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(54) Title: PROCESS FOR TREATMENT OF TEXTILES AND RINSING AGENT FOR USE IN THE PROCESS

(57) Abstract

In the process for treatment of textiles by use of an enzyme the enzyme is used in a rinse liquor for a rinsing step after washing. The rinsing agent is an enzyme preparation stabilized against microbial infection. Use of the enzyme in the process provides a better enzymatic effect when used in the rinse liquor than when used as a constituent of the main wash composition.

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PROCESS FOR TREATMENT OF TEXTILES AND RINSING AGENT FOR USE IN THE PROCESS

TECHNICAL FIELD

The invention relates to a process for treatment of textiles by use of an enzyme and to a rinsing agent for use therein.

BACKGROUND ART

Treatment of soiled or unsoiled textiles normally comprises washing with a detergent containing anionic and/or nonionic surfactant, followed by rinsing and drying. It belongs to the prior art that lipase can be incorporated into the detergent to improve the removal of fat soiling (EP 130,064, EP 214,761, US 4,810,414). Also, it belongs to the prior art that a non-dusting granulate containing a cellulase can be added to a main wash detergent composition in order to reduce the harshness of the laundry (US 4,435,307). Furthermore, it belongs to the prior art that proteases are useful in detergent compositions in order to remove proteinaceous stains (US 3,723,250), and that amylases are useful in detergent compositions in order to remove starchy stains (US 3,627,688 and 4,548,727).

However, a given enzyme may be incompatible with a given detergent composition due to unsatisfactory activity and stability of the enzyme, and this imposes some limitations in the choice of enzyme and detergent formulation. It is the object of the invention to provide an alternative method of using enzyme in textile treatment, so as to obtain efficient enzyme effects for a given enzyme dosage while avoiding the above limitations.

STATEMENT OF THE INVENTION

We have found that, surprisingly, use of an enzyme provides a better enzymatic effect when used in the rinse liquor than when used as a constituent of the main wash composition. The effect is particularly pronounced after repeated cycles of washing, rinsing and drying. Detergent is essentially absent during rinsing, and thus compatibility problems are avoided. The enzyme may advantageously be added in the last rinse step together with a fabric softening and/or antistatic agent.

Accordingly, the invention provides a process for treatment of textiles by use of an enzyme, characterized by the fact that the enzyme is used in a rinse liquor for a rinsing step after washing.

In a preferred embodiment of the process according to the invention the enzyme is used in the rinse liquor for the rinsing step after washing together with a fabric softening agent and/or an antistatic agent. An even better enzymatic effect is observed in this embodiment, demonstrating a synergism between enzyme and fabric softening agent and/or antistatic agent.

In a preferred embodiment of the process according to the invention the fabric softening agent and/or the antistatic agent is a cationic surfactant. A satisfactory enzymatic effect is observed in this embodiment.

In a preferred embodiment of the process according to the invention the enzyme is used in the rinse liquor for a second or later rinsing step, preferably in the last rinsing step. In this manner an even better enzymatic effect is observed.

In a preferred embodiment of the process according to the invention the enzyme is a lipase and/or a cellulase and/or a protease and/or an amylase. These are the four most commonly used detergent enzymes.

In a preferred embodiment of the process according to the invention the enzyme is a lipase and/or a cellulase. In relation to these two enzymes the best improvement according to the invention has been demonstrated.

In a preferred embodiment of the process according to the invention the enzyme(s) is (are) of microbial origin. In this manner cheap enzymes can be provided.

In a preferred embodiment of the process according to the invention the enzyme is cellulase, the softening agent and the antistatic agent is a cationic surfactant, and the rinse liquor contains additionally a non-ionic surfactant. A very marked improvement according to the invention has been demonstrated under these conditions.

In a preferred embodiment of the process according to the invention the concentration of enzyme in the rinse liquor is in the range of 0.001 to 5, preferably 0.01 to 2 mg pure enzyme protein per liter of rinse liquor. At a concentration below 0.001 mg pure enzyme protein per liter of rinse liquor no significant enzyme effect can be observed, and at a concentration above 5 mg pure enzyme protein per liter of rinse liquor the enzyme cost will be unreasonably high.

Also, the invention comprises a rinsing agent for use in the process according to the invention, this rinsing agent being characterized by the fact that it is an enzyme preparation stabilized against microbial infection.

In a preferred embodiment of the rinsing agent according to the invention the rinsing agent also comprises a fabric softening agent and/or an antistatic agent. In this manner the enzymatic effect during the rinsing process is improved.

In a preferred embodiment of the rinsing agent according to the invention the rinsing agent is aqueous and it exhibits an enzyme activity between 0.01 and 50 mg pure enzyme protein per liter of rinsing agent, preferably between 0.5 and 10 mg pure enzyme protein per liter of rinsing agent, and the content of fabric softening and/or antistatic agent is at least 1% (by weight) (% as active material). It is intended that this rinsing agent is added to the rinse liquor, both providing the proper concentration of enzyme and the proper amount of fabric softening agent and/or antistatic agent.

In a preferred embodiment of the rinsing agent according to the invention the enzyme is cellulase, the softening agent and the antistatic agent is a cationic surfactant, and the rinsing agent contains additionally a non-ionic surfactant. In this embodiment a very marked improvement according to the invention has been demonstrated.

DETAILED DESCRIPTION OF THE INVENTION

Lipases

Lipases of plant or animal origin (e.g. pancreas lipase) can be used in the invention, but microbial lipases are preferred for reasons of economy. Lipases already known to be active in detergents can be used in the invention, but since the conditions in the rinsing step are favourable for most lipases, many other lipases can also be used.

Examples are lipases derived from the following microorganisms. The indicated patent publications are incorporated herein by reference:

Humicola, e.g. *H. insolens* (US 4,810,414)

Pseudomonas, e.g. *Ps. cepacia* or *Ps. fluorescens* (WO 89/04361).

Fusarium, e.g. *F. oxysporum* (EP 130,064).

Mucor (also called *Rhizomucor*), e.g. *M. miehei*.

Candida, e.g. *C. cylindracea* (also called *C. rugosa*) or *C. antarctica* (WO 88/02775).

The lipase activity unit, LU, is defined in AF 95/5, which is available on request from Novo Nordisk A/S, Novo allé, DK-2880 Bagsvaerd, Denmark.

Cellulases, proteases, and amylases

Similar considerations as indicated in the preceding paragraph about lipases are valid for the cellulases, proteases, and amylases. Cellulases may be

derived from *Humicola insolens*, proteases from *Bacillus licheniformis*, and amylases may be derived from *Bacillus subtilis*.

The cellulase activity unit, CEVU, is defined in AF 253/2-GB, which is available on request from Novo Nordisk A/S, Novo allé, DK-2880 Bagsvaerd, Denmark.

Fabric softening agents and/or antistatic agents

According to a preferred embodiment of the invention a fabric softening and/or antistatic agent effective for fabric after-treatment is used together with the enzyme in the process according to the invention. Such after-treatment agents are known in the art. One example is quaternary ammonium salts such as dialkyl (C_{16}-C_{18}) dimethyl ammonium chloride, alkyl (C_{32}-C_{36}) trimethyl ammonium chloride or alkyl (C_8-C_{18}) dimethyl benzyl ammonium chloride (ABDAC); and alkyl imidazolinium methyl sulphate. Another example is layered clay of the smectite type, such as montmorillonite or bentonite.

Textile treatment process

The invention uses an enzyme in the rinsing stage of a conventional treatment process for soiled or unsoiled fabrics comprising washing, rinsing and drying. The rinsing stage generally comprises 2-5 subsequent steps of rinsing with water, usually 3 or 4 steps. In a preferred embodiment the enzyme is added together with a fabric softening and/or antistatic agent (e.g. 0.1-0.5 g/l). The enzyme is preferably added in the second or a later rinsing step, and most preferably in the last rinsing step, where the detergent concentration is lowest (thus minimizing any incompatibility problems between detergent and enzyme) and where it can conveniently be added together with the fabric softening agent and/or antistatic agent, if such agent is used in the process according to the invention. Conventional rinsing conditions can be used, e.g. 10-30°C water, 1-20 minutes at near-neutral pH (e.g. 5-9) depending on the use of cationic or clay.

Rinsing agent

The rinsing agent according to the invention is stabilized against microbial infection, preferably by incorporation of a stabilizing agent known in the art. Examples are inorganic salts (such as NaCl), sugars (such as sucrose and glucose), polyols (such as glycerol, propylene glycol and sorbitol) and alcohols (such as ethanol and iso-propanol). These are generally effective in amounts above 10%, especially above 20%. Another example is organic acids (such as benzoic, sorbic, propionic, lactic and formic), which are generally effective in amounts of 0.01-2% at low pH (below 5). Other examples of stabilizing agents are antioxidants (such as sulphur dioxide), 1,2-benz-iso-thiazolin-3-one (BIT) and parabens. Some of these may also serve to improve enzyme stability.

Further additives may be incorporated to improve enzyme stability or physical stability of the rinsing agent. The choice depends on the type of enzyme(s). Examples are $CaCl_2$ (e.g. 0.1-0.5%), a protease inhibitor (such as borate and formate) or 0.5-5% of lower alcohol (such as ethanol or iso-propanol, preferably used together with a larger amount of a polyol).

The rinsing agent ordinarily is preferably essentially devoid of anionic and nonionic surfactant (total amount of these below 5%, most preferably below 2%).

The rinsing agent may be in solid (e.g. as non-dusting granules) or liquid form (e.g. aqueous). It may be formulated by incorporating enzyme into known softening/antistatic compositions. The content of the softening/antistatic agent is preferably 1-50%, especially 5-50% (% as active material).

EXAMPLES

The lipase used in Examples 1 - 4 is from *Humicola lanuginosa* produced according to US 4,810,414 or from *Pseudomonas cepacia* produced according to WO 89/04361. The cellulase used in Example 5 is from *Humicola insolens* produced according to US 4,435,307.

EXAMPLE 1

Effect of different lipases under the rinse process

Washing and rinsing tests are made as follows:

Fabric:	Polyester/cotton (50/50) or cotton.
	Swatch size: 9x9 cm.
Soiling:	Lard/Sudan red (1000/0.75 w/w) is heated to 70°C, and 50 µl is spotted onto each swatch by a pipette. After application of stain, the swatches are heated in an oven for 30 minutes at 75°C.
Washing:	Terg-o-tometer (100 rpm).
	Commercial European detergent powder, 8 g/l in 18°dH water.
	pH not adjusted (approx. 10).
	30°C, 20 minutes.
	7 swatches per beaker containing 1 liter detergent solution.
Rinsing:	4-step rinse in Terg-o-tometer.
	1st-3rd rinse: 6 minutes in 1 l cold tap water.
	4th rinse: 10 minutes in 1 l tap water (22°C).
	Swatches squeezed by hand between each rinse.
	0 or 100 LU/l of lipase added in 4th rinse.
	0 or 0.20 g/l of Arquad 2HT-75 (dialkyl dimethyl ammonium chloride,
	75% active matter, Akzo-Chemicals) added in 4th rinse.
Drying:	Line-drying (16 hours) at room temperature.
Repeats:	Washing, rinsing and drying of swatches repeated 3 times.
Evaluation:	After the 3rd wash.
	Reflectance is measured at 460 nm.
	Content of fatty matter is determined by Soxhlet extraction with chloroform for 5 hours.

It appears from the following tables that addition of lipase during the last rinse improves the removal of fatty matter, in relation to both lipases, both with and without cationic.

Lipase	Arquad 2HT-75 (g/l)	Reflectance R	Reflectance ΔR	Fatty matter mg	Fatty matter Residual fat %
-		63.2	-	175	50.0
Humicola lanuginosa	0.00	86.8	23.6	65	18.6
Pseudomonas cepacia		70.7	7.5	118	33.7
-		73.3	-	138	39.4
Humicola lanuginosa	0.20	81.2	7.9	110	31.4
Pseudomonas cepacia		80.8	7.5	109	31.1

Effect of *Humicola lanuginosa* and of *Pseudomonas cepacia* lipase on lard/cotton swatches. The lipases were added initally under the 4th rinse step; no lipase was present during the washing process.

Lipase	Arquad 2HT-75 (g/l)	Reflectance R	Reflectance ΔR	Fatty matter mg	Fatty matter Residual fat %
-		57.4	-	188	53.7
Humicola lanuginosa	0.00	86.7	29.3	41	11.7
Pseudomonas cepacia		76.9	19.5	79	22.6
-		77.9	-	100	28.6
Humicola lanuginosa	0.20	84.6	6.6	69	19.7
Pseudomonas cepacia		83.1	5.2	67	19.1

Effect of *Humicola lanuginosa* and of *Pseudomonas cepacia* lipase on lard/polyester-cotton swatches. The lipases were added initially under the 4th rinse step; no lipase was present during the washing process.

EXAMPLE 2

Lipase performance when used in combination with different cationic surfactants

The performance evaluation was carried out as described in Example 1, the only difference being that other kinds of cationics were used in combination with the *Humicola lanuginosa* lipase and that the lipase dosage was varied between 0 and 3000 LU per litre.

The cationics used were:

1) Dodigen 1490 from Hoechst (dialkyl dimethyl ammonium chloride, 75% active matter)
2) Empigen FRC75/S from Albright & Wilson (alkyl imidazoline methosulfate, 75% active matter)

Cationic surfactant	Lipase dosage (LU/l)	Reflectance R	Reflectance ΔR	Fatty matter mg	Fatty matter Residual fat %
	0	67.6	-	174	49.7
	30	85.8	18.3	74	21.1
	100	86.9	19.3	66	18.9
	300	87.6	20.0	60	17.1
	1000	86.9	19.3	57	16.3
	3000	89.2	21.6	44	12.6
	0	77.5	-	168	48.0
Dodigen	30	83.2	5.7	139	39.7
1490	100	83.2	5.7	134	38.3
(0.2 g/l)	300	83.4	5.9	128	36.6
	1000	85.2	7.7	120	34.3
	3000	86.5	9.1	107	30.6
	0	72.8	-	141	40.3
Empigen	30	81.5	8.7	114	32.6
FRC75/S	100	82.8	10.0	107	30.6
(0.2 g/l)	300	82.8	10.0	97	27.7
	1000	82.6	9.8	95	27.1
	3000	82.3	9.5	92	26.3

EXAMPLE 3

Lipase performance - comparison with prior art

Performance evaluation was carried out as described in Example 1. In this example the use of *Humicola lanuginosa* lipase in the rinse liquor (this invention) was compared to the use of the same enzyme in the main wash liquor (prior art). Lipase dosage was 100 LU/l.

		Residual fat %
Prior art	Lipase used in main wash liquor	44.9
Invention	0.2 g/l Dodigen 1490	38.3
	0.2 g/l Empigen FRC 75/S	30.6
	without cationics	18.9

EXAMPLE 4

Lipase stability in rinse preparations (after treatment preparations)

Composition % w/w	1	2	3	4
Lipolase® 100 L	0.5	0.5	0.5	0.5
Dodigen 1490	8.0	8.0		
$CaCl_2$	0.1	0.1	0.1	
Propylene glycol		20.0	25.0	25.0
Deionized water	91.4	71.4	74.4	74.5
pH	6.0	6.0	7.0	7.0

The above mentioned lipase containing rinse preparations were stored at room temperature (20-22°C) for 240 days and the lipase activity was followed. The following results expressed as % relative activity were obtained:

Formulation no.	Days of storage 0	1	9	14	24	240
1	100	95	82	68	72	35
2	100	84	72	80	88	42
3	100	90	92	97	98	102
4	100	92	83	90	93	84

EXAMPLE 5

Effect of cellulase on color clarification

The textile swatches are exposed to the enzyme in a Terg-o-tometer. The treatment consists of a washing process under US conditions followed by a 4 repeated rinsings. The enzyme is introduced respectively in the main wash liquor (prior art) and in the rinse liquor corresponding to the last rinsing step (the invention).

Wash:

Liquid volume	: 800 ml
Agitation	: 100 movements/minute
Washing time	: 10 minutes
Washing	: 20°C
pH	: 7.0
Water hardness	: 9 dH
Textile	: 100% cotton
Amount of textile	: 2 swatches with dimensions 10x15 cm in 800 ml of liquor
Detergent	: 5 g/l commercial US detergent based on anionics

Rinsing steps:

Liquid volume	: 800 ml
Agitation	: 100 movements/minute
Rinsing time, step 1-3	: 6 minutes
Rinsing time, step 4	: 10 minutes
Rinsing temperature	: 20°C
pH	: 7.0
Water hardness	: 9 dH
Drying	: The swatches are dried at room temperature after the last step

Cellulase dosages:

0 - 500 CEVU/l. The cellulase is produced by means of *Humicola insolens* DSM 1800, and the CEVU cellulase activity unit is defined in AF 253/2-GB.

Rinse liquor compositions:

Cationic: ARQUAD 2HT-75, dialkyl dimethyl ammonium chloride, Akzo Chemie GmbH.

Nonionic: Berol 081, 100% AEO C_{16}-C_{18}, 30 mole EO, Berol Kemi AB, Sweden.

Composition	Cationic	nonionic	Enzyme introduction
1	0.2 g/l	none	the 4th rinsing step
2	none	0.01 g/l	the 4th rinsing step
3	0.2 g/l	0.01 g/l	the 4th rinsing step
4	none	none	in wash

Analysis method:

The surface of the swatches is analysed by measuring reflected light. White light is projected onto the surface and the reflection/remission is detected at 16 wavelengths by means of the "Elrepho 2000", Datacolor Schweiz apparatus. The results from these detections are processed into Hunter coordinates of which the L-coordinate represents the grey scale value (100: black, 0: white).

The effect of the cellulase is removal of damaged cellulose fibres causing a grey/worn look, and by measuring the grey scale value a quantitative expression for the enzyme treatment is obtained. The results are represented in relation to the L-coordinate of a surface not treated with cellulase:

Composition:	1	2	3	4
Delta L	0.62	0.72	1.58	0.41

The relative standard deviation of the above figures is 6%.

CLAIMS

1. Process for treatment of textiles by use of an enzyme, characterized by the fact that the enzyme is used in a rinse liquor for a rinsing step after washing.

2. Process according to Claim 1, characterized by the fact that the enzyme is used in the rinse liquor for the rinsing step after washing together with a fabric softening agent and/or an antistatic agent.

3. Process according to Claims 1 - 2, wherein the fabric softening agent and/or the antistatic agent is a cationic surfactant.

4. Process according to Claims 1 - 3, wherein the enzyme is used in the rinse liquor for a second or later rinsing step, preferably the last rinsing step.

5. Process according to Claims 1 - 4, wherein the enzyme is a lipase and/or a cellulase and/or a protease and/or an amylase.

6. Process according to Claims 1 - 5, wherein the enzyme is a lipase and/or a cellulase.

7. Process according to Claims 1 - 6, wherein the enzyme(s) is (are) of microbial origin.

8. Process according to Claims 2 - 7, characterized by the fact that the enzyme is cellulase, that the softening agent and the antistatic agent is a cationic surfactant, and that the rinse liquor additionally contains a non-ionic surfactant.

9. Process according to Claims 1 - 8, wherein the concentration of enzyme in the rinse liquor is in the range 0.001 to 5, preferably 0.01 to 2 mg pure enzyme protein per liter of rinse liquor.

10. Rinsing agent for use in the process according to Claims 1 - 9, characterized by the fact that the rinsing agent is an enzyme preparation stabilized against microbial infection.

11. Rinsing agent according to Claim 10, characterized by the fact that it also comprises a fabric softening agent and/or an antistatic agent.

12. Rinsing agent according to Claim 11, characterized by the fact that the rinsing agent is aqueous and that it exhibits an enzyme activity between 0.01 and 50 mg pure enzyme protein per liter of rinsing agent, preferably between 0.5 and 10 mg pure enzyme protein per liter of rinsing agent, and by a content of at least 1% (by weight) of fabric softening and/or antistatic agent (% as active material).

13. Rinsing agent according to Claim 11 - 12, characterizd by the fact that the enzyme is cellulase, that the softening agent and the antistatic agent is a cationic surfactant, and that the rinsing agent additionally contains a non-ionic surfactant.

AMENDED CLAIMS
[received by the International Bureau on 31 July 1991 (31.07.91);
original claims 1-13 replaced by amended claims 1-9 (2 pages)]

1. Process for treatment of textiles by use of a lipase, which is used in a rinse liquor for a rinsing step after washing, wherein the lipase is producible by means of *Humicola lanuginosa* or *Pseudomonas cepacia*.

2. Process according to Claim 1, wherein the lipase is Lipolase.

3. Process according to Claim 1 or 2, characterized by the fact that the lipase is used in the rinse liquor for the rinsing step after washing together with a cationic surfactant, which is Dodigen 1490 and/or Empigen FRC 75/5.

4. Process according to Claims 1 - 3, wherein the lipase is used in the rinse liquor for a second or later rinsing step, preferably the last rinsing step.

5. Process according to Claims 1 - 4, wherein the concentration of lipase in the rinse liquor is in the range 0.001 to 5, preferably 0.01 to 2 mg pure enzyme protein per liter of rinse liquor.

6. Rinsing agent for use in the process according to Claims 1 - 5, characterized by the fact that the rinsing agent is a lipase preparation stabilized against microbial infection.

7. Rinsing agent according to Claim 6, characterized by the fact that the rinsing agent is aqueous and that it exhibits a lipase activity between 0.01 and 50 mg pure enzyme protein per liter of rinsing agent, preferably between 0.5 and 10 mg pure enzyme protein per liter of rinsing agent.

8. Rinsing agent according to Claims 6 - 7, characterized by the fact that it also comprises the cationic surfactant.

9. Rinsing agent according to Claim 8, characterized by the fact that the cationic surfactant is present in an amount of at least 1% (by weight) of active material.

STATEMENT UNDER ARTICLE 19

The new set of claims has been limited to a process for treatment of textiles by use of lipase after washing, which is characterized by the fact that the lipase is selected from two identified lipases, which are specially well suited for this purpose.

The differences between the claims as filed and the claims as amended appear from the copy of the claims as filed (typewritten) with handwritten amendments, corresponding to the typewritten claims as amended forwarded herewith.

INTERNATIONAL SEARCH REPORT

International Application No PCT/DK 91/00057

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

IPC5: C 11 D; D 06 M

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
IPC5	C 11 D 3/386, D 06 M 16/00

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched[8]

SE,DK,FI,NO classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category *	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
X	GB, A, 1368599 (UNILEVER LIMITED) 2 October 1974, see the description, page 3, line 28-44 and the claims 1-14 --	1-13
X	EP, A2, 0220016 (NOVO INDUSTRI A/S) 29 April 1987, see the description, page 2, line 21-34; page 4, line 28-32; page 5, line 6-15 and the claims 1-16 --	1-13
A	EP, A1, 0276547 (THE UNIVERSITY OF READING) 3 August 1988, see the example and the claims 1-10 --	1-13

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* Special categories of cited documents: [10]

"A" document defining the general state of the art which is not considered to be of particular relevance

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"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.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
4th June 1991	1991-06-13
International Searching Authority	**Signature of Authorized Officer**
SWEDISH PATENT OFFICE	Dagmar Järvman Dagmar Järvman

Form PCT/ISA/210 (second sheet) (January 1985)

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.PCT/DK 91/00057

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 Swedish Patent Office EDP file on 91-04-30
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Patent document cited in search report	Publication date	Patent family member(s)		Publication date
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