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(54) Detergent compositions.

(57) A high-foaming liquid detergent composition, suitable *inter alia* for hand dishwashing, contains from 2 to 60% by weight of an active detergent system including a dialkyl sulphosuccinate (at least 2%), and also contains an added magnesium salt providing from 0.02 to 0.24% by weight of magnesium ions. The last-mentioned ingredient gives improved product stability, as demonstrated by a lower clear point, and increased viscosity at low concentrations, as well as enhanced soft water foaming performance.

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DETERGENT COMPOSITIONS

The present invention relates to high-foaming liquid detergent compositions suitable for use in fabric washing, shampoos, and above all, in manual dishwashing operations in both hard and soft water.

The term "dishes" as used herein means any utensils involved in food preparation or consumption which may be required to be washed to free them from food particles and other food residues, greases, proteins, starches, gums, dyes, oils and burnt organic residues.

GB 1 429 637 (Unilever) discloses hand dishwashing compositions containing as detergent-active material a water-soluble salt of a di(C_7-C_9) alkyl ester of sulphosuccinic acid, in combination with an alkyl sulphate or an alkyl ether sulphate.

GB 2 108 520, GB 2 104 913, GB 2 105 325, EP 71413 and EP 71414 Unilever) disclose high-foaming detergent compositions based on certain dialkyl sulphosuccinates, particularly those having C_6 and C_8 chains.

GB 1 164 854 (Shell) discloses the inclusion of 0.5 to 10% by weight of a water-soluble inorganic magnesium salt in aqueous liquid detergent compositions based on an

alkyl or aryl sulphonate in conjunction with an alkyl ether sulphate or a nonionic detergent.

The present invention is based on the observation that the presence of small quantities of magnesium ions, derived from an added electrolyte, in liquid detergent compositions based on dialkyl sulphosuccinates gives both performance and formulation benefits.

The present invention accordingly provides a foaming liquid detergent composition in the form of a clear, stable aqueous solution containing from 2 to 60% by weight of active detergent comprising at least 2% by weight, preferably at least 5% by weight, based on the whole composition, of a water-soluble salt of a dialkyl sulphosuccinic acid, the composition containing at least 0.02% by weight of magnesium ions derived from an added electrolyte.

According to the invention, magnesium ions arrive in the composition as part of an added water-soluble magnesium salt, for example, magnesium sulphate, chloride, or toluene or xylene sulphonate.

The presence of magnesium ions has been found to give improved foaming performance in very soft water. This effect has already been observed with detergent-active materials other than dialkyl sulphosuccinates, for example, alkylbenzene sulphonates, but the addition of magnesium salts is generally detrimental to low-temperature product stability, causing both cloud and clear points to rise. It should be explained that the cloud point is the temperature at which clarity of the composition is lost as the external temperature is lowered; the clear point is the temperature at which a clear solution is regained as the external temperature is raised again.

Surprisingly, it has been found that the addition of low levels of magnesium salts to liquid compositions based on dialkyl sulphosuccinates actually causes a lowering of the clear point, and in relatively low-concentration formulations also increases the viscosity.

The amount of added magnesium salt is within the range of from 0.02 to 0.24 % by weight of magnesium ions. The optimum level of addition within these limits for any particular formulation will depend on the total active detergent concentration, the total level and type of electrolyte present and the hydrotrope level, and can readily be determined experimentally by one skilled in the art.

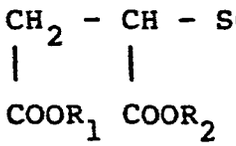
The magnesium ions are added to the compositions of the invention in the form of water-soluble magnesium salts which are electrolytes. The preferred salts are magnesium sulphate and magnesium chloride. For magnesium chloride hexahydrate, the limits given above correspond to 0.17 to 2.02 % by weight; for magnesium sulphate heptahydrate, the limits given above correspond to 0.21 to 2.46 % by weight. Accordingly the amount of added magnesium salt present in the compositions of the invention is preferably from 0.15 to 2.50% by weight, and more preferably from 0.50 to 1.50% by weight.

The total active detergent level of the compositions of the invention can range from 2 to 60% by weight, preferably from 5 to 40% by weight. The addition of magnesium salts in accordance with the invention is especially beneficial at total active detergent levels of 30% by weight and below. At the low end of the concentration range, 20% and below, the additional benefit of viscosity increase is observed. Thus the present invention alleviates the problem of low viscosities that

can arise with low-active-detergent dialkyl sulphosuccinate-based compositions.

The dialkyl sulphosuccinate, which is an essential detergent-active material in the composition of the invention, may be either symmetrical (both alkyl groups the same) or unsymmetrical. It may if desired be constituted by a mixture of materials of different chain lengths, of which the individual dialkyl sulphosuccinates themselves may be either symmetrical or unsymmetrical.

The detergent-active dialkyl sulphosuccinates are compounds of the formula I:



(I)

wherein each of R_1 and R_2, which may be the same or different, represents a straight-chain or branched-chain alkyl group having from 3 to 12 carbon atoms, preferably from 4 to 10 carbon atoms and more preferably from 6 to 8 carbon atoms, and X_1 represents a solubilising cation, that is to say, any cation yielding a salt of the formula I sufficiently soluble to be detergent-active. In the context of the present invention, the solubilising cation X_1 will generally be monovalent, for example, alkali metal, especially sodium; ammonium; or substituted ammonium, for example, ethanolamine.

The alkyl groups R_1 and R_2 in the dialkyl sulphosuccinate are preferably straight-chain or (in mixtures) predominantly straight-chain.

Among dialkyl sulphosuccinates that may advantageously be used in the composition of the invention

are the C_6/C_8 unsymmetrical materials described and claimed in GB 2 105 325 (Unilever); the dioctyl sulphosuccinate/ dihexyl sulphosuccinate mixtures described and claimed in GB 2 104 913 (Unilever) and the mixtures of symmetrical and unsymmetrical dialkyl sulphosuccinates described and claimed in GB 2 108 520 (Unilever).

The composition of the invention may of course with advantage contain other detergent-active agents in addition to the dialkyl sulphosuccinate, provided that the total composition contains at least 2% by weight, preferably at least 5%, of dialkyl sulphosuccinate(s). The composition may, for example, include one or more of the sulphonate-type detergents conventionally used as the main detergent-active agent in liquid compositions, for example, alkylbenzene sulphonates (especially C_9-C_{15} linear alkylbenzene sulphonates), secondary alkane sulphonates, alpha-olefin sulphonates, alkyl glyceryl ether sulphonates, and fatty acid ester sulphonates. Of course dialkyl sulphosuccinates are themselves sulphonate-type detergents. If such additional sulphonate-type materials are present, the total sulphonate preferably predominates in the active detergent mixture of the composition of the invention. If no such additional sulphonate-type materials are present, the sulphosuccinate alone preferably predominates.

Among the additional sulphonate-type detergents that may be present, alkylbenzene sulphonates and secondary alkane sulphonates are especially preferred. The ratio of dialkyl sulphosuccinate to alkylbenzene sulphonate or secondary alkane sulphonate is preferably within the range of from 4:1 to 0.1:1, more preferably from 2.5:1 to 1:1.

Our copending application of even date, claiming priority from British Patent Application No. 82 32643 filed on 16 November 1982, describes and claims liquid detergent

compositions containing dialkyl sulphosuccinate, alkylbenzene sulphonate and/or secondary alkane sulphonate, and alkyl ether sulphate.

If desired there may also be present one or more primary or secondary alkyl sulphates. If present, these together with any sulphonate material as mentioned above, including the dialkyl sulphosuccinate, preferably predominate in the active detergent mixture of the composition of the invention.

The composition of the invention advantageously contains one or more further detergent-active materials in addition to the dialkyl sulphosuccinate and optional additional sulphonate already mentioned. Preferably there are present one or more alkyl ether sulphates and/or one or more ethoxylated nonionic detergents having an alkyl chain length of C_8 to C_{15} and an average degree of ethoxylation of from 5 to 14.

Preferred alkyl ether sulphates are materials of the general formula

$$R_3 - O - (CH_2CH_2O)_n - SO_3X_2$$

wherein R_3 is a C_{10} to C_{18} alkyl group, X_2 is a solubilising cation, and n, the average degree of ethoxylation, is from 1 to 12. R_3 is preferably a C_{11} to C_{15} alkyl group and n is preferably from 1 to 8.

In any given alkyl ether sulphate, a range of differently ethoxylated materials, and some unethoxylated material, will be present and the value of n represents an average. The unethoxylated material is, of course, alkyl sulphate. If desired, additional alkyl sulphate may be admixed with the alkyl ether sulphate, to give a mixture in

which the ethoxylation distribution is more weighted towards lower values.

It is especially preferred to use alkyl ether sulphates containing less than 20% by weight of C_{14} and above material, as described and claimed in our copending application of even date, claiming priority from British Patent Application No. 82 32686 filed on 16 November 1982.

Examples of preferred ether sulphates for use in the present invention are Dobanol (Trade Mark) 23-3 and Dobanol (Trade Mark) 23-2 ex Shell, both based on C_{12} to C_{13} (50% of each) primary alcohol (about 75% straight chain, 25% 2-methyl branched), and having average degrees of ethoxylation n of 3 and 2 respectively.

The ratio of dialkyl sulphosuccinate, plus any other sulphonate-type detergent present plus any alkyl sulphate present other than that intrinsically present in the ether sulphate, to ether sulphate, is preferably within the range of from 5:1 to 0.5 to 1, more preferably from 3:1 to 1:1.

Nonionic detergents that may be used in the composition of the invention include short-chain high-foaming ethoxylated alcohols of the general formula III:

$$R_4 - O - (CH_2CH_2O)_m - H \qquad (III)$$

wherein R_4 is an alkyl group, preferably straight-chain, having from 8 to 12 carbon atoms, and the average degree of ethoxylation m is from 5 to 12.

The weight ratio of alkyl ether sulphate to nonionic detergent is preferably at least 1:1 and more preferably

within the range of from 1.5:1 to 3:1, especially about 2:1. An especially preferred nonionic detergent is Dobanol (Trade Mark) 91-8 ex Shell, in which R_4 is C_9-C_{11} (predominantly straight-chain) and m is 8.

Advantageously the compositions of the invention include within their active detergent mixture a C_{10}-C_{18} carboxylic acid (C_2-C_3 alkanol)amide, preferably a diethanolamide. The inclusion of this material, at a level not exceeding 30% by weight of the active detergent mixture, gives both performance and formulation advantages. Our copending application of even date, claiming priority from British Patent Application No. 82 32688 filed on 16 November 1982, describes and claims liquid detergent compositions containing a dialkyl sulphosuccinate, an alkyl ether sulphate and/or a nonionic detergent, and a C_{10}-C_{18} carboxylic acid di(C_2-C_3)alkanolamide, the last-mentioned component being present in an amount not exceeding 30% by weight of the total active detergent present.

As well as active detergent, magnesium ions and water, the composition of the invention will generally need to contain one or more hydrotropes. These are materials present in a formulation to control solubility, viscosity, clarity and stability, but which themselves make no active contribution to the performance of the product. Examples of hydrotropes include lower aliphatic alcohols, especially ethanol; urea; lower alkylbenzene sulphonates such as sodium toluene and xylene sulphonates; and combinations of these. Hydrotropes are expensive and take up room in a formulation without contributing to its performance, and it is therefore desirable to use as small quantities of them as possible.

The compositions of the invention may also contain

the usual minor ingredients such as perfume, colour, preservatives and germicides.

The liquid detergent compositions of the invention, containing 2 to 60% by weight of active detergent in clear, stable aqueous solution, may be used for all normal detergent purposes where foaming is advantageous, for example, fabric washing products, general purpose domestic and industrial cleaning compositions, carpet shampoos, car wash products, personal washing products, shampoos, foam bath products, and, above all, manual dishwashing.

The invention is further illustrated by the following non-limiting Examples.

EXAMPLES 1 & 2

The cloud points, clear points and viscosities of formulations containing dialkyl sulphosuccinate and ether sulphate, at a total active-detergent level of 24%, with 12% urea as hydrotrope, were compared at different levels of magnesium chloride hexahydrate.

The dialkyl sulphosuccinate used was a statistical mixture (mole ratio 1:2:1) of di-n-octyl sulphosuccinate, n-hexyl n-octyl sulphosuccinate and di-n-hexyl sulphosuccinate, prepared from a 1:1 mixture of n-hexanol and n-octanol by the method described in Example 1 of GB 2 108 520 (Unilever). This material contained 2.5% of sodium sulphate in a 40% solution, so that in the formulations the sodium sulphate level was 1% by weight.

The ether sulphate used was Dobanol 23-2S ex Shell (50% C_{12}, 50% C_{13}; n = 2; sodium salt).

The magnesium was added in the form of magnesium

chloride hexahydrate.

The results were as follows:

	A	1	2
Sulphosuccinate	16	16	16
Dobanol 23-2S	8	8	8
Urea	12	12	12
$MgCl_2.6H_2O$	-	0.5	1.0
Cloud point (°C)	-8	-5	3
Clear point (°C)	13.5	-1	7
Viscosity (cp)	153	166	162

Although the cloud point rose slightly with increasing magnesium ion level, the clear point was depressed considerably at 0.5% $MgCl_2.6H_2O$, (0.06% Mg^{2+}) giving an advantage in terms of product stability; at 1.0% $MgCl_2.6H_2O$ (0.12% Mg^{2+}) this advantage was smaller, and was offset by the rise in the cloud point. There was no significant effect on viscosity.

EXAMPLES 3 & 4

Examples 1 and 2 were repeated using a dialkyl sulphosuccinate sample containing a lower level (1%) of sodium sulphate, so that the overall level of that material

in the composition was only 0.4% by weight. At this lower electrolyte level the urea content could be reduced to 11%.

The results were as follows:

	B	3	4
Sulphosuccinate	16	16	16
Dobanol 23-2S	8	8	8
Urea	11	11	11
$MgCl_2.6H_2O$	-	0.5	1.0
Cloud point (°C)	-9	-8	-4
Clear point (°C)	15	14	2

It will be seen that at this lower electrolyte level, a higher amount of magnesium chloride hexahydrate was required to effect a significant lowering of the clear point.

EXAMPLES 5 & 6

Examples 3 and 4 were repeated using Dobanol 23-2A (the corresponding ammonium salt) instead of Dobanol 23-2S. The results were as follows:

	C	5	6
Sulphosuccinate	16	16	16
Dobanol 23-2A	8	8	8
Urea	10	10	10
$MgCl_2.6H_2O$	-	0.5	1.0
Cloud point (°C)	-8	-4	4
Clear point (°C)	8	0.5	6.5

As can be seen, the use of the ammonium salt of the ether sulphate instead of the sodium salt, at the same total electrolyte level, decreased the level of $MgCl_2.6H_2O$ required to give a clear point lowering from 1.0 to 0.5%, the level of 1.0% now being higher than optimum.

EXAMPLE 7 & 8

Examples 1 and 2 were repeated using a dialkyl sulpho-succinate sample free of electrolyte (sodium sulphate). The urea level was 10%.

The results were as follows:

	D	7	8
Sulphosuccinate	16	16	16
Dobanol 23-2S	8	8	8
Urea	10	10	10
$MgCl_2.6H_2O$	-	0.5	1.0
Cloud point (°C)	-9	-7	0
Clear point (°C)	15	16	2

It can be seen that a magnesium chloride level of 0.5% was insufficient to lower the clear point, but the higher level of 1.0% was effective.

EXAMPLE 9

This Example demonstrates the improvement in soft-water foaming performance obtained by the addition of magnesium chloride hexahydrate to a dialkyl sulpho-succinate/ether sulphate formulation.

The comparison was carried out by means of a plate-washing test in 1°H (French hardness) water.

In the test, plates soiled with a standard starch/fat/ fatty acid mixture were washed in a standard manner with 5 litres of test solution (total concentration 1 g/litre at 45°C) in a bowl, until only a third of the surface of the solution in the bowl was covered with foam. The number of plates washed before this arbitrary end-point

was reached was taken as an indicator of dishwashing and foaming performance.

The dialkyl sulphosuccinate used was the C_6/C_8 statistical mixture of Example 2, at a concentration of 16% by weight, and the ether sulphate was Dobanol 23-2S, at a concentration of 8% by weight.

This composition without added magnesium chloride hexahydrate washed 23 plates, and with the addition of 1.0% by weight of magnesium chloride hexahydrate washed 27 plates.

EXAMPLES 10 - 12

This experiment shows the effect of the addition of a magnesium salt to a relatively low-concentration (16%) formulation containing a fatty acid diethanolamide in addition to a dialkyl sulphosuccinate and an alkyl ether sulphate. The dialkyl sulphosuccinate was the C_6/C_8 mixture used in Examples 1 and 2, containing 2.5% by weight of electrolyte (sodium sulphate) in a 40% solution. The alkyl ether sulphate was Dobanol 23-3A, and the fatty acid diethanolamide was Empilan (Trade Mark) CDE ex Albright & Wilson, a coconut diethanolamide.

The results are shown in the following Table. All three levels of magnesium salt were effective in lowering the clear and cloud points, and all three levels raised the viscosity, the level of 1.0% being optimum in this latter respect.

	E	10	11	12
Sulphosuccinate	8	8	8	8
Dobanol 23-3A	4	4	4	4
Empilan CDE	4	4	4	4
Urea	8	8	8	8
$MgCl_2.6H_2O$	-	0.5	1.0	1.5
Cloud point (°C)	-7	-8	-8	-8
Clear point (°C)	3	-	-	-
Viscosity	50	113	133	104

EXAMPLES 13 & 14

The procedure of Examples 10-12 was repeated using a formulation having the same total active detergent level (16%) but containing a higher proportion (10%) of the dialkyl sulphosuccinate. In this case a magnesium salt level of 0.5% was found to give optimum benefits.

	F	13	14
Sulphosuccinate	10	10	10
Dobanol 23-3A	3	3	3
Empilan CDE	3	3	3
Urea	8	8	8
$MgCl_2.6H_2O$	-	0.5	1.0
Cloud point (°C)	-5	-8	1
Clear point (°C)	9	-	3
Viscosity (cp)	44	90	86

EXAMPLES 15 & 16

This experiment illustrates the effect of the addition of a magnesium salt on a relatively low-concentration (18%) formulation containing an alkylbenzene sulphonate (Dob (Trade Mark) 102 ex Shell) in addition to dialkyl sulphosuccinate and alkyl ether sulphate. The dialkyl sulphosuccinate was as used in Examples 10-14.

	G	15	16
Sulphosuccinate	10	10	10
Dob 102	4	4	4
Dobanol 23-3A	4	4	4
Urea	10	10	10
$MgCl_2.6H_2O$	-	0.5	1.0
Cloud point (°C)	-7	-7	-2
Clear point (°C)	10	10	2.5
Viscosity (cp)	27	60	104

Both levels of magnesium chloride raised the viscosity, the higher level of 1.0% being more effective. At 10% the clear point was also lowered, at the cost of a rise in the cloud point.

EXAMPLES 17 & 18

The procedure of Examples 15 and 16 was repeated at a slightly higher total active detergent level (20%).

	H	17	18
Sulphosuccinate	10	10	10
Dob 102	4	4	4
Dobanol 23-3A	6	6	6
Urea	10	10	10
$MgCl_2.6H_2O$	-	0.5	1.0
Cloud point (°C)	<-10	<-10	-8
Clear point (°C)	-	-	4.5
Viscosity (cp)	93	129	142

Comparative Composition H already had excellent low-temperature stability. The addition of 0.5% magnesium chloride increased the viscosity without detriment to the cloud point. The higher level of magnesium chloride (1.0%) improved the viscosity further to the slight, but insignificant, detriment of the low-temperature stability.

CLAIMS

1. A foaming liquid detergent composition in the form of a clear, stable aqueous solution containing from 2 to 60% by weight of an active detergent mixture comprising at least 2% by weight, based on the total composition, of a water-soluble salt of a dialkyl ester of sulphosuccinic acid, characterised in that the composition further comprises from 0.02 to 0.24% by weight of magnesium ions derived from an added electrolyte.

2. A detergent composition according to claim 1, characterised in that it comprises from 0.15 to 2.50% of an added inorganic magnesium salt selected from magnesium sulphate, magnesium chloride and mixtures thereof.

3. A detergent composition according to claim 2, characterised in that it comprises from 0.50 to 1.50% by weight of the added magnesium salt.

4. A detergent composition according to any one of claims 1 to 3, characterised in that the total concentration of active detergent is within the range of from 5 to 40% by weight.

5. A detergent composition according to claim 4, characterised in that the total concentration of active detergent is within the range of from 5 to 30% by weight.

6. A detergent composition according to claim 5, characterised in that the total concentration of active detergent is within the range of from 5 to 20% by weight.

7. A detergent composition according to any one of claims 1 to 6, characterised in that the dialkyl sulphosuccinate constitutes at least 5% by weight of the total composition.

8. A detergent composition according to any one of claims 1 to 7, characterised in that the alkyl groups of the dialkyl sulphosuccinate each have from 4 to 10 carbon atoms.

9. A detergent composition according to claim 8, characterised in that the alkyl groups of the dialkyl sulphosuccinate each have from 6 to 8 carbon atoms.

10. A detergent composition according to any one of claims 1 to 9, characterised in that the active detergent mixture further comprises an alkylbenzene sulphonate and/or a secondary alkane sulphonate.

11. A detergent composition according to claim 10, characterised in that the weight ratio of dialkyl sulphosuccinate to alkylbenzene sulphonate and/or secondary alkane sulphonate is within the range of from 2.5:1 to 1:1.

12. A detergent composition according to any one of claims 1 to 11, characterised in that the active detergent mixture further comprises a C_{10} to C_{18} alkyl polyethoxy sulphate having an average degree of ethoxylation of from 1 to 12.

13. A detergent composition according to claim 12, characterised in that the alkyl polyethoxy sulphate has an alkyl chain length of C_{11} to C_{15}.

14. A detergent composition according to claim 12 or claim 13, characterised in that the alkyl polyethoxy sulphate contains less than 20% by weight of material of chain length of C_{14} and above.

15. A detergent composition according to any one of claims 12 to 14, characterised in that the weight ratio of dialkyl sulphosuccinate, plus any alkylbenzene sulphonate and/or secondary alkane sulphonate present, to alkyl polyethoxy sulphate is within the range of from 5:1 to 0.5:1.

16. A detergent composition according to any one of claims 1 to 15, characterised in that the active detergent mixture further comprises a C_{10}-C_{18} carboxylic acid di(C_2-C_3 alkanol)amide, in an amount not exceeding 30% by weight of the active detergent mixture.

17. A detergent composition according to claim 1, characterised in that the active detergent system consists essentially of:

a) (i) a water-soluble salt of a dialkyl sulphosuccinic acid in which the alkyl groups may be the same or different, and, optionally,

(ii) an alkylbenzene sulphonate and/or a secondary alkane sulphonate,

and

b) (i) a C_{10}-C_{18} alkyl polyethoxy sulphate having an average degree of ethoxylation of from 1 to 12, and, optionally,

(ii) a C_8-C_{15} ethoxylated nonionic surfactant having an average degree of ethoxylation of from 5 to 14, the weight ratio of (b)(i) to (b)(ii) being at least 1:1,

the weight ratio of (a) to (b) being within the range of from 5:1 to 0.5:1, and, optionally,

c) a C_{10}-C_{18} carboxylic acid diC_2-C_3 alkanol) amide, in an amount of from 0 to 30% by weight of the total active detergent system.

European Patent Office

EUROPEAN SEARCH REPORT

0112045
Application number

EP 83 30 6947

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
D,A	GB-A-1 164 854 (SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ N.V.)		C 11 D 1/12 C 11 D 3/02
P,A	EP-A-0 071 410 (UNILEVER N.V.) * Page 33, example 16; claims 1, 8, 18 *		
A	DE-A-2 619 668 (UNILEVER N.V.) * Claim 1 *		
P,A	EP-A-0 071 412 (UNILEVER N.V.) * Claims 1, 3 *	1	
			TECHNICAL FIELDS SEARCHED (Int. Cl. ³)
			C 11 D 1/00 C 11 D 3/00

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	09-02-1984	SCHULTZE D

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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E : earlier patent document, but published on, or after the filing date
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