salt, in an amount such that the molar ratio of total magnesium ion from (a) and (b) to alkylbenzene sulfonate ranges from 0.65:1.0 to 1.0:1.0;
  - (c) from 1 to 20% by weight of a water-soluble C<sub>10</sub>-C<sub>16</sub> primary alkyl ethoxy sulfate salt containing an average of from 1 to 6 ethylene oxide groups per alkyl group in the alkyl ether sulfate; and
  - (d) from 0 to 20% by weight of a primary,

secondary or tertiary amine or alkali metal salt of C<sub>10</sub>-C<sub>18</sub> alkyl sulfate; and

- (B) from 0.5 to 8% by weight of a suds promoting agent selected from the group consisting of C<sub>10</sub>-C<sub>18</sub> ethoxylated and non-ethoxylated mono-or di-C<sub>1</sub>-C<sub>5</sub> alkanolamides and C<sub>12</sub>-C<sub>14</sub> alkyl amides condensed with up to about 15 moles ethylene oxide per mole of amide;
- (C) from 0 to 10% by weight of a low irritant organic solvent;
- (D) from 0 to 10% by weight hydrotrope; and
- (E) from 0 to 10% by weight of one or more optional additive chosen from chelating agents, coloring agents, dyes, perfumes, bactericides, fungicides, preservatives, suncreening agents, pH modifiers, pH buffering agents, opacifiers, antioxidants, proteins and
- (F) the balance, water.

4. A physically stable liquid dishwashing detergent base composition comprising:

- (A) from 30 to 70% by weight deterative active ingredients comprising
  - (a) from 10 to 50% by weight of the magnesium salt of a C<sub>10</sub>-C<sub>18</sub> alkylbenzene sulfonite acid;
  - (b) from 1 to 5% by weight of a water-soluble magnesium salt, in an amount such that the molar ratio of total magnesium ion from (a) and (b) to alkylbenzene sulfonate ranges from 0.65:1.0 to 1.0:1.0;
  - (c) from 1 to 27% by weight of a water-soluble C<sub>10</sub>-C<sub>16</sub> primary alkyl ethoxy sulfate salt containing an average of from 1 to 6 ethylene oxide groups per alkyl group;
  - (d) from 0 to 27% by weight of a primary amine or alkali metal salt of a C<sub>10</sub>-C<sub>16</sub> alkyl sulfate; and

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(10) 654508

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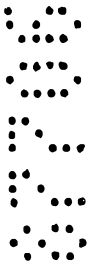
- (B) from 0.6 to 11% by weight of a suds promoting agent selected from the group consisting of a C<sub>10</sub>-C<sub>16</sub> mono- and di-C<sub>1</sub>-C<sub>5</sub> alkanolamides and C<sub>12</sub>-C<sub>14</sub> alkyl amides condensed with up to 15 moles ethylene oxide per mole of amide;
- (C) from 0 to 10% by weight of a low irritant organic solvent;
- (D) from 0 to 8% by weight of a hydrotrope; and
- (E) the balance, water.

AUSTRALIA

Patents Act 1990

COLGATE-PALMOLIVE COMPANY

ORIGINAL  
COMPLETE SPECIFICATION  
STANDARD PATENT



Invention Title:

CONCENTRATED LIQUID DETERGENT COMPOSITION CONTAINING  
ALKYL BENZENE SULFONATE AND MAGNESIUM



The following statement is a full description of this invention including the best method of performing it known to us:-

Field Of The Invention:

This invention relates to aqueous liquid detergent compositions containing alkyl benzene sulfonate and added detergent boosting levels of magnesium ions.

Description Of The Prior Art:

Light-duty liquid detergent compositions, such as those suitable for use in washing dishes, are well known. Performance of such compositions is a measurement of both foam mileage, i.e. number of dishes washed, and grease cutting ability. The liquid dishwashing detergent compositions presently on the market are designed to remove oily/greasy soils from glasses, dishes, and other tableware and kitchen utensils while maintaining an acceptable layer of suds.

Liquid detergent formulations containing magnesium salts and magnesium surfactants show enhanced performance, as is disclosed in U.S. Patents 2,908,651 and 2,437,253. Certain combinations of active ingredients can also provide a range of enhanced detergent properties. An enhanced performance liquid detergent composition, disclosed in U.S. Patent 4,435,317, contains (a) a C<sub>10</sub>-C<sub>16</sub> alkyl sulfate, (b) a C<sub>10</sub>-C<sub>16</sub> alkyl ethoxy sulfate and (c) a linear C<sub>10</sub>-C<sub>16</sub> alkyl benzene sulfonate in a ratio of the total weight of (a) + (b) to the weight of (c) of less than or equal to 33:1, and containing magnesium in a molar amount corresponding to 0.20x-0.70x where x is the number of moles of alkyl sulfate. The amount of magnesium salts that can be added to the compositions disclosed in U.S. Patent 4,435,317



is limited because the salts raise the temperatures at which inorganic salt crystals form in the compositions upon cooling.

Enhanced performance in relation to grease cutting ability and foam mileage are also achieved by increasing the concentration of surfactant active ingredients in the liquid detergent formulation. There are, however, several problems associated with the preparation of liquid detergent compositions containing high proportions of active detergent materials in order to satisfy the requirements of clarity, viscosity and stability of the product.

Generally, it is necessary to add a fairly high proportion of hydrotrope to those detergent compositions that have a high concentration of active detergent materials.

Hydrotropes are fairly expensive and are generally inactive materials and thus do not contribute to detergency but have the effect of rendering the active ingredients water soluble and the composition homogeneous at those temperatures normally encountered in transport and storage. Hydrotropes generally lower the "cloud point" of liquid detergent compositions, thus maintaining a clear liquid composition at temperatures at which a detergent composition lacking hydrotrope would become cloudy and unattractive in appearance. U.S. Patent 4,235,758 discloses that the use of a magnesium alkylbenzene sulfonate derived from a linear C<sub>10</sub>-C<sub>13</sub> alkylbenzene of average molecular weight of 220-250 significantly reduces the requirement for hydrotrope at any level of active detergent material.

The present invention provides compositions comprised of certain specific detergent active ingredients, in unique combinations and proportions, which are unexpectedly capable of

superior performance to other liquid detergents in relation to both foam mileage and grease cutting.

It is, accordingly, an object of this invention to provide physically stable liquid detergent compositions that offer superior grease cutting and foam mileage performance.

It is another object of this invention to provide detergent compositions containing magnesium, alkylbenzene sulfonate and added detergency boosting levels of magnesium ions, which are especially efficient in removing greasy soils, and which have a reduced requirement for hydrotropes to maintain a suitable cloud point.

Still another object of this invention is to provide a concentrated detergent base composition containing detergency boosting levels of magnesium ions and alkyl benzene sulfonate which may be stored as a concentrated liquid base composition and which may be manufactured using conventional low shear liquid mixing equipment.

#### SUMMARY OF THE INVENTION

According to the present invention, there is provided an aqueous liquid detergent composition especially suitable for use in dishwashing, and containing

(A) from 20 to 70%, preferably 30 to 55%, of a surfactant mixture of (a) from about 15 to about 35% by weight of the magnesium salt of a C<sub>10</sub>-C<sub>18</sub> alkylbenzene sulfonic acid, (b) from about 1 to 5% by weight of a magnesium salt, in an amount such that the molar ratio of total magnesium ion to alkylbenzene sulfonate ranges from about 0.65:1.0 to 1.0:1.0, (c) from about 1 to about 20% by weight of a water-soluble C<sub>10</sub>-C<sub>16</sub> primary alkyl ethoxy sulfate containing an average of from about 1 to 6 ethylene oxide groups per alkyl group in the alkyl ether

sulfate and, (d) from about 0 to 20% by weight of a primary, secondary or tertiary amine or alkali metal salt of C<sub>10</sub>-C<sub>16</sub> alkyl sulfate; and

5 (B) from about 0.5 to about 8% by weight of a suds promoting agent selected from the group consisting of C<sub>10</sub>-C<sub>18</sub> ethoxylated and non-ethoxylated mono- or di- C<sub>1</sub>-C<sub>5</sub> alkanolamides and C<sub>12</sub>-C<sub>14</sub> alkyl amides condensed with up to about 15 moles ethylene oxide per mole of amide, and balance, primarily water.

10 In a preferred embodiment the composition may also include:

(C) from about 0 to about 10% by weight of a low irritant organic solvent; and

(D) from about 0 to about 10% by weight of a hydrotrope. The compositions may also include

15 (E) other adjuvants, such as chelating agents, coloring agents, dyes, perfumes, bactericides, fungicides, preservatives, sunscreens agents, pH modifiers or buffering agents, opacifiers, antioxidants, proteins, and the like.

~~The present invention also provides a pourable liquid~~  
20 detergent base composition of concentrated light duty liquid detergents containing alkylbenzene sulfonic acid and detergency boosting levels of magnesium ions and which can be stored at  
... ambient temperature for future use without gelling.

#### DETAILED DESCRIPTION OF THE INVENTION

25 Detergent compositions in accordance with the present invention comprise a mixture of two or three essential anionic surfactants of defined constitution and proportions and have a specified molar ratio of magnesium ion to alkylbenzene sulfonate

~~ranging from 0.65:1.0 to 1.0:1.0.~~



The present invention also provides a pourable liquid detergent base composition of concentrated light duty liquid detergents containing alkylbenzene sulfonic acid and detergency boosting levels of magnesium ions and which can be stored at ambient temperature for future use without gelling. That is, the invention also provides a physically stable liquid dishwashing detergent base composition comprising:

- 10 (A) from 30 to 70% by weight deterative active ingredients comprising
- (a) from 10 to 50% by weight of the magnesium salt of a C<sub>10</sub>-C<sub>18</sub> alkylbenzene sulfonite acid;
- 15 (b) from 1 to 5% by weight of a water-soluble magnesium salt, in an amount such that the molar ratio of total magnesium ion to alkylbenzene sulfonate ranges from 0.65:1.0 to 1.0:1.0;
- 20 (c) from 1 to 27% by weight of a water-soluble C<sub>10</sub>-C<sub>16</sub> primary alkyl ethoxy sulfate salt containing an average of from 1 to 5 ethylene oxide groups per alkyl group;
- 25 (d) from 0 to 27% by weight of a primary amine or alkali metal salt of a C<sub>10</sub>-C<sub>16</sub> alkyl sulfate; and
- (B) from 0.6 to 11% by weight of a suds promoting agent selected from the group consisting of a C<sub>10</sub>-C<sub>16</sub> mono- and di-C<sub>1</sub>-C<sub>5</sub> alkanolamides and C<sub>12</sub>-C<sub>14</sub> alkyl amides condensed with up to 15 moles ethylene oxide per mole of amide;
- 30 (C) from 0 to 10% by weight of a low irritant organic solvent;
- (D) from 0 to 3% by weight of a hydrotrope; and
- (E) the balance, water.



DETAILED DESCRIPTION OF THE INVENTION

Detergent compositions in accordance with the present invention comprise a mixture of two or three essential anionic surfactants of defined constitution and proportions and have a specified molar ratio of magnesium ion to alkylbenzene sulfonate ranging from 0.65:1.0 to 1.0:1.0.

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The first essential surfactant ingredient is a water-soluble salt of alkylbenzene sulfonic acid, which may be linear or branched. The alkyl group preferably contains from about 10 to 18 carbon atoms, most preferably from about 11 to 13, especially about 12 carbon atoms, preferably in a linear chain configuration. The alkyl benzene sulfonate may be present as an alkali metal, amine, ammonium, or alkaline earth metal salt, preferably the magnesium salt. Thus, the most preferred alkylbenzene sulfonate is the magnesium salt of linear dodecylbenzene sulfonate.

The acid form of alkylbenzene sulfonic acid can be converted to the magnesium salt form either prior to admixture with the other deterative ingredients or thereafter. The conversion may be accomplished either by direct neutralization with magnesium oxide or hydroxide or by ion exchange between, for example, an alkali metal salt or the ammonium salt of alkylbenzene sulfonate and a water-soluble alkaline earth metal salt. Preferably, a source of magnesium ions is added to the detergent mixture to form the desired salt of alkylbenzene sulfonate. Magnesium chlorides, magnesium sulfates, magnesium acetates or magnesium hydroxides are examples of magnesium sources. The preferred magnesium sources are magnesium oxides or hydroxides and sulfates.

The alkylbenzene sulfonate component is present at a level of from about 15 to 35% by weight of the composition, preferably in the range from about 15 to 22%, most preferably in the range from about 19 to 21% by weight.

The point of attachment of the alkyl chain to the benzene nucleus, referred to as the phenyl isomer distribution,

is not critical but, preferably, less than 20% of the alkyl benzene of this invention comprises the 2-phenyl isomer.

A second essential surfactant component of the present composition is a primary alkyl ethoxy sulfate derived from the condensation product of a C<sub>10</sub>-C<sub>16</sub> alcohol with an average of  
5 from about 1 to about 6 ethylene oxide groups, preferably about 2 to 4, especially 3 ethylene oxide groups per mole of alcohol. The C<sub>10</sub>-C<sub>16</sub> alcohols may be derived, for example, from natural fats or Ziegler olefin build-up or Oxo synthesis. It is  
10 preferable to use alkyl ethoxy sulfates and alkyl sulfates of the same carbon chain length, preferably C<sub>12</sub>-C<sub>13</sub>. The level of alkyl ethoxy sulfate in the present composition to achieve the desired foam and deterative properties is about 1-30% by weight, preferably in the range from about 10-20% by weight, based on the  
15 total composition.

The surfactant system of the detergent of the present invention may optionally also include an amount ranging from  
20 0-20% of the total composition, preferably 10-15% by weight, of an alkali metal or ammonium or amine salt of a linear or branched C<sub>10</sub>-C<sub>18</sub> alkyl sulfate anionic surfactant. The cationic counter-ion of the alkyl sulfate may be taken from the group consisting of alkali metals, ammonia and amines, e.g., triethylammonium, triethanolamine (TEA), diethanolamine or monoethanolamine, etc. The alkali metal may be, for example, sodium or potassium,  
25 preferably sodium.

The composition of the present invention contains from about 0.027 to about 0.064 moles of total magnesium ion. Generally, 0.023 to about 0.053 moles of magnesium ion are provided through the incorporation of the magnesium alkylbenzene  
30 sulfonate. Additional magnesium may be added to the composition

in the form of a water-soluble compound, most preferably a salt, such as, for example, magnesium sulfate or magnesium chloride. The preferred magnesium source is magnesium sulfate heptahydrate, added to the composition at about from 1 or 2 to 5% by weight, preferably from about 2.5 to 3.5% by weight, corresponding to 0.01 to 0.014 moles of added magnesium. The total magnesium ion to alkylbenzene sulfonate in the composition is controlled to correspond to a molar ratio of from 0.65:1.0 to 1.0:1.0, preferably from 0.65:1.0 to 0.8:1.0.

The additional magnesium ions added in salt form provide improved grease cutting performance and also lower the cloud point of the formula better than the more expensive hydrotropes. The additional magnesium salt also provides better phase stability to the composition. Using magnesium to boost detergency is known in the art, however, when used in a molar ratio of total magnesium ion to alkylbenzene sulfonate in a range of 0.65:1.0 to 0.8:1.0, the excess magnesium salt not only enhances foam mileage but also increases fluidity, allowing for a decrease in hydrotrope and alcohol levels, thus lowering the concentration of inactive ingredients that do not boost detergency of the composition. This decrease in the level of inactive ingredients leaves room in the composition for an even higher level of deterative active ingredients.

The level of deterative active ingredients in the compositions of this invention is in the range from 20-70% by weight, preferably from 30 to 55%. This increased level of active ingredients results in enhanced foam mileage, thus providing a superior light duty liquid detergent. The usual problems of increased viscosity, decreased stability and product clouding is avoided by using an increased level of magnesium ions



in a molar ratio of magnesium ions to alkylbenzene sulfonate in the range of 0.65:1.0 to 1.0:1.0, preferably from 0.8:1.0 and none or only a low concentration of hydrotrope. The additional magnesium ions in the compositions of this invention also boosts  
5 detergency of the surfactant system.

In general, the consumer views sudsing ability as a measure of cleaning ability. A suds promoting agent, such as, for example, a mono- or di-alkanolamide, may, therefore, be added to the present composition at a level of from 0.5 to about 8% by  
10 weight, preferably about 1 to 6% and, more preferably about 1 to 5% by weight. The preferred suds promoting agent is a mixture of lauric and myristic monoethanolamides. Alternatively, the alkyl group of the amide may be derived from coconut or palm kernel  
15 oil. Suitable alkanolamides include either mono or dialkanolamides having a carbon chain distribution of C<sub>1</sub>-C<sub>5</sub>, preferably C<sub>1</sub>-C<sub>3</sub>, especially mono- or di-ethanolamide. The alkanolamide may be ethoxylated or may be a mixture of non-  
20 ethoxylated alkanolamide and ethoxylated alkanolamide in order to increase the solubility of the alkanolamide in the composition.

Although not essential, clarity and homogeneity of the invention compositions may be improved by inclusion of, for  
25 example, organic solvents or hydrotropes and these and other non-essential additives may be included in the compositions in amounts that do not adversely effect the desired properties.

Organic solvents may be included for their thinning effect, ability to lower the clear point and for solubilization.

Organic solvents, such as, for example isopropanol, n-propanol, ethanol, propylene glycol or mixtures thereof may be included.

When used, the solvent level is usually less than or equal to 10%  
30 by weight, preferably less than 8%, more preferably in the range

of 5 to 8% by weight. Ethanol is the preferred solvent. It is also customary to include a hydrotropic substance in the composition such as, for example, urea, sodium xylene sulfonate, potassium xylene sulfonate, sodium cumene sulfonate, and ammonium xylene sulfonate, and the like in order to assist in solubilizing various components of the composition and maintaining a low clear point. Mixtures of two or more hydrotropes may also be used. The hydrotrope, when used, is generally present in amounts below 8%, preferably below 6%, such as 1 to 6%, especially 2 to 5% by weight.

Various other optional ingredients may also be included in the composition for their desirable aesthetic or functional characteristics. Such ingredients include, for example, opacifying agents to make the composition appear pearly, such as behenic acid or ethylene glycol distearate; perfumes; heavy metal chelating agents such as EDTA; bactericides such as trichlorocarbanilide, tetrachlorosalicylanilide, hexachlorophene, or chlorobromosalicylanilide; antioxidants; thickeners such as guar gum, polyacrylates, polyacrylamide or Irish moss; dyes, water dispersible pigments; preservatives, such as formaldehyde or hydrogen peroxide; pH modifiers, etc. When used, these optional adjuvants may be present in total amounts up to about 10% by weight, preferably up to about 3% by weight, based on the total composition. The pH of the compositions may be within the range from 6 to 8, preferably from about 6.5 to 7.5, and may be adjusted if necessary by addition of suitable acids or bases, such as HCl, NaOH, and the like. Excellent greasy soil removal is attained when an effective amount of the instant compositions is dissolved in an aqueous dishwashing solution. Typical use concentrations are usually at least about

0.05% by weight in water. Of course, this can be adjusted, depending on the soil level and type and desires of the user.

Currently, great commercial interest is shown in the more concentrated liquid detergent compositions. The advantages of highly concentrated liquids are evident, i.e., reduced transport and packaging costs and smaller quantities of product needed by the consumer. The major disadvantage in developing compositions with high active ingredient levels is the difficulty in making the formula due to the increased "solids" concentrations. The word "solids" is used herein to describe all those ingredients of the composition other than solvents, such as water or alcohol.

The compositions of the present invention employ high levels of anionic surfactants, which are, in general, less water-soluble than nonionic surfactants. The decreased solubility of the anionic surfactants coupled with the increased concentration of active ingredients often results in thick pastes with very high viscosities, thereby necessitating the use of heavy duty or high shear mixing equipment. Accordingly, in a preferred mode for carrying out the present invention a non-conventional process is used for the manufacture of the compositions of the present invention.

According to this preferred method, a detergent base comprising magnesium alkylbenzene sulfonate and amide foam booster is preferably manufactured by a process wherein alkanolamide, in the specified amount, is added to an aqueous slurry containing magnesium oxide or hydroxide, hydrotropes, and any inorganic salts or organic solvents of the liquid detergent compositions of this invention, in the specified amounts, and the alkylbenzene sulfonic acid, is added after the alkanolamide has

been dissolved in the slurry. This procedure is described in  
greater detail in our <sup>Australian Patent Application No 87728/91</sup> ~~pending application Serial No.~~

~~filed on the same date as the subject application under  
attorneys docket IR-4863, titled "PROCESS FOR PRODUCING~~

5 CONCENTRATED LIQUID DETERGENTS~~BASE~~ CONTAINING MAGNESIUM  
ALKYLBENZENE SULFONATE AND ALKANOLAMIDE", the disclosure of  
which is incorporated herein in its entirety by reference  
thereto. This method has the advantage of utilizing conventional  
liquids mixing equipment, while resulting in a composition with  
10 enhanced fluidity during neutralization and upon cooling.

The following representative non-limiting examples  
will illustrate the invention although applicants do not intend  
to be bound thereby.



Example 1.

The following compositions are prepared with conventional low shear mixing equipment by mixing the materials in the proportions as listed.

5		<u>Run No.</u>	
		<u>1</u>	<u>2</u>
		(Wt.%)	
	Water, deionized	11.66	11.28
	TEA lauryl sulfate (40% solution)	28.75	28.75
10	Sodium alkyl ethoxy sulfate, 3EO	18.02 (66.6%)	17.7 (67.8%)
	Sodium xylene sulfonate (40% sol.)	-	5.23
	Sodium cumene sulfonate (93%)	2.9	1.0
	Magnesium oxide	1.16	1.16
15	MgSO <sub>4</sub> ·7H <sub>2</sub> O	1.0	3.0
	Na <sub>2</sub> SO <sub>4</sub>	1.5	0.5
	Lauric/myristic monoethanolamide (prill)	-	3.49
	Lauric/myristic monoethanolamide/sodium xylene sulfonate 5:3 blend	8.72	-
20	Ethanol	5.5	7.5
	Dodecylbenzene sulfonic acid	18.75	18.87
	Minor ingredients (dye, perfume, etc.)	<u>2.04</u>	<u>1.52</u>
		100%	100%
	Molar ratio of Mg <sup>+2</sup> : Alkylbenzene sulfonate	0.54:1.0	0.67:1.0
25	Cloud point	6°C	0°C

The composition of Run No. 2 is a physically stable liquid upon accelerated aging at elevated temperature. The composition of Run No. 1, while having a higher hydrotrope level,

was found to exhibit phase separation on aging. Each of the compositions of Runs 1 and 2 are evaluated by the following performance criteria:

(1) Grease Removal Test

5 This test is used to measure grease removal of the composition at a concentration of 1% liquid detergent at 50 ppm water hardness, at 80°F, with 600 dipping cycles at 60 rpm. For this test, 0.5 grams of lard at room temperature is evenly applied to a frosted glass microscope slide using a serrated  
10 knife blade. The results are calculated as milligrams of lard removed.

15 A modified version of the Grease Removal test is used to measure grease removal of a 0.05% solution of liquid detergent at 50 ppm water hardness and 108°F, with 600 dipping cycles at 60 rpm. For this test, 0.1 ± 0.02 grams of lard is applied to plastic test tubes by dipping the pre-weighed tubes in melted lard and blotting off any excess grease before it solidifies. The tubes are then re-weighed and used the same day they are prepared.

20 Detergent compositions to be tested are prepared, warmed to the appropriate temperature and poured into 250 ml beakers. The desired temperature of the solution is maintained by a circulating water bath. The test composition is agitated with a stirring rod until a layer of foam covers the top of each.  
25 The beakers and soiled tubes are placed in the dipping apparatus which is then run at 600 cycles at 60 rpm. Upon completion of the test cycle, the tubes are removed, rinsed in deionized water at 77°F, air dried overnight and weighed. The soil removal performance (percent cleaned) is then calculated by the following  
30 formula:

$$\% \text{ cleaned} = \frac{B-C}{B-A} \times 100,$$

where A = the weight of the tube, B = the weight of the tube plus soil, C = the weight of the tube after washing.

A comparison of the average amount of greasy soil removed by the composition of Run No. 2 with a best selling light duty dishwashing composition (Control 2) as determined by the Grease Removal test at 0.05% detergent concentration, 50 ppm water hardness, 108°F, 600 dipping cycles at 60 rpm is shown in Table 1.

#### (B) Hand Dish Washing Test

Six liters of a diluted (0.1% or 0.075%) test solution prepared using 50 ppm water at 120°F is delivered to a dish pan from a separatory funnel suspended above the pan to generate a layer of foam. Plates soiled with 5.4 grams lard are washed to a foam end point. This test measures the total number of plates that can be washed with the detergent composition until the foam completely disappears.

#### (C) Dynamic Foam Stability Test:

This test is used to determine the dynamic foam stability of a liquid detergent composition at 50 ppm water hardness at 113°F.

A diluted (0.05 or 0.04%) test solution is titrated with constant delivery of mixed soil to a foam end point under constant agitation. The test measures the amount of mixed soil required to deplete the surfactants of the composition.

#### (D) Cylinder Foam Test:

This test is used to determine the sudsing ability of a liquid detergent composition at 50 ppm water hardness at 113°F.

100 mls of a 0.1% LOL test solution is placed in a 500 ml graduated cylinder. The cylinder is inverted 20 times and the amount of foam is measured. Soil (0.01 grams) is added to the solution and the cylinder is again inverted 20 times. The decrease in foam level is then noted.

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The results of the testing for the compositions of Run Nos. 1 and 2 and the results of two different best selling commercial light duty dishwasher compositions are shown in Table 2.



TABLE 1

Average Soil Removal %

	<u>108°F</u>
Run 2	50.9
5 Control 2	22.1

The results of this test indicate that the composition of Run No. 2 removes twice as much greasy soil at 108°F than does a leading commercial brand of hand dishwashing detergent containing similar deterative active ingredients but which has a molar ratio of magnesium ion to alkylbenzene sulfonate of 0.31:1.0.

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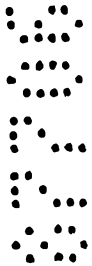


TABLE 2

<u>Test Method</u>	<u>Performance Tests-Set I</u>			<u>Performance Tests-Set II</u>		
	<u>Run. 1</u>	<u>Control 1</u>	<u>Ratio</u>	<u>Run 2</u>	<u>Control 2</u>	<u>Ratio</u>
5 Hand Dish (1) 0.1% LDL 5.4g lard 120 F	34.5	28.5	1.21	- - -	- - -	- - -
10 Hand Dish (1) 0.075% LDL 5.4g lard 120 F	- - -	- - -	- - -	35.7	33.4	1.07
15 Grease Removal(2) 1% LDL 600 cycles 60 rpm lard soil 80F	107	40	2.68	220	190	1.16
20 Dynamic Foam Stability Test(3) 113 F	1.21	1.09	1.11	1.37	1.28	1.07
25 Cylinder Foam (4) 0.1% LDL 20 cycles 30 rpm 100 F (0.01 mixed food soil)	375/ 342	205/ 165	1.83/ 2.07	365/ 335	285/ 265	1.28/ 1.26

All tests use 50 ppm hardness water.

- (1) Hand dish results are in plate count to foam end point.
- (2) Grease Removal results are milligrams of lard removed.
- (3) Dynamic Foam Stability results are foam performance ratios versus a standard liquid detergent composition containing 34% surfactant ingredients.
- (4) Cylinder test results are cc's of foam (no soil/with soil).

Control 1: Leading commercial brand of hand dishwashing composition containing 50% active ingredients including 17% linear alkylbenzene sulfonate, 23% alkyl ethoxy sulfate, 10% fatty acid monoethanolamide and a total magnesium content of 0.70% . This composition has a molar ratio of magnesium to alkylbenzene sulfonate of 0.58:1.0.

Control 2: A second leading commercial brand of hand dishwashing composition containing 56% active ingredients including 24% linear alkyl benzene sulfonate, 22% alkyl ethoxy sulfate·3EO, 10% ethoxylated fatty acid monoethanolamide and also containing 0.5% magnesium, corresponding to a molar ratio of magnesium to alkylbenzene sulfonate of 0.31:1.0.

Example 2.

A stable pourable liquid detergent composition is prepared by mixing the following ingredients using a conventional low shear liquids mixing apparatus. The proportion of each ingredient is as follows:

5

		<u>Run No.</u>	
		<u>Wt. %</u>	
		<u>1</u>	<u>2</u>
	Water, deionized	22.1	17.6
10	TEA lauryl sulfate	-	12.0
•••••	Sodium alkyl ethoxy sulfate·3EO	14.0	15.2
•••••	Sodium xylene sulfonate (40% sol)	12.0	13.1
•••••	Sodium cumene sulfonate (45% sol.)	4.7	5.2
•••••	Magnesium oxide	1.8	1.1
•••••	MgSO <sub>4</sub> ·7H <sub>2</sub> O	4.8	5.5
15	Na <sub>2</sub> SO <sub>4</sub>	-	0.5
	Lauric/myristic monoethanolamide (prill)	8.0	8.0
•••••	Propylene glycol	2.3	2.5
•••••	Dodecylbenzene sulfonic acid	<u>30.3</u>	<u>19.3</u>
•••••	Total	100	100
•••••	Consistency at 25°C	Physically stable liquid	Physically stable liquid
•••••			

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:-

1. A liquid dishwashing detergent composition providing stable foaming characteristics and which is especially effective in removing greasy soils, said composition comprising:
- 5
- (A) from 20 to 70% by weight of a surfactant system comprising
- (a) from 15 to 35% by weight of the magnesium salt of a C<sub>10</sub>-C<sub>18</sub> alkylbenzene sulfonic acid anionic surfactant;
- 10
- (b) from 1 to 5% by weight of a water-soluble magnesium salt, in an amount such that the molar ratio of total magnesium ion from (a) and (b) to alkylbenzene sulfonate ranges from 0.65:1.0 to 1.0:1.0;
- 15
- (c) from 1 to 20% by weight of a water-soluble C<sub>10</sub>-C<sub>16</sub> primary alkyl ethoxy sulfate salt containing an average of from 1 to 6 ethylene oxide groups per alkyl group in the alkyl ether sulfate; and
- 20
- (d) from 0 to 20% by weight of a primary, secondary or tertiary amine or alkali metal salt of C<sub>10</sub>-C<sub>18</sub> alkyl sulfate; and
- (B) from 0.5 to 8% by weight of a suds promoting agent selected from the group consisting of C<sub>10</sub>-C<sub>18</sub> ethoxylated and non-ethoxylated mono- or di-C<sub>1</sub>-C<sub>5</sub> alkanolamides and C<sub>12</sub>-C<sub>14</sub> alkyl amides condensed with up to about 15 moles ethylene oxide per mole of amide;
- 25
- (C) from 0 to 10% by weight of a low irritant organic solvent;
- 30
- (D) from 0 to 10% by weight hydrotrope; and
- (E) from 0 to 10% by weight of one or more optional additive chosen from chelating agents, coloring agents, dyes, perfumes, bactericides, fungicides, preservatives, suncreening agents,
- 35



pH modifiers, pH buffering agents, opacifiers, antioxidants, proteins and

(F) the balance, water.

2. The composition of claim 1 that comprises

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(A) from about 30 to 55%;

(B) from about 1 to 6%;

(C) from about 5 to 8%;

(D) from about 0 to 6%;

(E) from 0 to about 3%; and

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(F) the balance, water.

3. The composition of claim 1 wherein the surfactant system (A) comprises (a) from about 19 to 21% by weight of a magnesium salt of a C<sub>10</sub>-C<sub>18</sub> alkylbenzene sulfonic acid; (b) an amount of a water-soluble magnesium salt such that the molar ratio of magnesium ion to alkylbenzene sulfonate is 0.65:1.0 to 0.8:1.0; (c) from about 10 to 13% by weight of a C<sub>10</sub>-C<sub>16</sub> alkyl ethoxy sulfate with 3 groups of ethylene oxide per mole of alcohol; and (d) from about 10 to 15% by weight of a C<sub>10</sub>-C<sub>16</sub> alkyl sulfate.

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4. A physically stable liquid dishwashing detergent base composition comprising:

(A) from 30 to 70% by weight deterative active ingredients comprising

(a) from 10 to 50% by weight of the magnesium salt of a C<sub>10</sub>-C<sub>18</sub> alkylbenzene sulfonate acid;

25

(b) from 1 to 5% by weight of a water-soluble magnesium salt, in an amount such that the molar ratio of total magnesium ion from (a) and (b) to alkylbenzene sulfonate ranges from 0.65:1.0 to 1.0:1.0;

30

(c) from 1 to 27% by weight of a water-soluble C<sub>10</sub>-C<sub>16</sub> primary alkyl ethoxy sulfate salt containing an average of from 1 to 6 ethylene oxide groups per alkyl group;



(d) from 0 to 27% by weight of a primary amine or alkali metal salt of a C<sub>10</sub>-C<sub>16</sub> alkyl sulfate; and

5

(B) from 0.6 to 11% by weight of a suds promoting agent selected from the group consisting of a C<sub>10</sub>-C<sub>16</sub> mono- and di-C<sub>1</sub>-C<sub>5</sub> alkanolamides and C<sub>12</sub>-C<sub>14</sub> alkyl amides condensed with up to 15 moles ethylene oxide per mole of amide;

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(C) from 0 to 10% by weight of a low irritant organic solvent;

(D) from 0 to 8% by weight of a hydrotrope; and  
(E) the balance, water.

5. The composition of claim 4 that comprises

15

(A) from about 60 to 65%;  
(B) from about 1 to 8%;  
(C) from about 2.5 to 3.5%;  
(D) from about 1 to 5%; and  
(E) the balance, water.

20

6. The composition of claim 4 wherein the surfactant system (A) comprises a water-soluble magnesium salt in an amount sufficient to provide a molar ratio of total magnesium ion to magnesium alkylbenzene sulfonate in the range of 0.65:1.0 to 0.8 to 1.0

DATED this 31st day of August 1994

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Abstract Of The Disclosure

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A concentrated light duty liquid detergent composition is described. The composition, which offers superior grease cutting and foam mileage, contains a surfactant mixture of magnesium alkylbenzene sulfonate, alkyl ethoxy sulfate and optionally, alkyl sulfate. The composition also contains a magnesium ion source in an amount such that the molar ratio of total magnesium ion to alkylbenzene sulfonate ranges from about 0.65:1.0 to 1.0:1.0.

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