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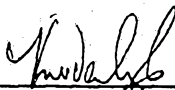
DECLARATION IN SUPPORT OF AN APPLICATION FOR A PATENT

In support of the Convention application made by BP Chemicals Limited for a patent for an invention entitled: "Anhydrides in detergent compositions".

I, Keith Warwick DENBIGH of the Applicant's address, do solemnly and sincerely declare as follows:

1. I am authorised by BP Chemicals Limited the applicant for the patent to make this Declaration on their behalf.
2. The basic application as defined by Section 141 of the Act was made in England on the 11th day of February, 1988 by BP Chemicals Limited.
3. Stephen Robert HODGE and Andrew PEARCE residing at 48 Annandale Road, Kirkella, Hull, HU10 7UU, England and 1 Ladywell Court, Ladywell Gate, Welton, Borough, North Humberside, HU15 1LY, England respectively of British nationality are the actual inventors of the invention and the facts upon which the said company is entitled to make the application are as follows:
The applicant is the assignee of the inventors
4. The basic application referred to in paragraph 2 was the first application made in a Convention country in respect of the invention the subject of the application.

Declared at Britannic House, Moor Lane, London, EC2Y 9BU, England
this 2nd day of February, 1989


K.W. DENBIGH
by Power of Attorney

To: The Commissioner of Patents

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ANHYDRIDES IN DETERGENT COMPOSITIONS

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(56) Prior Art Documents
AU 30606/89 C11D 3/39

(57) Claim

1. A detergent composition in aqueous solution comprising

- (i) a surfactant selected from anionic, non-ionic, zwitterionic and cationic surfactants and mixtures thereof,
- (ii) a precursor compound capable of giving rise to a peroxygen compound in the presence of water,
- (iii) a bleach activator capable of enhancing the bleaching activity of the peroxygen compound so formed,
- (iv) a suds suppressing agent, and
- (v) a detergent builder,

characterised in that the bleach activator comprises one or more cyclic anhydrides containing at least one nitrogen atom in the alpha position with respect to at least one of the carbonyl functions in the anhydride, and the activator is at least partially soluble in water.

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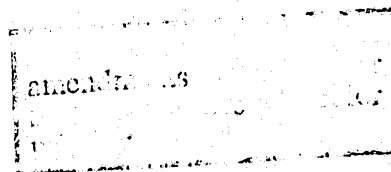
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(54) Title: ANHYDRIDES IN DETERGENT COMPOSITIONS

(57) Abstract

The present invention relates to a detergent composition in aqueous solution comprising, (i) a surfactant selected from anionic, nonionic, zwitterionic and cationic surfactants and mixtures thereof, (ii) a precursor compound capable of giving rise to a peroxygen compound in the presence of water, (iii) a bleach activator capable of enhancing the bleaching activity of the peroxygen compound so formed, (iv) a suds suppressing agent and (v) a detergent builder, characterised in that the bleach activator comprises one or more cyclic anhydrides containing at least one nitrogen atom in the alpha position with respect to at least one of the carbonyl functions in the anhydride and the activator is at least partially soluble in water. These compounds show bleaching activity at relatively low temperatures.

ANHYDRIDES IN DETERGENT COMPOSITIONS

The present invention relates to the use of anhydrides as bleach activators, especially in detergent compositions.

Compounds such as tetraacetyl ethylene diamine (hereafter referred to as "TAED") are well known. Processes for the production of such compounds are disclosed for instance in published German patent application no. 2832021. These compounds are said to be efficient in activating the conventional inorganic salts used as bleach precursors in detergent compositions and generate peracetic acid in situ by the reaction thereof with alkaline hydrogen peroxide. The activating agent for the bleach precursor is the so-called bleach activator. Specific examples of such bleach precursors are sodium perborate and sodium percarbonate. In the absence of the activators the bleach precursor is satisfactorily effective only at elevated temperatures, its effectiveness being very slow at lower temperatures. The use of compounds such as TAED enable the bleach precursor to function more effectively at temperatures of the order of 60°C.

It has now been found that certain anhydrides function as efficient additives in activating the bleach precursor in detergent compositions, especially at low temperatures.

Accordingly the present invention is a detergent composition in aqueous solution comprising

- (i) a surfactant selected from anionic, nonionic, zwitterionic and cationic surfactants and mixtures thereof,
- (ii) a precursor compound capable of giving rise to a peroxygen

compound in the presence of water,

(iii) a bleach activator capable of enhancing the bleaching activity of the peroxygen compound so formed,

(iv) a suds suppressing agent and

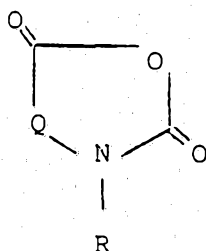
5 (v) a detergent builder,

characterised in that the bleach activator comprises one or more cyclic anhydrides containing at least one nitrogen atom in the alpha position with respect to at least one of the carbonyl functions in the anhydride and the activator is at least partially soluble in

10 water.

Thus such compounds may be represented by the generic formula

15



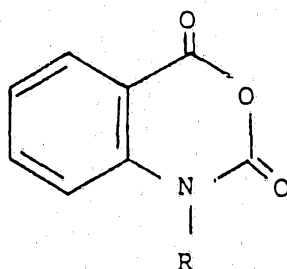
(I)

20 wherein Q is a divalent organic grouping such that Q and N together with the carbonyl and oxygen functions in the anhydride group form one or more cyclic structures, and R is H, an alkyl, aryl, halogen, a carboxylic or a carbonyl containing function.

Where R has an aryl, alkaryl or aralkyl containing functions, 25 it is essential that such functions also carry a substituent capable of solubilising the activator in aqueous systems eg. a sulphonic acid group. Where R is a halogen containing functions, the halogen is preferably chlorine or bromine.

An example of such compound is that shown in formula (II) 30 below:

35



(II)

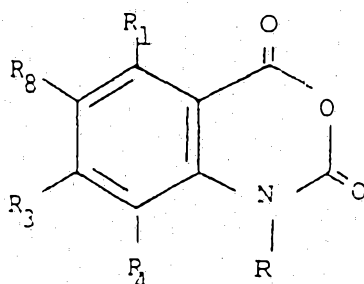
In the formula (II) above, where $R=H$; isatoic anhydride or
2H-3,1-Benzoxazine-2,4-(1H)dione

[Chem. Reg. No. 118-48-91]

$R=Me$; N-Methyl Isatoic anhydride or 2H-3,1-Benzoxazine-2,4-(1H)dione
1-methyl

[Chem. Reg. No. 10328-92-4]

Another compound of this type is a compound of formula (III) below:

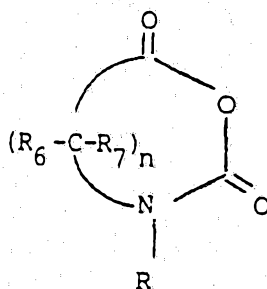


(III)

wherein R has the same significance as in formula (I) above, and R_1 ,
 R_2 , R_3 and R_4 may be the same or different nuclear substituents and
may be selected from H , halogen, alkyl, alkenyl, aryl, alkoxy,
amino, $COOR_5$, (where R_5 is an H or an alkyl group) and carbonyl
containing functions. Specific examples of such compounds include
cases where:

- (a) $R=R_1=R_4=H$ and $R_2=R_3=OCH_3$
- (b) $R=R_1=R_2=R_3=H$ and $R_4=CH_3$
- (c) $R=R_1=R_3=R_4=H$ and $R_2=CH_3$
- (d) $R=R_2=R_3=R_4=H$ and $R_1=CH_3$
- (e) $R=R_1=R_3=R_4=H$ and $R_2=Cl$ or Br
- (f) $R=R_1=R_2=R_4=H$ and $R_3=Cl$ or CO_2H or NO_2 , or
- (g) $R_1=R_2=R_3=R_4=H$ and $R=CH_3CO$. or Cl

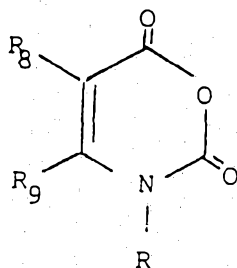
A further compound of this type is shown in formula (IV) below:



(IV)

where R has the same significance as in formula (I) above, and R_6 and R_7 may be the same or different groups and may be any one of the groups listed in the context of R_1 to R_4 above and n has a value from 1-3.

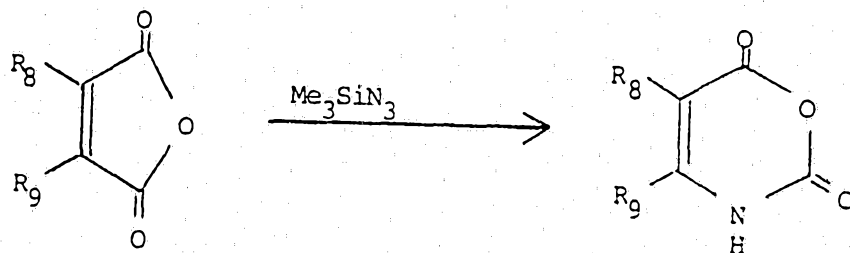
Another compound falling within formula (I) can be represented by (V)



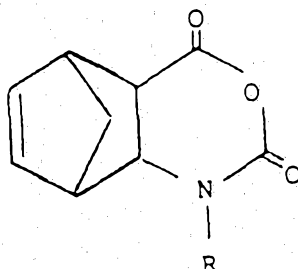
(V)

wherein R has the same significance as in formula (I) above, and R_8 and R_9 are the same or different groups and may be any one of the groups designated for the substituents R_1 - R_4 above except that both R_8 and R_9 should not be H, or which together form one or more cyclic structure with or without additional hetero atoms. One example of such a compound in where R_8 and R_9 together represent one or more benzene rings as in formula (III) above, or, where they together with the hydrocarbyl carbon atoms in the anhydride ring represent a pyridine, pyrazole, pyrimidine or an imidazole ring.

Compounds of structure (V) can be synthesised by the reaction of trimethyl silylazide with the corresponding anhydride. The reaction involves a Hofmann rearrangement of the intermediate acyl azide.



A further example of such a compound is that represented by formula (VI) below:



(VI)

wherein R has the same significance as in formula (I) above.

The bleach activator is at least partially soluble in water. Thus the solubility is at least 0.01%w/w in water at ambient temperature, eg. 25°C.

It is believed that the activators of the present invention preferentially react with the peroxygen compound, eg. hydrogen peroxide generated by contacting the precursor compound with water. This is believed to result in a peroxygen species of enhanced bleaching activity than that initially generated.

The bleach activators of the present invention can be used or in conjunction with other conventional activators such as TAED, phthalic anhydride, maleic anhydride, succinic anhydride isononanoyl oxybenzene sulphonate (also known as isonobs') and tetracetyl glycoluril (also known as "TAGU") and the like or with mixtures of such known activators.

Any of the well known surfactants can be used in the detergent compositions of the present invention. A typical list of these surfactants can be found in EP 0120591 and in USP 3,663,961.

Examples of water soluble anionic surfactants include the salts of alkyl benzene sulphonates, paraffin sulphonates, alpha-olefin sulphonates, alkyl glyceryl ether sulphonates and 2-acyloxy alkane-1-sulphonate, and beta-alkyloxy alkane sulphonate. Similarly, salts of alkyl sulphates, alkyl polyalkoxy ether sulphates, alpha-sulpho-carboxylates and their esters, fatty acid monoglyceride sulphates and sulphonates and alkyl phenol polyalkoxy ether sulphates may also be used.

Suitable examples of the above surfactants are linear straight chain alkyl benzene sulphonates having alkyl groups with 8-16 carbon atoms and methyl branched alkyl sulphates having 8-16 carbon atoms which are also effective.

5 Other anionic detergent compounds suitable for use herein include the sodium alkyl glyceryl ether sulphonates derived from tallow and coconut oil; sodium fatty acid monoglyceride sulphonates and sulphates derived from coconut oil; and sodium or potassium salts of C₈-C₁₂ alkyl phenol alkylene oxide ether sulphate
10 containing up to 10 alkylene oxide units per molecule. Mixtures of anionic surfactants may also be used.

A substantial list of such compounds can be found in e.g. McCutcheon's Dictionary of Emulsifiers and Detergents, International Edition (1981), published by the Manufacturing Confectioner
15 Publishing Co. and in "Surfactants Europa: A Directory of Surface Active Agents available in Europe", Ed. Gordon L. Hollis, Vol 1 (1982), published by George Goodwin.

The nonionic surfactants which may be used in the present invention are condensates of an alkylene oxide e.g. ethylene oxide
20 with a hydrophobic group to form a surfactant having an appropriate hydrophilic-lipophilic balance (HLB) in the range from 8 to 17, suitably from 9.5 to 13.5, preferably from 10 to 12.5. The hydrophobic group may be an aliphatic or aromatic type and the length of the polyoxyethylene group condensed therewith can be
25 readily adjusted to yield a water-soluble compound having the desired degree of HLB.

Examples of suitable nonionic surfactants include:

(a) The polyethylene oxide condensates of alkyl phenol in which the alkyl group e.g. contains from 6 to 12 carbon atoms and in
30 which from 3 to 30 moles, preferably 5 to 14 moles of ethylene oxide are present. Other examples include a mole of dodecylphenol condensed with 9 moles of ethylene oxide, a mole of dinonylphenol condensed with 11 moles of ethylene oxide and a mole of nonylphenol and octadecylphenol condensed with
35 13 moles of ethylene oxide.

(a) The nonionic surfactant may also be formed as a condensation product of a mole of primary or secondary C₈-C₂₄ aliphatic alcohols with from 2 to 40 moles, preferably 2 to 9 moles of ethylene oxide.

Specific examples of nonionic surfactants useful for the purposes of the invention include the various grades of Dobanol (Registered Trade Mark, supplied by Shell) Lutensol (Registered Trade Mark, supplied by BASF) and Synperonics (Registered Trade Mark, supplied by ICI).

Other useful nonionic surfactants include the synthetic nonionic detergents available on the market under "Pluronic" (Registered Trade Mark) and supplied by Wyandotte Chemicals Corporation.

Zwitterionic compounds such as betaines and sulphobetaines, particularly those with a C₈-C₁₆ alkyl substituent on the nitrogen atom can also be used as surfactants.

Examples of cationic surfactants that can be used include e.g. quaternary ammonium surfactants and surfactants of a semi-polar nature, for example amine oxides. Suitable quaternary ammonium surfactants are the mono C₈-C₁₆, N-alkyl or alkenyl ammonium surfactants in which remaining N valences are methyl, hydroxyethyl or hydroxypropyl groups. Suitable examples of amine oxides are the mono C₈-C₂₀, N-alkyl or alkenyl amine oxides and the propylene-1,3-diamine dioxides in which the remaining N valences are methyl, hydroxyethyl or hydroxypropyl substituents.

The detergent compositions can comprise from 1 to 70% w/w, suitably from 1 to 20% w/w of the total composition. Mixtures of anionic with nonionic or zwitterionic surfactant types are preferred

Suitable bleach precursors which act as a source of a peroxygen compound eg. hydrogen peroxide include sodium perborate mono and tetrahydrate, sodium percarbonate, sodium persulfate and the clathrate 4Na₂SO₄:2H₂O₂:1 NaCl.

If clathrate materials are used as peroxygen source (bleach precursor), a separate source of alkalinity will be required and for stability reasons these are preferably stored separately from the hydrogen peroxide source. The precursor compound (ii) acting as the

hydrogen peroxide source can be present in an amount of from 1 to 40% w/w suitably from 5 to 35% by weight, preferably from 10 to 30% by weight of the total composition.

In the detergent compositions of the present invention the
5 molar ratio of peroxygen compound eg. hydrogen peroxide generated from a bleach precursor to bleach activator is suitably greater than 1.5:1, preferably at least 2.0. Under the usage conditions encountered in domestic laundry practice, the molar ratio of bleach precursor to bleach activator is generally greater than 5.0:1 and is
10 most preferably greater than 10:1.

Suds suppressing agents which are useful in the detergent compositions of the invention are suitably selected from silicone, wax, vegetable and hydrocarbon oil and phosphate ester varieties. Suitable silicone suds controlling agents include
15 polydimethylsiloxanes having a molecular weight in the range from 200 to 200,000 and a kinematic viscosity in the range from 20 to 2,000,000 mm²/s (cSt), preferably from 3000 to 30,000 mm²/s (cSt),

Water-insoluble detergent builders can also be used. A specific example of such builders are the zeolites especially the
20 sodium type A zeolite typified by SASIL (Registered Trade Mark). and mixtures of siloxanes and hydrophobic silanated (e.g. trimethylsilanated) silica having a particle size in the range from 10 to 20 millimicrons and a specific surface area above 50 m²/g. Suitable waxes include microcrystalline waxes having a melting point
25 in the range from 65°C to 100°C, a molecular weight in the range from 4,000-10,000 and a penetration value of at least 6, measured at 77°C by ASTM-D1321 and also paraffin waxes, synthetic waxes and natural waxes. Suitable phosphate esters include mono- and/or di-C₁₆-C₂₂ alkyl or alkenyl phosphate esters, and the corresponding
30 mono- and/or di alkyl or alkenyl ether phosphates containing up to 6 ethoxy groups per molecule.

Suds suppressors are normally present in an amount from 0.01 to 5% w/w of the total composition depending upon the type of suds suppressor used, and is preferably from 0.1 to 2% w/w.

35 A highly preferred component of detergent compositions in

accordance with the invention is one or more detergent builder salts which may comprise up to 90% of the composition, more typically from 10 to 70% by weight thereof. Suitable detergent builder salts useful herein can be of the polyvalent inorganic and polyvalent organic types or mixtures thereof. Examples of suitable water-soluble, inorganic alkaline detergent builder salts include the alkali metal carbonates, borates, phosphates, pyrophosphates, tripolyphosphates and bicarbonates.

Examples of suitable organic alkaline detergency builder salts are water-soluble polycarboxylates such as the salts of nitrilotriacetic acid, lactic acid, glycollic acid and ether derivatives thereof; succinic acid, malonic acid, (ethylenedioxy)diacetic acid, maleic acid, diglycollic acid, tartaric acid, tartronic acid and fumaric acid; citric acid, aconitic acid, citraconic acid, carboxymethyloxysuccinic acid, lactoxysuccinic acid, and 2-oxy-1,1,3propane tricarboxylic acid; oxydisuccinic acid, 1,1,2,2-ethane tetracarboxylic acid, 1,1,3,3-propane tetracarboxylic acid and 1,1,2,3-propane tetracarboxylic acid; cyclopentane cis, cis,cistetracarboxylic acid, cyclopentadiene pentacarboxylic acid, 2,3,4,5-tetrahydrofuran-cis, cis, cis-tetracarboxylic acid, 2,5-tetrahydrofuran-cis-dicarboxylic acid, 1,2,3,4,5,6-hexanehexacarboxylic acid, mellitic acid, pyromellitic acid and the phthalic acid derivatives.

Mixtures of organic and/or inorganic builders can also be used.

Chelating agents, soil suspending and anti-redeposition agents, optical brightening agents, enzymes, colours and perfumes may also be added to the detergent composition.

Chelating agents that can be incorporated include citric acid, nitrilotriacetic and ethylene diamine tetra acetic acids and their salts, organic phosphonate derivatives such as those disclosed in US Patent Nos. 3,213,030, 3,433,021, 3,292,121 and 2,599,807 and carboxylic acid builder salts such as those disclosed in US Patent No. 3,308,067. The chelating agents can be present in amounts ranging from 0.1 to 3%, suitably from 0.2 to 2% by weight of the total composition.

The detergent compositions containing the bleach activators of the present invention may contain, in addition, minor conventional additives such as fragrances perfumes and the like.

Thus the bleach activators of the present invention should find wide use in detergent compositions which use the inorganic bleach precursors. The fact that these anhydrides activate the bleach precursors at relatively lower temperatures e.g. from 20-60°C than those used hitherto should enable a considerable energy saving to be achieved, when the detergents are used.

The present invention is further illustrated with reference to the accompanying Examples.

Example 1

Preliminary washing/bleaching tests were carried out on standard stained cloth swatches (EMPA* red wine 1" x 4") in a beaker maintained at a constant temperature of 40°C using the base detergent powder composition (Table 1) and the various bleach activators described in Table 2.

2.4g of base detergent plus 0.45g of sodium perborate tetrahydrate plus bleach activator (0.15g, 5.0% w/w) as described in Table 2 were added to 600ml of tap water at 40°C having a hardness of about 290 ppm of calcium carbonate. Red wine stained swatches were added, then the composition stirred for 30 minutes at 40°C after which the swatches were removed, rinsed in tap water and dried at 24°C. The stain removal achieved by each bleach activator was assessed visually, using standard lighting conditions (ICS-TeXicon Multilight Cabinet, D65) and compared to the stain removal achieved by TAED (10) and that of a blank run (using perborate alone, 0), in which no bleach activator was used, and a rating awarded.

*The following Examples were carried out using a testing technique prescribed by the Swiss Federal testing agency, Eidgenössische Material Prüfungs und Versuchsanstalt Ch-9001, St Gallen Unterstrasse, PO Box 977, Switzerland.

This is hereafter identified as "EMPA".

Table 1

		%
	Linear sodium alkyl benzene sulphonate (mean length of alkyl chain C _{11.5})	8.0
5	Ethoxylated tallow alcohol (14 EO)	2.9
	Sodium soap (chain length C ₁₂ - C ₁₆ : 13-26% C ₁₈ - C ₂₂ : 74-87%)	3.5
	Sodium triphosphate	43.8
	Sodium silicate (SiO ₂ :Na ₂ O = 3.3:1)	7.5
10	Magnesium silicate	1.9

Table 2

Visual Rating of Stain Removal

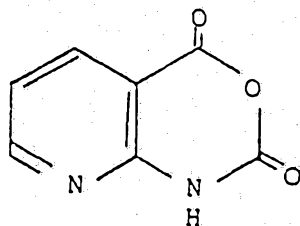
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Isatoic Anhydride Derivatives

(Beaker, Red Wine Cloths, 30 Minutes, 40°C 5% Activator)

	R	X	Method of Preparation	Visual Rating
20	H	H	ref 1	10
	H	6,7-Dimethoxy	ref 2	10
	H	8-Methyl	ref 2	9
	H	6-Methyl	ref 2	9
25	H	5-Methyl	ref 2	10
	H	6-Chloro	ref 2	9
	H	7-Chloro	ref 2	9
	Methyl	H	ref 4	7

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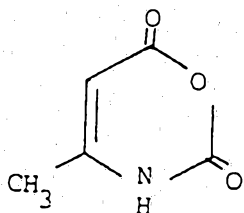


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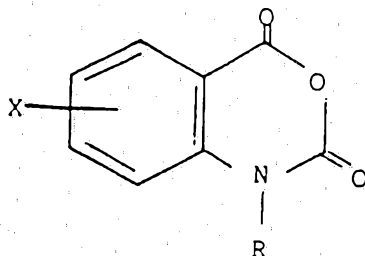
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ref 5

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(III)

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15 References

1. Aldrich Catalogue No. I-1,280-8, (1988-89).
2. See Synthesis below.
3. US Patent 3,622,573.
4. Aldrich No. 12,988-7, (1988-89).
- 20 5. J. D. Warren, J. H. MacMillan, and S. S. Washburne, J. Org. Chem., 1975, 40, 7413.

Synthesis of Bleach Activators

The respective anthranilic acid (1×10^{-2} mol) was dissolved with stirring in dry THF (100 ml) under nitrogen. Bis(trichloromethyl)carbonate ("triphosgene") (1×10^{-2} mol) was added dropwise and then the mixture heated under reflux for 15 minutes. On cooling a solid precipitated, and this was the desired activator, which was collected by filtration and dried under vacuum.

30 Example 2

Further washing/bleaching tests were carried out on standard stained cloth swatches (EMPA red wine stained 2" x 6") using the base detergent powder composition (Table 1) and the various bleach activators described in Table 3 in a terg-o-tometer. The terg-o-tometer was maintained at a constant temperature of 40°C and

operated at 75 rpm.

4g of base detergent plus 0.75g sodium perborate tetrahydrate plus bleach activator (0.25g, 5.0% w/w) as described in Table 3 were added to one litre of tap water at 40°C, having a hardness of about 290 ppm as calcium carbonate. Red wine swatches were added, then the composition agitated for 20 minutes at 40°C after which the swatches were removed, rinsed in tap water and dried at 24°C. The reflectance of the swatches were taken before and after using an ICS Micromatch reflectometer and the percentage stain removal (% SR) calculated by applying the following formula where L represents whiteness parameter generated by the reflectometer (black=0 and white=100).

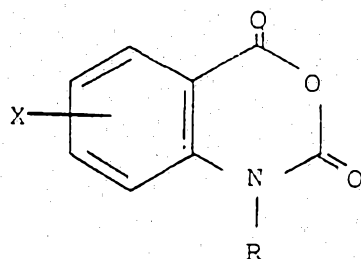
$$\%SR = \frac{L_{\text{sample}} - L_{\text{redwine}}}{L_{\text{white}} - L_{\text{redwine}}} \times 100$$

Three replicates were run and the average result is quoted.

Table 3
Percentage Stain Removal
Isatoic Anhydride Derivatives
(Terg-o-Tometer, Red Wine Cloths, 20 Minutes, 40°C, 5% Activator)

R	X	% Stain Removal
25	-----	
H	H	76.0
H	6,7-dimethoxy	78.5
H	8-Methyl	75.3
H	6-Methyl	74.2
30 H	5-Methyl	79.1
H	6-Chloro	73.8
H	7-Chloro	76.3
	TAED	76.0
35	Perborate	60.3

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(III)

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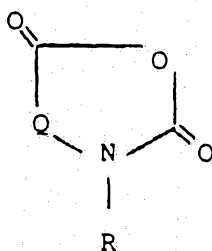
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Claims:

1. A detergent composition in aqueous solution comprising
(i) a surfactant selected from anionic, non-ionic, zwitterionic and cationic surfactants and mixtures thereof,
(ii) a precursor compound capable of giving rise to a peroxygen compound
5 in the presence of water,
(iii) a bleach activator capable of enhancing the bleaching activity of the peroxygen compound so formed,
(iv) a suds suppressing agent, and
(v) a detergent builder,
10 characterised in that the bleach activator comprises one or more cyclic anhydrides containing at least one nitrogen atom in the alpha position with respect to at least one of the carbonyl functions in the anhydride, and the activator is at least partially soluble in water.
- 15 2. A detergent composition according to claim 1 wherein the bleach activator has the generic formula



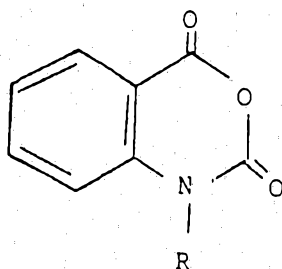
(I)

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wherein Q is a divalent organic grouping such that Q and N together with the carbonyl and oxygen function in the anhydride form one or more
25 cyclic structures, and R is a group selected from H, an alkyl, aryl,

halogen, and a carboxylic or a carbonyl containing function.

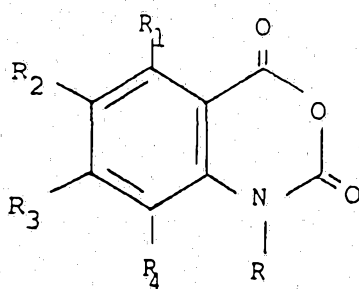
3. A detergent composition according to claim 1 or 2 wherein the bleach
5 activator has the formula



(II)

wherein R=H or an alkyl group.

4. A detergent composition according to claim 1 or 2 wherein the bleach
20 activator has the formula



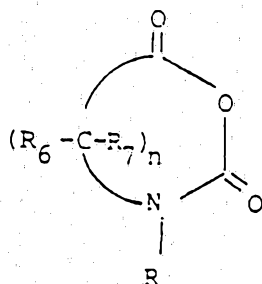
(III)

wherein R has the same significance as in claim 2 and, R₁, R₂, R₃ and R₄
may be the same or different nuclear substituents and may be selected
30 from H⁹ halogen, alkyl, alkenyl, aryl, alkoxy, amino, COOR₅ (where R₅
is an H or an alkyl group) and carbonyl containing functions.

5. A detergent composition according to claim 4 wherein the bleach
activator is a compound of formula (III) and wherein the specific
35 compounds have the following combination of substituents.

- (a) $R=R_1=R_4=H$ and $R_2=R_3 = OCH_3$
 (b) $R=R_1=R_2=R_3=H$ and $R_4 = CH_3$
 (c) $R=R_1=R_3=R_4=H$ and $R_2 = CH_3$
 (d) $R=R_2=R_3=R_4=H$ and $R_1=CH_3$
 5 (e) $R=R_1=R_3=R_4=H$ and $R_2 = Cl$ or Br
 (f) $R=R_1=R_2=R_4=H$ and $R_3=Cl$ or CO_2H or NO_2 , or
 (g) $R_1=R_2=R_3=R_4=H$ and $R=CH_3CO.$ or Cl

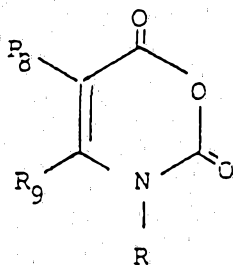
6. A detergent composition according to claim 1 or 2 wherein the bleach
 10 activator has the formula



(IV)

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 20 wherein R has the same significance as in claim 2 and R_6 and R_7 are the same or different groups described in respect of R_1 to R_4 in claim 4 and n has a value from 1 to 3.

7. A detergent composition according to claim 1 wherein the bleach
 25 activator has the formula (V)



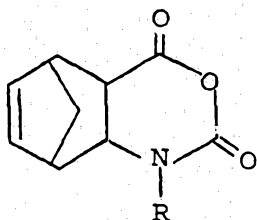
(V)

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 wherein R has the same significance as in claim 2, and R_8 and R_9 are the same or different groups denoted by the substituents R_1 - R_4 in claim 4,
 35 or, which together form one or more cyclic structures with or without



additional hetero atoms.

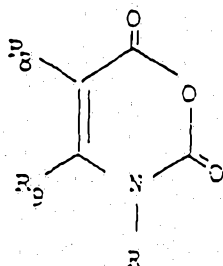
8. A detergent composition according to claim 7 wherein the bleach activator has the formula



(VI)

5 wherein R has the same significance as in claim 2.

9. A detergent composition according to claim 1 or 2 wherein the bleach activator has the formula (VI):



(V)

10 10. A detergent composition according to any one of the preceding claims wherein the surfactant (i) is present in an amount from 1 to 70%w/w of the total composition.

11. A detergent composition according to any one of the preceding claims wherein the precursor compound (ii) acts as a source of hydrogen peroxide.

15 12. A detergent composition according to claim 11 wherein the precursor compound (ii) is selected from sodium perborate mono or tetra-hydrate, sodium percarbonate, sodium persulfate, and the clathrate $4\text{Na}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}_2 \cdot \text{NaCl}$.

20 13. A detergent composition according to any one of the preceding claims 11-12 wherein the precursor compound acting as hydrogen peroxide source is present in an amount from 1-40% w/w of the total composition.

14. A detergent composition according to any one of the preceding claims 12-14, wherein the molar ratio of the hydrogen peroxide generated by the precursor compound (ii) to the bleach activator (iii) is greater than



1.5:1.

15. A detergent composition according to any one of the preceding claims wherein the amount of suds suppressing agents (iv) range from 0.01 to
5 5%w/w of the total composition.

16. A detergent formulation according to any one of the preceding claims, wherein the detergent builder (v) is a salt and comprises up to
10 90%w/w of the total composition.

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INTERNATIONAL SEARCH REPORT

International Application No PCT/GB 89/00131

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC ⁴ : C 11 D 3/39; C 11 D 3/28; C 11 D 3/20; C 07 D 265/26		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System ¹	Classification Symbols	
IPC ⁴	C 11 D; C 07 D	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹		
Category ¹⁰	Citation of Document, ¹¹ with Indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
A	EP, A, 0021043 (BASF AG) 7 January 1981 --	
A	US, A, 3984406 (H.-J. QUADBECK-SEEGER) 5 October 1976 --	
A	GB, A, 1436810 (IMPERIAL CHEMICAL IND.) 26 May 1976 --	
A	EP, A, 0020969 (BASF AG) 7 January 1981 --	
A	CH, A, 575401 (THE SHERWIN-WILLIAMS CO.) 14 May 1976 --	
A	Chemical Abstracts, vol. 94, no. 11, 16 March 1981, (Columbus, Ohio, US), see page 712, abstract 83968m, & DE, A, 3011994 (BOOTS CO. LTD) 16 October 1980 --	
./.		
<p>¹⁰ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"A" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
29th April 1989	12 JUN 1989	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	P.C.G. VAN DER PUTTEN	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
------------	--	----------------------

A	Chemical Abstracts, vol. 104, no. 5, 3 February 1986, (Columbus, Ohio, US), see page 549, abstract 34077m, & US, A, 4537966 (PENNWALT CORP.) 27 August 1985	
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A	Chemical Abstracts, vol. 92, no. 15, 14 April 1980, (Columbus, Ohio, US), N.R. Naik et al.: "Synthesis and spectral study of 1-methyl-2(R)- 1,4-dihydro-4-oxoquinazoline deriva- tives", see pages 726-727, abstract 129152e, & J. Indian Chem. Soc. 1979, 56(7), 708-11	
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**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO.**

GB 8900131
SA 26807

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the European Patent Office EDP file on 08/06/89
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A- 0021043	07-01-81	DE-A- 2925175	08-01-81
		AT-T- 799	15-04-82
		JP-A- 56049372	02-05-81
		US-A- 4316020	16-02-82
US-A- 3984406	05-10-76	DE-A, C 2258150	30-05-74
		DE-A- 2346308	27-03-75
		BE-A- 807899	28-05-74
		CA-A- 989402	18-05-76
		CH-A- 592073	14-10-77
		FR-A, B 2207919	21-06-74
		GB-A- 1442386	14-07-76
		JP-A- 49082674	08-08-74
		NL-A- 7316217	30-05-74
GB-A- 1436810	26-05-76	None	
EP-A- 0020969	07-01-81	DE-A- 2924789	22-01-81
		JP-A- 56045444	25-04-81
		US-A- 4306074	15-12-81
CH-A- 575401	14-05-76	CA-A- 979454	09-12-75
		DE-A- 2230374	08-03-73
		GB-A- 1401033	16-07-75