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54 **Detergency builder additive and detergent system containing it.**

57 A detergency builder additive, for boosting the detergency of a main wash powder in hard water, comprises alkali metal aluminosilicate (at least 50 wt%), sodium carbonate (15-20 wt%) and an organic sequestrant (2-12 wt%), preferably citrate. The builder additive may form part of a Baukasten (building block) detergent system comprising as separately packaged components a main wash powder, a builder additive, and optionally a bleach additive.

**EP 0 419 036 A2**

## DETERGENT COMPOSITIONS

TECHNICAL FIELD

The present invention relates to a detergency builder additive containing alkali metal aluminosilicate, an organic sequestrant, and sodium carbonate. The invention also relates to the use of the detergency builder additive in a detergent system of the "Baukasten" or building block type.

BACKGROUND AND PRIOR ART

On the German market there are a number of products of the Baukasten or building block type: a main wash powder is supplied together with separately packaged additives, for example, water softener (builder) and bleach, that need only be used when the wash conditions demand them.

GB 1 505 274 (Colgate-Palmolive) discloses a detergent composition comprising separately packaged unit doses (tablets, sachets etc) of surfactant, builder, bleach and other ingredients, which can be mixed at will by the consumer to formulate a wash liquor.

EP 243 908A (Henkel) discloses a granular water-softening composition comprising 50-60 wt% fine-crystalline zeolite, 4-12 wt% sodium carbonate, 4-15 wt% sodium citrate, 3-5 wt% acrylic or acrylic/maleic polymer (sodium salt), optionally up to 2 wt% sodium carboxymethyl cellulose, and 12-25 wt% water. It can be used as an additive to supplement the performance of a commercially available detergent powder.

The composition exemplified contains 62.5 wt% (sic) zeolite A, 5.1 wt% sodium carbonate, 10.5 wt% sodium citrate, 4.2 wt% acrylic/maleic copolymer, 0.3 wt% carboxymethylcellulose, and 17.5 wt% water.

DE 3 144 297A (Degussa) discloses a detergency builder composition (phosphate substitute) comprising zeolite A and alkali metal or ammonium citrate, preferably prepared by spray-drying. The preferred content of alkali metal or ammonium citrate is from 5 to 50 wt%.

EP 22 023B (Union Carbide) discloses a zeolite agglomerate comprising crystals of zeolite contained in a matrix of nonionic surfactant and sodium citrate dihydrate.

DE 3 316 513A (Benckiser) discloses a free-flowing particulate granulate comprising a water-insoluble aluminosilicate, at least 5 wt% of a homo- or copolymeric polycarboxylic acid or salt, for example, an acrylate/maleate copolymer, and optionally an aminopolycarboxylic acid or salt thereof.

DE 3 534 888A (Toyo Soda) discloses a homogeneous granular detergency builder composition comprising an ion-exchanging zeolite, sodium carbonate, and another inorganic salt, for example, sodium sulphate, sodium bicarbonate or a sodium phosphate.

DEFINITION OF THE INVENTION

The present invention provides a detergency builder additive, for use in the washing of soiled laundry, in conjunction with a main wash detergent powder comprising one or more organic surfactants and one or more detergency builders, the builder additive being substantially free of organic surfactants and comprising:

- (i) at least 50 wt% (anhydrous basis) of a water-insoluble alkali metal aluminosilicate,
- (ii) from 15 to 20 wt% of sodium carbonate, the total of (i) and (ii) preferably being at least 70 wt%, and
- (iii) from 2 to 12 wt% of organic sequestrant builder.

The present invention also provides a detergent system of the Baukasten (building block) type for washing soiled laundry, comprising as separate components:

- (a) a main wash powder comprising one or more organic surfactants and one or more detergency builders;
- (b) a detergency builder additive as defined in the previous paragraph, and, optionally,
- (c) a bleach additive.

The present invention further provides a method for washing soiled laundry in a domestic washing machine, using a detergent system as defined in the previous paragraph, which method includes the steps of separately introducing a main wash powder as defined in paragraph (a) above, a detergency builder additive as defined in paragraph (b) above, and optionally a bleach additive (c), into the washing machine.

DETAILED DESCRIPTION OF THE INVENTIONThe detergency builder additive

5 In its first aspect the invention relates to a detergency builder additive comprising three essential components: alkali metal aluminosilicate, organic sequestrant builder, and sodium carbonate.

It is well known that the detergency builder performance of aluminosilicates is deficient in some respects and needs to be supplemented by the incorporation of other builders, of which the organic sequestrants have proved especially effective. The detergency builder additive of the invention differs from that disclosed in the above-mentioned EP 243 908A (Henkel) in having a significantly lower proportion of organic sequestrant builder, yet surprisingly its performance as a builder additive is at least as good. The lower level of organic material is environmentally beneficial because the oxygen demand during degradation is lower.

15 The detergency builder additive described in EP 243 908A also contains two different organic sequestrants, an acrylate/maleate copolymer (3-5 wt%, for example, 4.2 wt%) and a citrate (4-15 wt%, for example, 10.5 wt%), while the content of sodium carbonate is only 4-12 wt% (for example, 5.1 wt%). The total level of organic sequestrant builder in the exemplified composition is therefore 14.7 wt%, and the total level of inorganic builder 67.6 wt%. The present inventors have found, as will be demonstrated below, that equivalent or slightly better performance may be obtained from a composition containing only 5 wt% of organic sequestrant builder (sodium citrate) together with a rather similar level (58.52 wt%) of zeolite but a significantly higher level (18 wt%) of sodium carbonate (total inorganic builder 76.52 wt%). This is all the more surprising in view of the absence of acrylic/maleic polymer which is generally considered to be a more weight-effective builder than citrate.

25 As indicated below, especially preferred detergency builder additives in accordance with the invention contain a citrate as the sole organic sequestrant builder.

The alkali metal aluminosilicate

30 The alkali metal (preferably sodium) aluminosilicates used in the detergency builder additive of the invention may be either crystalline or amorphous or mixtures thereof, and they have the general formula:  $0.8-1.5 \text{ Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 0.8-6 \text{ SiO}_2$ .

These materials contain some bound water and are required to have a calcium ion exchange capacity of at least about 50 mg CaO/g. The preferred sodium aluminosilicates contain 1.5-3.5  $\text{SiO}_2$  units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

Suitable crystalline sodium aluminosilicate ion-exchange detergency builders are described, for example, in GB 1 473 201 (Henkel) and GB 1 429 143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A and X, and mixtures thereof. Especially preferred is zeolite 4A.

The novel zeolite P described and claimed in our copending British Patent Applications Nos. 88 25783.7 filed 3 November 1988 and 89 10029.1 filed 2 May 1989 may also be used in the detergency builder additive of the invention.

45 Calculated on an anhydrous basis, the builder additive of the invention contains at least 50 wt%, preferably from 50 to 65 wt%, more preferably from 55 to 65 wt%, of aluminosilicate.

Sodium carbonate

50 An important characteristic of the builder additive of the invention is the relatively high content of sodium carbonate: 15 to 20 wt%.

Use of 15 wt% or more of sodium carbonate apparently enables a substantially smaller amount of organic sequestrant builder to be used without loss of building efficacy; while levels above 20 wt%, which might give rise to objections of excessive alkalinity and a requirement under some legislations for hazard warning labelling, have been found to be unnecessary.

The use of the builder additive of the invention in conjunction with a separate main wash powder enables builder level to be tailored to suit conditions of water hardness so that optimum alkalinity and wash

performance are obtained.

#### Total inorganic builder content

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The total amount of aluminosilicate and sodium carbonate in the builder additive of the invention is preferably at least 70 wt%, and more preferably at least 75 wt%.

#### The organic sequestrant builder

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The organic sequestrant builder is present in an amount of from 2 to 12 wt%. If more than one organic sequestrant builder is present, the total amount should lie within this range. Preferably the amount of organic sequestrant builder present is from 2 to 10 wt%, more preferably from 2 to 7 wt%, and most preferably from 4 to 7 wt%.

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The organic sequestrant builder is preferably a polycarboxylate material. Polycarboxylate detergency builders are well known to the skilled detergent formulator, and include polymeric polycarboxylates such as polyacrylates, acrylic/maleic copolymers and polyacetal carboxylates, and monomeric polycarboxylates such as nitrilotriacetates, citrates, oxydisuccinates, tartrate monosuccinates and disuccinates, and carboxymethyloxysuccinates; this list is not intended to be exhaustive. A mixture of two or more organic sequestrant builders may if desired be present.

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According to a preferred embodiment of the invention, the organic sequestrant builder comprises a water-soluble salt of citric acid, for example, an alkali metal citrate or ammonium citrate, conveniently sodium citrate. Citrates have the known advantage of being readily biodegradable and environmentally innocuous.

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According to an especially environmentally friendly embodiment of the invention, the builder additive contains a water-soluble citric acid salt as the sole organic sequestrant builder.

Other polycarboxylate builders known to have environmental advantages, for example, carboxymethyloxysuccinates, may alternatively be used.

30

Another class of polycarboxylate builders suitable for use in the detergency builder additive of the invention comprises the water-soluble salts of polymeric polycarboxylic acids. Especially preferred are water-soluble salts of homo- and copolymers of acrylic acid, and more especially acrylic/maleic copolymers. Like citrates, these are conveniently and preferably used in sodium salt form.

The detergency builder additive of the invention may also, if desired, contain both polymer and citrate, provided that the total amount of the two is within the range of 2 to 12 wt%; but the use of a single organic sequestrant, especially citrate, is generally preferred for good performance combined with simplicity and ease of formulation.

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Polymeric polycarboxylate builders which may suitably be used, alone or in combination, in the detergency builder additive of the invention include the following:

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	<u>Polymer type</u>	<u>Trade name</u>	<u>Supplier</u>	<u>Average m wt</u>
5	Polyacrylate	Versicol* E5	Allied Colloids	3500
10	Polyacrylate	Versicol* E7	Allied Colloids	27 000
15	Polyacrylate	Versicol* E9	Allied Colloids	70 000
20	Polyacrylate	Narlex* LD 30	National Adhesives	5000
25	Polyacrylate	Narlex* LD 34	National Adhesives	25 000
30	Polyacrylate	Acrysol* LMW-10	Röhm & Haas	1000
35	Polyacrylate	Acrysol* LMW-20	Röhm & Haas	2000
40	Polyacrylate	Acrysol* LMW-45	Röhm & Haas	4500
45	Polyacrylate	Acrysol* A1-N	Röhm & Haas	60 000
50	Polyacrylate	Sokalan* PA-20	BASF	2500
55				

	<u>Polymer type</u>	<u>Trade name</u>	<u>Supplier</u>	<u>Average m wt</u>
5	Polyacrylate	Sokalan* PA-40	BASF	15 000
10	Polyacrylate	Sokalan* PA-70	BASF	70 000
15	Polyacrylate	Sokalan* PA-110	BASF	250 000
20	Ethylene/ maleic acid .	EMA*	Monsanto	
25	Methyl vinyl ether/maleic acid	Gantrez* AN 119	GAF Corporation	
30	Acrylate/ maleate	Sokalan* CP5, CP7	BASF	
35	Acrylic phosphinate	DKW*	National Adhesives	
40	Acrylic phosphinate	Belsperse*	Ciba-Geigy	

\*denotes Trade Mark

45

#### Ratio of aluminosilicate to organic builder

The ratio of aluminosilicate to organic sequestrant builder is preferably within the range of from 5:1 to 30:1, more preferably from 10:1 to 15:1.

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#### Preparation of the builder additive

The builder additive is suitably prepared by processes involving dry-mixing of the sodium carbonate with the remaining ingredients.

55

The alkali metal aluminosilicate is conveniently incorporated in the form of a granular spray-dried material, for example, as described in EP 191 187B, EP 195 127A, EP 279 038A, EP 279 039A and EP 279

040A (Degussa).

If the organic sequestrant builder is available in granular form, that may conveniently be dry-mixed with the granular aluminosilicate and the sodium carbonate. Both sodium citrate and acrylic/maleic copolymer may conveniently be incorporated by this method.

5 An alternative procedure involves spraying an aqueous solution of the organic sequestrant builder onto the granular aluminosilicate, and then dry-mixing the resulting granules with the sodium carbonate.

Other suitable processes for preparing the detergency builder additive of the invention will readily suggest themselves to the skilled detergent formulator.

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#### Other ingredients

The detergency builder additive of the invention may if desired contain minor amounts of other ingredients, for example, processing aids, binders, inorganic salts, perfumes or colourants. Some of these  
15 minor ingredients may be imported directly from granular aluminosilicate raw material.

As indicated previously, the detergency builder additive is substantially free of organic surfactants. That does not preclude the presence of low levels, for example, up to 5 wt% and preferably not more than 3 wt%, of surfactant material, for example, imported from commercially available aluminosilicate granules, as a binder or processing aid.

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#### The Baukasten detergent system

In its second aspect the invention is concerned with a wash system of the Baukasten (building block)  
25 type which provides in separate packs a main wash powder, adequate in itself for lightly soiled loads and low water hardness, and a builder additive that can be used to supplement the main wash powder when the load is heavily soiled or the water hardness high. The Baukasten system has the environmental benefit of reducing the unnecessary usage of chemicals.

The main wash powder is thus sufficiently fully formulated to enable it to be used alone under  
30 favourable wash conditions. It contains at least one organic surfactant, and at least one detergency builder.

The total amount of detergent-active material (surfactant) in the main wash powder is suitably from 2 to 50% by weight, and is preferably from 5 to 40% by weight.

The main wash powder may contain one or more soap or non-soap anionic, nonionic, cationic, amphoteric or zwitterionic surfactants, or combinations of these. Many suitable detergent-active compounds  
35 are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic surfactants and nonionic surfactants.

Synthetic anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene  
40 sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of C<sub>8</sub>-C<sub>15</sub>; primary and secondary alkyl sulphates, particularly sodium C<sub>12</sub>-C<sub>15</sub> primary alcohol sulphates; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

Suitable nonionic detergent compounds which may be used include in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols,  
45 acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Specific nonionic detergent compounds are alkyl (C<sub>6</sub>-<sub>22</sub>) phenol-ethylene oxide condensates, the condensation products of linear or branched aliphatic C<sub>8</sub>-<sub>20</sub> primary or secondary alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called nonionic detergent compounds include long-chain tertiary amine  
50 oxides, tertiary phosphine oxides, and dialkyl sulphoxides.

Especially preferred are the primary and secondary alcohol ethoxylates, especially the C<sub>12</sub>-<sub>15</sub> primary and secondary alcohols ethoxylated with an average of from 5 to 20 moles of ethylene oxide per mole of alcohol.

It may also be desirable to include one or more soaps of fatty acids. These are preferably sodium  
55 soaps derived from naturally occurring fatty acids, for example, the fatty acids from coconut oil, beef tallow, sunflower or hardened rape seed oil.

Detergent compositions suitable for use in automatic fabric washing machines generally contain anionic surfactant, or nonionic surfactant, or both together in any proportions, and soap may also be present if

desired.

The main wash powder also contains one or more detergency builders. Since it is intended that the performance of the main wash powder in hard or even moderately hard water should be boosted by the additional use of the detergency builder additive, the level of builder in the main wash powder may be relatively modest.

Detergency builders are well-known in the art. Inorganic builders that may be present include alkali metal (generally sodium) ortho-, pyro- and tripolyphosphate, the latter material being especially effective. According to a preferred embodiment of the invention, however, the main wash powder contains less than 10 wt% of inorganic phosphate, and is more preferably substantially free of inorganic phosphate.

Non-phosphate detergency builders are also well known to the skilled detergent formulator. Inorganic non-phosphate builders include alkali metal (generally sodium) carbonates and aluminosilicates; and examples of organic non-phosphate builders have been listed above in the description of the builder additive of the invention.

Preferably the main wash powder comprises a builder system based on an alkali metal aluminosilicate and/or an alkali metal carbonate. An example of a carbonate-based builder system suitable for use in the main wash powder is an alkali metal carbonate in combination with finely divided calcite as described and claimed in GB 1 437 950 (Unilever).

According to a preferred embodiment of the invention, however, the main wash powder contains a builder system similar to that of the builder additive, comprising an alkali metal aluminosilicate, optionally an alkali metal carbonate, and an organic sequestrant builder. The preferred organic sequestrant builders for use in the main wash powder are those mentioned above as preferred for use in the builder additive; the organic sequestrant builders used in the two components may be the same or different.

The preferred ratio of aluminosilicate to organic sequestrant builder in the main wash powder is within the range of from 5:1 to 30:1.

If desired, the main wash powder may contain other functional ingredients commonly encountered in detergent powders, for example, peroxy bleaches, bleach activators, bleach stabilisers, enzymes, fabric softeners, antifoams, antiredeposition agents, sodium silicate, perfumes and other materials well known to the skilled detergent formulator. Bleaching ingredients, however, are preferably separated out into a second additive, as described in more detail below, which need only be used when the soiling of the washload necessitates it.

The main wash powder is preferably free or substantially free of inert fillers, for example, inorganic salts such as sodium sulphate, that do not contribute to wash performance. This applies also to the builder additive and, if appropriate, the bleach additive. Small amounts of apparently inert ingredients, for example, inorganic salts may however be present in order to ensure that powder properties such as flow and resistance to caking are acceptable.

#### The bleach additive

The bleach additive which optionally, and preferably, forms part of the Baukasten detergent system of the invention, suitably comprises a persalt or peracid, if necessary or desired together with a bleach precursor and/or a bleach stabiliser.

The bleach additive may, for example, comprise a major proportion of an inorganic persalt, for example, sodium perborate monohydrate or tetrahydrate; a lesser proportion of a bleach precursor, for example, tetracetylene diamine; and any suitable minor ingredients, for example, bleach stabiliser, colourant, perfume.

#### The wash method

In its third aspect, the invention provides a method for washing soiled laundry in a domestic washing machine, which utilises the flexibility of the Baukasten detergent system defined above.

Thus when washing a lightly soiled load in soft water, the main wash powder alone may be employed. If the load is stained, the main wash powder is suitably used in conjunction with the bleach additive. In hard water conditions, the main wash powder and builder additive should both normally be used, if necessary in conjunction with the bleach additive. Different amounts of the main wash powder, the bleach and builder additives will generally be appropriate for different circumstances.

The main wash powder, the builder additive and (if present) the bleach additive will generally be packed



in separate containers within a larger outer container, and instructions will be provided to enable the consumer to use amounts and proportions of the components most suited to the wash conditions on any particular occasion.

Of course, it is also essential to the flexibility of the Baukasten detergent system that the main wash powder, the builder additive and (if appropriate) the bleach additive should also be available separately as refills, since supplies of the three components are unlikely to be exhausted simultaneously.

With the initially supplied multiple pack, instructions will be provided indicating recommended dosages of the various components under different conditions of water hardness, wash load size, degree of soiling of the washload, washing machine programme and other relevant parameters. The recommended amounts and proportions will of course depend on the exact formulations of those components.

According to a preferred embodiment of the invention, the various components of the Baukasten system of the present invention may be colour-coded to aid the consumer, that is to say, the main wash powder, the builder additive and (if present) the bleach additive may be of different colours. Preferably the main wash powder, which is the component used in the larger (or largest) amount, is uncoloured (ie of natural colour) in order to minimise the usage of colourant, while the builder additive and (if present) the bleach additive are deliberately given a distinctive appearance by the use of colourant.

The powders may be homogeneously coloured, or, preferably, may contain a small percentage - about 0.5 to 3 wt%, preferably 1 to 2 wt% - of coloured speckles, the bulk remaining of natural colour, thus achieving distinctiveness with a minimum usage of colourant. In a system containing both a builder additive and a bleach additive, the additives may contain differently coloured speckles, for example, green for the builder additive and blue for the bleach additive. The speckles should preferably consist simply of coloured particles of the component itself, or of a functional ingredient that would in any case be present either in the component in question or in another component.

The invention will now be illustrated by the following non-limiting Examples, in which parts and percentages are by weight unless otherwise stated.

#### EXAMPLES 1 TO 5, COMPARATIVE EXAMPLE A :

##### PREPARATION OF BUILDER ADDITIVES

##### Example 1

A builder additive was prepared to the following formulation by dry-mixing:

	<u>%</u>
Granular zeolite A <sup>1</sup>	77.0
Granular sodium citrate dihydrate	5.0
Sodium carbonate (anhydrous)	
- natural colour	15.9
- green speckles <sup>2</sup>	2.0
Perfume	0.1

<sup>1</sup>Wessalith (Trade Mark) CS ex Degussa

<sup>2</sup>containing 0.1 wt% green colourant

The true composition of this builder additive was as follows:

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	Builder additive	Granular zeolite
	%	%
Zeolite A (anhydrous basis)	58.52	76.0
Sodium carbonate	17.9	-
Sodium sulphate	2.23	2.9
Sodium hydroxide	0.38	0.5
Sodium citrate	5.0	-
Nonionic surfactant	2.0	2.6
Sodium carboxymethyl cellulose	1.54	2.0
Perfume	0.1	-
Water	12.32	16.0

This builder additive contained 76.52 wt%, in total, of zeolite (anhydrous basis) and sodium carbonate; and 5.0 wt% of organic sequestrant builder (sodium citrate).

The ratio of aluminosilicate to organic sequestrant in this builder additive was 11.7 : 1.

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#### Comparative Example A

25

A builder additive in accordance with EP 243 908A (Henkel) was prepared to the following formulation:

30

	%
Zeolite A (anhydrous basis)	62.5
Sodium carbonate	5.1
Sodium citrate	10.5
Acrylic/maleic copolymer <sup>3</sup>	4.2
Sodium carboxymethylcellulose	0.3
Water	17.5

35

<sup>3</sup>Sokalan (Trade Mark) CP5 ex BASF

This builder additive contained 67.6 wt%, in total, of zeolite (anhydrous basis) and sodium carbonate; and 14.7 wt%, in total, of organic sequestrant builder (sodium citrate and acrylic/maleic copolymer).

The ratio of aluminosilicate to organic sequestrant in this builder additive was 4.25 : 1.

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#### Example 2

A builder additive was prepared to the following formulation by dry-mixing:

50

	%
Granular zeolite A	75.0
Granular sodium citrate dihydrate	5.0
Sodium carbonate (anhydrous)	20.0

55

The true composition of this builder additive was as follows:

## EP 0 419 036 A2

		Builder additive	Granular zeolite
		%	%
5	Zeolite A (anhydrous basis)	57.0	76.0
	Sodium carbonate	20.0	-
	Sodium sulphate	2.18	2.9
	Sodium hydroxide	0.38	0.5
10	Sodium citrate	5.0	-
	Nonionic surfactant	1.95	2.6
	Sodium carboxymethyl cellulose	1.5	2.0
	Water	12.0	16.0

15 This builder additive contained 77.0 wt%, in total, of zeolite (anhydrous basis) and sodium carbonate; and 5 wt% of organic sequestrant builder (sodium citrate).

The ratio of aluminosilicate to organic sequestrant in this builder additive was 11.4 : 1.

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### Example 3

A builder additive was prepared by dry-mixing the following materials to the following formulation:

25

	%
Granular zeolite A	80.0
Granular acrylic/maleic copolymer	5.0
30 Sodium carbonate (anhydrous)	15.0

The true composition of this builder additive was as follows:

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		Builder additive	Granular zeolite
		%	%
40	Zeolite A (anhydrous basis)	60.8	76.0
	Sodium carbonate	15.0	-
	Sodium sulphate	2.32	2.9
	Sodium hydroxide	0.4	0.5
	Acrylic/maleic copolymer	5.0	-
45	Nonionic surfactant	2.08	2.6
	Sodium carboxymethyl cellulose	1.6	2.0
	Water	12.8	16.0

50 This builder additive contained 75 wt%, in total, of zeolite (anhydrous basis) and sodium carbonate; and 5.0 wt% of organic sequestrant builder (acrylic/maleic copolymer).

The ratio of aluminosilicate to organic sequestrant in this builder additive was 12.16 : 1.

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### Example 4

A builder additive was prepared by the method of Example 1 to the following formulation:

	<u>%</u>
Granular zeolite A	75.0
Granular acrylic/maleic copolymer	5.0
Sodium carbonate (anhydrous)	20.0

The true composition of this builder additive was as follows:

	<u>Builder additive</u>	<u>Granular zeolite</u>
	<u>%</u>	<u>%</u>
Zeolite A (anhydrous basis)	57.0	76.0
Sodium carbonate	20.0	-
Sodium sulphate	2.18	2.9
Sodium hydroxide	0.38	0.5
Acrylic/maleic copolymer	5.0	-
Nonionic surfactant	1.95	2.6
Sodium carboxymethyl cellulose	1.5	2.0
Water	12.0	16.0

This builder additive contained 77.0 wt%, in total, of zeolite (anhydrous basis) and sodium carbonate; and 5.0 wt% of organic sequestrant builder (acrylic/maleic copolymer).

The ratio of aluminosilicate to organic sequestrant in this builder additive was 11.4 : 1.

#### Example 5

A builder additive was prepared to the following formulation by spraying an aqueous solution of the acrylic/maleic copolymer onto the granular zeolite, and then dry-mixing the sodium carbonate and sodium carbonate speckles:

		<u>%</u>
Granular zeolite A		78.0
Acrylic/maleic copolymer		4.5
Sodium carbonate (anhydrous)	- natural colour	15.5
	- green speckles	2.0

The true composition of this builder additive was as follows:

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10

	Builder additive	Granular zeolite
	%	%
Zeolite A (anhydrous basis)	59.28	76.0
Sodium carbonate	17.5	-
Sodium sulphate	2.26	2.9
Sodium hydroxide	0.39	0.5
Acrylic/maleic copolymer	4.5	-
Nonionic surfactant	2.03	2.6
Sodium carboxymethyl cellulose	1.56	2.0
Water	12.48	16.0

15

This builder additive contained 76.78 wt%, in total, of zeolite (anhydrous basis) and sodium carbonate; and 4.5 wt% of organic sequestrant builder (acrylic/maleic copolymer).

The ratio of aluminosilicate to organic sequestrant in this builder additive was 13.17 : 1.

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#### EXAMPLES 6 TO 11 - BAUKASTEN SYSTEMS

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##### Example 6

A main wash powder was prepared to the following formulation by spray-drying and postdosing:

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	%
Linear alkylbenzene sulphonate	14.40
Nonionic surfactant	6.40
Zeolite (anhydrous)	38.38
Sodium silicate	1.12
Sodium carbonate	12.60
Acrylic/maleic copolymer	4.70
Proteolytic enzyme	0.60
Antifoam granules*	4.50
Minor ingredients, salts, moisture	to 100.00

\*as disclosed in Example 3 of EP 266 863A (Unilever).

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This main wash powder was used to form a Baukasten detergent system together with the builder additive of Example 5 above, and a bleach additive having the following composition, prepared by dry mixing:

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	%
Sodium perborate monohydrate	74.25
TAED granules (83 wt% active)	24.75
Blue speckles (of main wash powder)	1.0

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Recommended dosages for use in a standard washing machine (wash liquor volume about 20 litres) were as follows:

Main wash powder 80 or 110 g (main wash), 55 g (prewash);

Builder additive 0, 10, 20 or 30 g;

Bleach additive 0, 20 or 30 g.

Usage of the main wash powder and the bleach additive will depend on soiling conditions (and thus machine programme selected), as follows:

5 Light soil, fine/coloured wash:

80 g main wash powder

Light soil, other wash:

80 g main wash powder,

20 g bleach additive

10 Normal soil:

110 g main wash powder

30 g bleach additive

Heavy soil:

prewash

15 55 g main wash powder

main wash

110 g main wash powder

30 g bleach additive

Usage of the builder additive, however, will depend only on water hardness, and a suitable recommendation is as follows:

25

Water hardness 1 ( 0 - 7 ° German hard):	0 g
Water hardness 2 ( 7 - 14 ° German hard):	10 g
Water hardness 3 (14 - 21 ° German hard):	20 g
Water hardness 4 ( >21 ° German hard):	30 g

30 A Tergotometer (Trade Mark) experiment was carried out to show the effect of the builder additive in bringing hard water detergency performance up to soft water level.

The experiment was carried out using a commercially available test cloth (oily/particulate soil), a 40 ° C heat up and 30 minute wash cycle, and the results were as shown in Table 1.

Table 1

35

Water hardness ( ° DH)	5.6	17
Main wash powder (g/l)	4	4
Builder additive (g/l)	0	1
Reflectance increase ( $\Delta R_{460^*}$ )	20.1	21.2

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#### Example 7

A main wash powder was prepared to the following formulation by spray-drying and postdosing:

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	%
Linear alkylbenzene sulphonate	12.0
Nonionic surfactant (7EO)	6.0
Nonionic surfactant (3EO)	8.0
Soap	1.5
Zeolite (anhydrous basis)	37.5
Sodium carbonate	12.5
Sodium silicate	1.0
Acrylic/maleic copolymer	2.2
Sodium carboxymethylcellulose	1.0
Proteolytic enzyme	0.8
Antifoam granules*	3.0
Water	14.5
	100.0

\*as disclosed in Example 3 of EP 266 863A (Unilever).

This main wash powder was used to form a Baukasten detergent system together with the builder additive of Example 1 above.

Detergencies under various conditions of water hardness were assessed in the Tergotometer (Trade Mark), using a 40 °C heat-up and 30-minute wash cycle. Detergencies were monitored by means of two different test cloths, reflectances at 460 nm being measured before and after the wash procedure on an ICS Micromatch Flectometer (Trade Mark). Conditions and results are shown in Table 2 below.

Table 2

Conditions				
Water hardness				
French (° FH)	7	20	32	46
German (° DH)	4	11	18	24
Main wash powder dosage (g/l)	3.5	3.5	3.5	3.5
Builder additive dosage (g/l)	0	0 or 1	0 or 2	0 or 3
Reflectance increase ( $\Delta R_{460}$ )				
Test cloth 1 (oil/silica/ink on cotton):				
without builder	25.33	23.03	19.73	18.03
with builder	-	25.63	25.73	25.93
Test cloth 2 (kaolin/woolfat on cotton):				
without builder	17.18	12.58	13.78	15.58
with builder	-	18.48	19.08	18.18

### Example 8

A main wash powder was prepared to the following formulation by spray-drying and postdosing:

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This main wash powder was used to form a Baukasten detergent system together with the builder additive of Example 1 above.

Detergencies were assessed as described in Example 7, using various test cloths. Conditions and results are shown in Table 3 below.

20

Table 3

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Conditions				
Water hardness				
French (° FH)	7	20	32	46
German (° DH)	4	11	18	24
Main wash powder dosage (g/l)	4	4	4	4
Builder additive dosage (g/l)	0	0 or 1	0 or 2	0 or 3
Reflectance increase ( $\Delta R_{460^*}$ )				
Test cloth 1 (oil/silica/ink on cotton):				
without builder	26.16	25.25	22.71	19.79
with builder	-	26.31	25.26	23.91
Test cloth 3 (casein on cotton):				
without builder	26.85	26.05	23.65	19.55
with builder	-	26.55	26.25	25.15
Test cloth 4 (Indian ink/olive oil on polyester/cotton):				
without builder	57.22	56.32	54.52	53.32
with builder	-	56.52	56.22	55.62

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#### Example 9

The effects of the builder additives of Example 1 and of Comparative Example A were compared using a Tergotometer as in Examples 7 and 8.

The main wash powder of Examples 8 and 9 was used, together with a bleach additive similar to that described in Example 6. Detergencies were compared using the Tergotometer as in Examples 6 to 8 (40 °C heat-up and wash). The results were as shown in Table 4.



Table 4Conditions

5

## Water hardness

10	French (°FH)	7	20	32	46
	German (°DH)	4	11	18	24

15	Main wash powder dosage (g/l)	4	4	4	4
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20	Bleach additive dosage (g/l)	1	1	1	1
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25	Builder additive dosage (g/l)	0	1	2	3
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Table 4 (continued)

5 Reflectance increase ( $\Delta R_{460*}$ )

Test cloth 1 (oil/silica/ink on cotton):

10	without builder	31.90	-	-	-
	Example 1	-	31.50	31.30	31.10
	Example A	-	31.30	30.90	30.80

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Test cloth 2 (kaolin/woolfat on cotton):

20	without builder	32.35	-	-	-
	Example 1	-	31.55	32.05	31.85
	Example A	-	31.85	30.95	31.35

25

Test cloth 3 (casein on cotton):

30	without builder	30.71	-	-	-
	Example 1	-	31.51	31.41	30.81
	Example A	-	31.01	30.51	30.41

35

Test cloth 5 (tea on cotton):

40	without builder	12.48	-	-	-
	Example 1	-	16.38	16.08	15.58
	Example A	-	12.98	13.88	13.38

45 It will be seen that the builder additive of Example 1 generally performed at least as well as the prior art builder additive of Comparative Example A, despite its substantially lower content of organic sequestrant and the absence of acrylic/maleic copolymer.

#### Example 10

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In this Example, the effect of the builder additive of Example 1 on the detergency of the main wash powder of Example 8 in 30° (French) hard water was investigated using an AEG Lavamat (Trade Mark) washing machine, using the 60° C wash cycle and a 2.5 kg load of clean cotton sheeting and terry towelling. Detergency was monitored by means of various test cloths. Conditions and results were as shown in Table 5.

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#### Example 11

The procedure of Example 10 was repeated using 40° (French) hard water. Conditions and results were as shown in Table 6.

Table 5

Conditions		
Water hardness (° FH)	12	30
Main wash powder dosage (g)	55	55
Builder additive dosage (g)	0	0 or 30
Reflectance increase ( $\Delta R_{460^*}$ )		
Test cloth 1 (oil/silica/ink on cotton):		
without builder	32.9	21.6
with builder	-	29.61
Test cloth 6 (casein on cotton):		
without builder	34.87	29.58
with builder	-	33.40
Test cloth 7 (oil/silica/ink on cotton):		
without builder	27.83	17.61
with builder	-	24.14

Table 6

Conditions		
Water hardness (° FH)	12	40
Main wash powder dosage (g)	55	55
Builder additive dosage (g)	0	0 or 30
Reflectance increase ( $\Delta R_{460^*}$ )		
Test cloth 1 (oil/silica/ink on cotton):		
without builder	30.97	22.69
with builder	-	31.95
Test cloth 2 (kaolin/woolfat on cotton):		
without builder	31.40	28.50
with builder	-	34.30
Test cloth 6 (casein on cotton):		
without builder	34.92	30.31
with builder	-	33.75
Test cloth 8 (kaolin/woolfat on polyester):		
without builder	8.03	2.50
with builder	-	7.10

## Claims

- 1 A detergency builder additive, for use in the washing of soiled laundry, in conjunction with a main wash detergent powder comprising one or more organic surfactants and one or more detergency builders, the  
5 builder additive being substantially free of organic surfactants and being characterised in that it comprises:
  - (i) at least 50 wt% (anhydrous basis) of a water-insoluble alkali metal aluminosilicate,
  - (ii) from 15 to 20 wt% of sodium carbonate.
  - (iii) from 2 to 12 wt% of organic sequestrant builder.
- 2 A detergency builder additive as claimed in claim 1, characterised in that the total of aluminosilicate (i)  
10 and sodium carbonate (ii) is at least 70 wt%.
- 3 A detergency builder additive as claimed in claim 2, characterised in that the total of aluminosilicate (i) and sodium carbonate (ii) is at least 75 wt%.
- 4 A detergency builder additive as claimed in any preceding claim, characterised in that the amount of organic sequestrant builder (iii) is within the range of from 2 to 7 wt%.
- 15 5 A detergency builder additive as claimed in any preceding claim, characterised in that the amount of aluminosilicate (i) is within the range of from 55 to 65 wt%.
- 6 A detergency builder additive as claimed in any preceding claim, characterised in that the ratio of aluminosilicate (i) to organic sequestrant builder (iii) is within the range of from 10:1 to 15:1.
- 7 A detergency builder additive as claimed in any preceding claim, characterised in that the organic  
20 sequestrant builder comprises a water-soluble salt of citric acid.
- 8 A detergency builder additive as claimed in claim 7, characterised in that the organic sequestrant builder consists solely of a water-soluble salt of citric acid.
- 9 A detergency builder additive as claimed in any one of claims 1 to 6, characterised in that the organic sequestrant builder comprises a water-soluble salt of a homo- or copolymer of acrylic acid.
- 25 10 A detergent system of the Baukasten (building block) type for washing soiled laundry, characterised in that it comprises as separate components:
  - (a) a main wash powder comprising one or more organic surfactants and one or more detergency builders;
  - (b) a builder additive as claimed in any one of claims 1 to 9;
  - and, optionally,
  - 30 (c) a bleach additive.
- 11 A detergent system as claimed in claim 10, characterised in that the main wash powder (a) contains an alkali metal aluminosilicate and an organic sequestrant builder selected from water-soluble salts of citric acid, water-soluble salts of homo- and copolymers of acrylic acid, and mixtures thereof.
- 12 A detergent system as claimed in claim 10 or claim 11, characterised in that the main wash powder (a),  
35 the builder additive (b) and, if present, the bleach additive (c), are of different colours.
- 13 A detergent system as claimed in claim 12, characterised in that the builder additive (b) contains coloured speckles.
- 14 A detergent system as claimed in Claim 13, characterised in that a bleach additive is present, and the builder additive (b) and the bleach additive (c) contain differently coloured speckles.
- 40 15 A method for washing soiled laundry in a domestic washing machine, characterised in that the method includes the steps of separately introducing into the washing machine
  - (a) a main wash powder comprising one or more organic surfactants and one or more detergency builders;
  - (b) a builder additive as claimed in any one of claims 1 to 9;
  - and, optionally,
  - 45 (c) a bleach additive.

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