

Trial decision

Invalidation No. 2014-800045

Tokyo, Japan

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The decision on the case of the patent invalidation trial between the above parties on Japanese Patent No. 4114820, entitled "Detergent Composition," dated September 16, 2014 came with a court decision of revocation of the trial decision (2014 (Gyo-Ke) 10235, rendition of decision on August 26, 2015) at the Intellectual Property High Court, the case was proceeded further and has resulted in the following trial decision:

#### Conclusion

The trial of the case was groundless.

The costs in connection with the trial shall be borne by the demandant.

#### Reason

##### No. 1 History of the procedures

###### 1 The Patent

The patent application regarding Japanese Patent No. 4114820 subjected to the invalidation trial of the case (hereinafter referred to as the "Patent") was filed on July 24, 1996 as Patent Application No. H8-194727 (Priority Date: December 11, 1995, Japanese Patent Application No. H7-321895) entitled "Detergent Composition," and the establishment of patent right with two claims was registered on April 25, 2008.

###### 2 History to the first trial decision

The trial for invalidation was demanded by HIROSE, Takami on July 13, 2009 (the case of the invalidation No. 2009-800152, hereinafter referred to as the "first trial"), a request for correction was made on October 5, 2009, and the trial decision was made on March 2, 2010; however, a court decision of revocation of the trial decision (2010 (Gyo-Ke) 10104, rendition of decision on November 10, 2010) was made at the Intellectual Property High Court, then the case was proceeded further, and the trial decision that "the correction shall be approved. The trial of the case was groundless" (hereinafter referred to as the "first trial decision") was made on January 31, 2011; the above decision became final and binding on March 14, 2011; and the binding was registered on April 20, 2011.

###### 3 History to the second trial decision

A trial for invalidation was demanded by AKZO NOBEL on August 25, 2011 (the case of the invalidation No. 2011-800147), the trial decision that "the trial of the case was

groundless." (hereinafter referred to as the "second trial decision") was made on April 12, 2012, and after a court decision of dismissal of the request (2012 (Gyo-Ke) 10177, rendition of decision on February 27, 2013) was made at the Intellectual Property High Court, the above decision became final and binding on March 13, 2013, and the binding was registered on April 4, 2013.

#### 4 History of the trial of the case

(1) The invalidation trial of the case was demanded by AKZO NOBEL (hereinafter referred to as the "demandant") on March 26, 2014 (the case of the invalidation No. 2014-800045, hereinafter referred to as the "trial of the case"), and then, a written reply was submitted by SHOWA DENKO K.K. (hereinafter referred to as the "demandee") on June 16, 2014.

(2) The trial decision that "the demand for trial of the case is dismissed" was made on September 16, 2014; however, a court decision of revocation of the trial decision (2014 (Gyo-Ke) 10235, rendition of decision on August 26, 2015) was made at the Intellectual Property High Court (hereinafter referred to as the "third court decision"), and the above decision has become final and binding.

(3) After the reception of a court decision of revocation of the trial decision, the case was proceeded further, a notification of trial examination was given to the demandant and the demandee on September 1, 2016, an oral proceedings statement brief was submitted by the demandant on October 14, 2016 while an oral proceedings statement brief was submitted by the demandant on October 18, 2016, and an oral proceeding was carried out on November 1, 2016.

#### No. 2 Description of the scope of claims in the specification of the case

The description of the scope of claims in the specification relating to the Patent after correction made on October 5, 2009 and admitted in the first trial (hereinafter referred to as the "specification of the case") is as follows;

"[Claim 1] A detergent composition comprising sodium hydroxide, aspartate diacetates and/or glutamate diacetates, and sodium glycolate, wherein the content of the sodium hydroxide is 0.1 to 40% by weight of the composition.

[Claim 2] The detergent composition according to claim 1, wherein the content of the sodium hydroxide is 5 to 30% by weight, the content of the aspartate diacetates and/or glutamate diacetates is 1 to 20% by weight, and the content of the sodium glycolate is 0.1 to 0.3 parts by weight with respect to 1 part by weight of the aspartate diacetates and/or glutamate diacetates."

(hereinafter the inventions relating to these claims are referred to as the "Invention 1" and the "Invention 2" so as to correspond to the claim numbers, and the patents corresponding to the inventions are referred to as "Patent 1" and "Patent 2.")

#### No. 3 Outline of allegations of the parties and means of proof submitted by the parties

1 The outline of the reasons for invalidation alleged by the demandant and the means of

proof submitted by the demandant

The objects of the demand for trial of the case that the demandant alleges is that the patent for the inventions according to Claim 1 to Claim 2 of Patent No. 4114820 shall be invalidated and the costs in connection with the trial shall be borne by the demandee. The reasons for invalidation the demandant alleges are summarized as follows;

(1) Reason for invalidation 1

Patent 1 and Patent 2 could have been provided easily by a person skilled in the art according to the inventions described in Evidence A No. 1, Evidence A No. 2, and Evidence A No. 3, or according to the inventions described in Evidence A No. 1, Evidence A No. 2, Evidence A No. 3, and Evidence A No. 4, and thus the grant of Patent 1 and Patent 2 cannot be obtained in accordance with the provisions of Article 29 (2) of the Patent Act, and therefore Patent 1 and Patent 2 should be invalidated under the provisions of Article 123(1)(ii) of the Patent Act.

Evidences that the demandant submitted at the time of demand for trial are as follows (hereinafter each is referred to "A1" or the like for short.);

Evidence A No. 1     UENO Keihei, "Introductory Chelate Chemistry", Nankodo Co., Ltd., September 15, 1988, the second revised edition

Evidence A No. 2     Japanese Unexamined Patent Application Publication No. H7-238299

Evidence A No. 3     Specification of United Kingdom patent application No. 1439518

Evidence A No. 4     Japanese Unexamined Patent Application Publication No. S50-3979

(2) Reason for invalidation 2

Since Patents 1 and 2 are not described in the detailed description of the invention, they are based on a patent application that does not meet the requirement stipulated in Article 36(6)(i) of the Patent Act, and thus fall under Article 123(1)(iv). Therefore, they should be invalidated.

2 Outline of the demandee's allegation

The demandee demands the decision, "the demand for trial of the case is groundless. The costs in connection with the trial shall be borne by the demandant," and alleges that the above-described reasons for invalidation that the demandant alleges are groundless.

#### No. 4 Content of main evidences

##### 1. "Introductory Chelate Chemistry" (A1)

A1, which was distributed on September 15, 1988, is obviously a publication distributed in Japan or abroad before the priority date for the Invention.

In addition, A1 has a description as follows;

(1a) Page 244, body text, lines 12 to 18

"A polymer phosphate had been added as a chelate agent to a household detergent before; however the phosphate concentration in living drainage had been increased with the increased use amount of the detergent, which was regarded to be a cause of serious proliferation of algae and red tide. For this reason, a metal ion scavenger such as zeolite started to be added instead nowadays because it does not cause environmental damage. The reason why a great amount of EDTA or NTA is not used in household soap or detergents is that EDTA or NTA is hardly decomposed by activated sludge in wastewater treatment plants."

(1b) Page 245, line 24 to page 246, line 4

"A large amount of chelate agent is consumed to wash glass bottles in addition to washing of metal. Glass bottles used as containers for soft drinks, beer, and alcoholic drinks are usually recovered to be used repeatedly while they are washed automatically and treated with a hot NaOH aqueous solution of 2% or more. However, calcium or magnesium that is leached out of the glass by hot alkali is gradually accumulated, and carbonate, phosphate, silicate, or hydroxide thereof is precipitated to prevent the washing effect. In this case, adding a chelate agent to the alkaline washing liquid can give a longer life to the liquid after washing. In this case, a polymer phosphate has a small effect in a high pH range, so that EDTA or NTA is mainly used regularly."

2 Japanese Unexamined Patent Application Publication No. H7-238299 (A2)

A2 was distributed on September 12, 1995, so that it is obvious that A2 is a publication distributed in Japan or abroad before the priority date of the patent application of the case.

A2 describes as follows:

(2a) "[0001]

[Field of the Invention] The present invention relates to a composition for washing a hard surface that has excellent detergency and is easily decomposed by microorganisms, and more particularly, to a potent composition for washing a hard surface that is suitable for washing glass bottles, glass products such as eating utensils, furniture and fixtures, and tanks and pipes in factories of various types, and metallic products and plastic products polluted with oils, and is easily decomposed by microorganisms.

[0002]

[Background of the Invention] In washing surfaces of places or washed objects with a shape difficult to wash in a normal working method, for example, bottles of various types such as beer bottles and milk bottles, food manufacturing equipment of various types such as tanks and pipes in food factories, and metallic products and plastic products such as heavily greasy ovens and grills, detergents with powerful detergency are desired, which are capable of washing them in a dipping method or by a simple method such as spraying a washing liquid on the surfaces of the washed objects. Conventionally, a detergent is used as a detergent of this kind, which is prepared by blending an ethylenediamine tetraacetic acid salt, sodium gluconate, or the like with an alkaline aqueous solution containing 1 to 5% by weight of sodium hydroxide with an added surface acting agent as necessary for the purpose of foam inhibition.

[0003]

However, while a detergent containing an ethylenediamine tetraacetic acid salt (hereinafter referred to as the 'EDTA' for short) is a detergent with very powerful detergency provided by powerful chelate generative capacity of EDTA, the EDTA and a metal complex compound thereof are difficult to decompose by the microorganisms, and thus discarding the washing liquid, especially the one after use, as it is has been posing a problem recently in terms of environmental protection. The detergent containing sodium gluconate has no problem about being discarded in terms of environmental protection because the sodium gluconate is easily decomposed by microorganisms; however, it is inferior in detergency and has defects of not being suitable for washing heavily dirty washed objects."

(2b) "[0005]

[Description of the Invention] In the above-mentioned background, the present inventors had devoted themselves to the research and development of a detergent that is easily

decomposed by microorganisms and is excellent in detergency, and as a result, they succeeded in the development of a detergent with powerful detergency by concomitant use of gluconate and a hydroxyethyl iminodiacetate salt. The present invention is made based on this knowledge. The present invention is to provide a composition for washing a hard surface that contains, as an active ingredient, alkali metal hydroxide, gluconate, and a hydroxyethyl iminodiacetate salt."

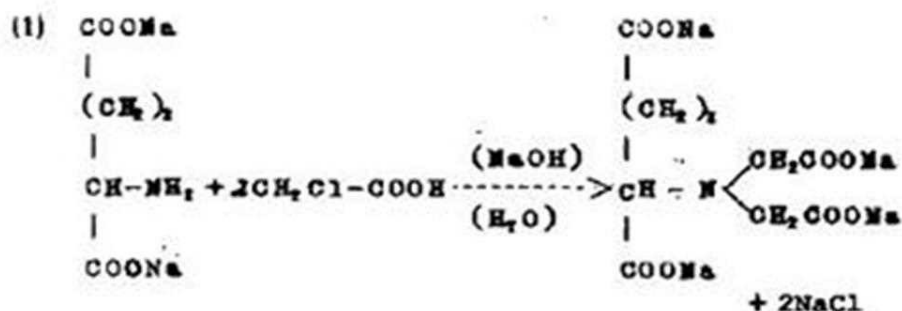
### 3 Specification of United Kingdom patent application No. 1439518 (A3)

A3 was distributed on June 16, 1976, so that it is obvious that A3 is a publication distributed in Japan or abroad before the priority date of the patent application of the case.

A3 describes as follows; however, since A3 is a piece of documentary evidence, the described matters therein are summarized based on the description of the complete translation of A3 (A3-2) submitted by the demandant;

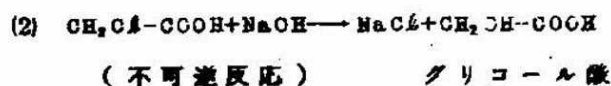
(3a) Page 2, line 19 to page 3, line 6 (Page 2, the second line from the bottom to page 4, line 6 in the complete translation)

"The reaction which takes place according to the invention is a replacement reaction which occurs in an aqueous medium and in the presence of an alkali and can be represented by the following scheme:



The two hydrogen atoms of the alpha amino group of the glutamic acid are replaced by two carboxymethyl groups originating from the monochloroacetic acid.

One of the main difficulties in obtaining the disubstituted derivative with a good yield originates in the hydrolysis of the monochloroacetic acid; this secondary reaction leads to the formation of sodium glycolate [see reaction (2) below]. To avoid this disadvantage, it is necessary to carry out the reaction in the presence of free monochloroacetic acid and only to add the alkali progressively, in order to favor reaction (1) above to the detriment of reaction (2) below. In effect, the relative speeds of these two reactions are influenced by the concentration of free OH groups.



(不可逆反応)                  (Irreversible reaction)

グリコール酸                  glycolic acid

It has been found possible to reduce this hydrolysis reaction (2) to a minimum provided that the reaction is conducted at a weakly alkaline pH value. Consequently, bearing in mind what has preceded, it is necessary to carry out the reaction at a pH which ranges from 8 to 10 and preferably is in the range 9 to 9.7. The yield obtained under these conditions is better than 75% of the theoretical value, based on the monochloroacetic acid being used, and is close to 100% with respect to the glutamate.

The adjustment of the pH is advantageously not effected with an alkaline earth compound, but preferably is effected with an alkali metal compound, particularly caustic soda in the form of a concentrated lye. The reactants can be reacted in aqueous medium. There may be used for this purpose softened water, demineralised water, or even distilled water.

The reaction is carried out at a temperature which ranges from 50 to 100 degrees C, preferably 70 to 100 degrees C and more preferably 80 to 90 degrees C, because the speed of the reaction (1) is more favorably influenced by raising the temperature than the speed of the reaction (2).

As starting material, there is advantageously used monosodium glutamate, which is cheap and which is available in abundant quantities commercially, although the acid itself and the disodium salt can be used.

It is necessary to work with an excess of monochloroacetic acid in order to



compensate for the possible hydrolysis (reaction 2). The reaction is therefore effected with from 2.4 to 2.7 moles, and most preferably with 2.7 moles, of monochloroacetic acid for each mole of glutamic acid (instead of a 2/1 molar ratio as required theoretically).

The replacement reaction involving the transfer of the carboxymethyl groups is advantageously effected by simultaneous pouring of a solution of monochloroacetic acid and alkali, e.g., caustic soda, into, for example, a disodium glutamate solution.

The end point of the reaction can be detected by any of the following means:

- 1) By measurement of the chloride ion formed.
- 2) By potentiometric determination of the complexing power.
- 3) By estimating -NH<sub>2</sub> or -NHR groups with ninhydrin."

(3b) Page 3, line 7 to line 45 (Page 4, line 7 to line 22 in the complete translation)

"It is possible to use the solution containing the reaction products directly as a metal ion sequestering agent composition. Alternatively, the solution may be spray dried to give impure N, N-dicarboxymethyl-2-amino-pentanedioic acid or a salt thereof. This impure product may then be purified by conventional techniques, preferably so that it is at least 95% pure. The solution, which has a content of dry material close to 50%, can be sprayed into a stream of hot air to give a white powder.

The production of a metal ion sequestering agent composition of the invention is advantageously carried out continuously, which may represent a considerable industrial advantage, which is shown by a reduction of the investment cost, and a reduction in the sale price of the metal ion sequestering agent composition, because of productivity on a relatively large scale.

The powder resulting from the spray drying operation can be purified in the following two ways:

- (1) Acidification to pH 3 by concentrated HCl, then spray drying, extraction of the dry product with acetone, and evaporation of the extract to dryness. The oil obtained is treated with methanol, and this crystallizes the final product with a purity, according to

measurements of sequestering power, of about 95%.

(2) Adsorption on a strongly acid cationic resin, then elution with caustic soda, followed by concentration. The oil obtained is treated with methanol, and this crystallizes the final product with a purity of 96 to 98%, according to measurements of its sequestering power."

(3c) Page 3, line 46 to line 81 (Page 4, line 23 to page 5, line 5 in the complete translation)

The compositions of the invention possess remarkable sequestering properties, not only with respect to alkaline agents, but also with respect to heavy metallic ions, these properties being much better than those which have so far been obtained. Consequently, undeniable industrial advantages and applications arise in the following fields:

- separation of metals on ion exchange columns,
- treatment of metals and the degreasing of metallic surfaces,
- decontamination of radioactive surfaces,
- treatment of textiles,
- formulation of detergents, lyes, and products used in the washing of crockery,
- applications in connection with perfumes and cosmetics,
- and applications in the milk industry.

The compositions according to the invention completely fulfil their sequestering function. They form soluble complexes of cations (Ca, Mg, Li, Fe, etc.) in the presence of chemical agents which would normally produce precipitates in aqueous solution, particularly in buffered alkali media.

They can also be used for masking or modifying the chemical activity of cations in reactions other than precipitation reactions. The compositions, when added to a solution of sodium soap in hard water, more particularly form a complex of the calcium, which would otherwise decrease the detergent action. Thus, as well as the action of inactivating cations, the metal ion sequestering agent compositions potentialize the detergent action of the soap."

(3d) Page 3, line 89 to line 126 (Page 5, line 9 to line 27 in the complete translation)

Actually, most detergents in liquid or powder form as marketed contain a metal ion sequestering agent which permits their use in water containing lime (i.e., hard water) without causing the precipitation of calcium soaps. Nevertheless, the metal ion sequestering agents used at present have appreciable disadvantages as regards the problems of pollution or undesirable ecological effects.

Thus, the metal ion sequestering agents most frequently employed, tripolyphosphate (abbreviated as TPP), contain phosphorus in a considerable quantity, and the discharge of washing waters into rivers or lakes has largely contributed to their eutrophy. By contrast, the compositions of the present invention permit the preparation of detergent media which are free from or contain reduced quantities of phosphorus and, as a consequence, can avoid this problem.

Other organic compounds have been proposed for replacing the "TPP" in detergents, particularly tertiary amine derivatives, such as the sodium salts of ethylenediamine tetraacetic acid (EDTA) or those of nitrilotriacetic acid (NTA), or even those of diethylene triamine pentaacetic acid.

The sequestrating power of these compounds is similar (equivalent of complexed cation/mole of metal ion sequestering agent). On the other hand, these compounds are biodegradable only with difficulty, and often, during their decomposition, give products which are toxic to the flora and fauna of waters. This behavior thus limits their use or the quantity which can be introduced into the cleansing circuits of urban waters without posing danger to the bacterial flora."

(3e) Page 3, line 127 to page 4, line 4 (Page 5, line 28 to the seventh line from the bottom in the complete translation)

"In addition, it has been verified that the compositions of the invention maintain all their properties in a broad pH zone and particularly in neutral or alkaline media: more especially, their sequestrating action is at a maximum between pH 8 and 11, which

corresponds to the level of alkalinity of the usual detergent media."

(3f) Page 4, line 27 to line 31 (Page 6, line 5 to line 7 in the complete translation)

"A metal ion sequestering agent composition of the invention, for use in a detergent composition, preferably contains at least 40% of a sodium salt of N, N-dicarboxymethyl-2-amino-pentanedioic acid."

(3g) Page 4, line 66 to line 79 (Page 6, line 22 to line 26 in the complete translation)

"It has also been noted that it is possible to sequester heavy metals while at the same time limiting the degradation of cellulose caused by successive washing operations carried out in oxidizing medium, by using detergent compositions in which about 5% of the detergent composition is the metal ion sequestering agent 'OS<sub>1</sub>', (the dried solids obtainable from the product 'OS<sub>1</sub>L' prepared as described hereinafter in Example 2), associated with magnesium silicate, the latter constituting about 1% of the detergent composition."

(3h) Page 4, line 91 to line 98 (Page 6, line 32 to the second line from the bottom in the complete translation)

"All the detergent compositions which contain N, N-dicarboxymethylated-2-aminopentanedioic acid or its salts as a metal ion sequestering agent, are characterized by a reduction in content or by the absence of phosphate, and by their biodegradability and their nontoxicity with respect to the flora and fauna of lakes and rivers."

(3i) Page 4, line 102 to line 119 (Page 7, line 1 to line 11 in the complete translation)

"For all the tests carried out, there is used a composition resulting from the reaction of monochloroacetic acid with the sodium salt of glutamic acid in an aqueous medium and in the presence of an alkali, and under the conditions as described above.

The composition which is obtained and which is called OS<sub>1</sub> in the tables contains:

N, N-dicarboxymethyl-2-amino-pentanedioic acid,

as sodium salt ... 60% by wt.

Sodium glycolate ... 12% by wt.

Salt ... balance to 100% by wt.

It is in the form of a white powder of apparent density 0.56.

The biodegradable metal ion sequestering agent composition is non-toxic, and does not cause any injurious irritation to the eyes or skin."

(3j) Page 5, line 73 to page 6, line 19 (Page 8, line 15 to page 9, line 14 in the complete translation)

## "EXAMPLE 2

68 kg (364 moles) of monosodium glutamate and 74 liters of water were placed in a jacketed tank. By means of the jacket, the temperature was brought to 50 degrees C, and then the pH was adjusted to 9.12 by adding 4 liters of 50.2% caustic soda solution. The simultaneous addition of solutions of monochloroacetic acid and 50.2% caustic soda solution was then started, while respecting the following two parameters: pH value between 9.2 and 9.5 and temperature between 70 and 75 degrees C. After operating for 14 hours, with introduction of 78 liters of monochloroacetic acid solution and 93 liters of caustic soda solution, the solution which was obtained was left in the tank while being stirred for 30 minutes and then a measurement was carried out. The measurement revealed the presence of 16% of N-carboxymethyl-L or DL-glutamic acid; i.e. 58 moles. A solution of 5 kg of monochloroacetic acid in 4 liters of water was prepared, and this solution was introduced

into the reaction mixture at 55 to 60 degrees C, and simultaneously 4 liters of 50.2% caustic soda solution were added to maintain the pH value. The solution obtained was left while being stirred for 2 1/2 hours at 60 degrees C, and then it was removed for subsequent concentration.

In this operation, the following quantities of initial materials were used: 68 kg (364 moles) of monosodium glutamate, 90 kg (953 moles) of monochloroacetic acid, 97 l + 4 l of caustic soda solution, 154 kg of 50.2% caustic soda; i.e. 77.4 kg of pure sodium hydroxide (1930 moles), water including that provided by the monosodium glutamate and monochloroacetic acid, 175.6 liters.

Concentration was carried out under vacuum and the concentrate was centrifuged, and there was obtained 204 kg of a liquid composition in accordance with the invention which had a concentration of 46.2% by weight of the trisodium salt of N, N-dicarboxymethyl-2-amino-pentanedioic acid.

The liquid composition in accordance with the invention thus obtained, called 'OS<sub>1</sub>L,' is a solution which conforms to the following standards:

glutamic acid:  $\leq 0.2\%$

total nitrogen:  $2\% \pm 0.2$

ammoniacal nitrogen:  $< 100$  ppm

dry extract:  $64 \pm 1$  g/l

density at 20 degrees C:  $1.475 \pm 0.005$

sodium %:  $15\% \pm 1$

iron:  $< 100$  ppm

chlorine:  $2.5 \pm 0.5$  g/l

pH (dilution to 10%):  $9.2 \pm 0.3$

viscosity at 20 degrees C: 8 poises  $\pm$  2 p

coloration (dilution to 10%):  $\leq$  iodine N

complexing power: 52 mg calcium/g

concentration %:  $\geq$  45% solution"

(3k) Page 6, line 20 to line 58 (Page 9, line 15 to the second line from the bottom in the complete translation)

### "EXAMPLE 3

The efficiency concerning the sequestering power of the preparations according to Examples 1 and 2 was determined by the hydrotimetric liquor method. It consisted in establishing the evolution curve of the hydrotimetric degree as a function of the added dose of metal ion sequestering agent. The softening power on water with the composition of the invention in the liquid or solid state was compared with those of different metal ion sequestering agents, viz. E.D.T.A. (ethylenediamine tetraacetic acid), N.T.A. (nitrilotriacetic acid), and T.P.P. (tripolyphosphate). It was determined by starting with a natural hard water of 25 degrees hardness (French standard) by measuring the hardness as a function of the dosage of metal ion sequestering agent in ammoniacal buffering medium (25 ml/l) at a pH of 10.

Figure 1 of the accompanying drawing illustrates the results obtained with the liquid composition. The dosage of metal ion sequestering agents in g per liter is plotted on the abscissae and the lime hardness in degrees Th (French standard) is plotted on the ordinates. Curve (1) corresponds to E.D.T.A. 4Na at 90%, curve (2) to N.T.A. 3Na, curve (3) to the composition of the invention in the liquid state, and curve (4) to T.P.P.

Figure 2 of the accompanying drawing illustrates the results obtained with the composition of the invention in solid form. Plotted on the axis of the abscissae is the dosage of metal ion sequestering agents in g per liters, while the lime hardness in degrees

Th is plotted on the ordinates. As in Figure 1, curve (1) corresponds to E.D.T.A. 4Na at 90%, curve (2) to N.T.A. 3Na, curve (3) to the composition in solid form, and curve (4) to T.P.P."

(31) Page 6, line 59 to page 7, line 11 (Page 9, the last line to page 11, the 11<sup>th</sup> line from the bottom in the complete translation)

#### "EXAMPLE 4

Washing tests were carried out in a TERG-O-TOMETER apparatus. The water used had a hardness expressed in degrees of French hardness of 22 degrees. The fabrics serving for the washing tests were linens which were soiled artificially and standardized. The standard soilings corresponding to the following references: EMPA 101, KREFELD, ACH, TNO cotton, and TNO polyester are effected together. After washing, the detergent effect was measured and there was deduced therefrom the percentage in improvement of whiteness provided by the treatment. For this purpose, there was used the formula:

$$\% \text{ improvement in whiteness} = \frac{DBF - DBS}{DBI - DBS} \times 100$$

in which:

DBF represents the degree of whiteness after washing,

DBS represents the degree of whiteness of the soiled fabric before washing,

DBI represents the degree of whiteness of the initial fabric before soiling.

The degrees of whiteness were measured by determining the quantity of reflected light; an ELREPHO reflectometer fitted with a green filter was used.

The values given in the tables of results each correspond to the average of several significant tests.



The washing operations were carried out at 60 degrees and 90 degrees C, which are the extreme working temperatures being used at present.

The detergent media have the following composition:

sodium carbonate: 3 g/liter

soap flakes: 0.25 and 0.50 g/liter

sodium tripolyphosphate (TPP): 0 and 3 g/liter

metal ion sequestering agent composition of the invention (indicated by "OS<sub>1</sub>"): 0, 1.5, and 2 g/liter

water of hardness 22 degrees: to make 1 liter

第 I 表

A-洗淨温度 60℃

	炭酸ナトリウム 3 g/l					炭酸ナトリウム 3 g/l				
	石鹼 0.5 g/l					石鹼 0.25 g/l				
TPP	0	3	0	0	0	0	3	0	0	0
“組成物OS <sub>1</sub> ”	0	0	1	1.5	2	0	0	1	1.5	2
白色度の改善	33	52	36	44	53	31	54	33	53	54

第 I 表 TABLE I

A-洗淨温度 60℃ A - Washing at 60 degrees C

炭酸ナトリウム sodium carbonate

石鹼 soap

‘組成物OS<sub>1</sub>’

‘Composition OS<sub>1</sub>’

白色度の改善

Improvement in whiteness

第 II 表									
B-洗淨温度 90℃									
炭酸ナトリウム					炭酸ナトリウム				
3 g/l					3 g/l				
石鹼 0.5 g/l					石鹼 0.25 g/l				
TPP	0	3	0	0	0	0	3	0	0
“組成物OS <sub>1</sub> ”	0	0	1	1.5	2	0	0	1	1.5
白色度の改善	56	58	51	58	61	54	60	50	58

第 I I 表 TABLE II

B-洗淨温度 90℃ B - Washing at 90 degrees C

炭酸ナトリウム sodium carbonate

石鹼 soap

‘組成物OS<sub>1</sub>’

‘Composition OS<sub>1</sub>’

白色度の改善

Improvement in whiteness

It was confirmed that in a simple detergent medium containing sodium carbonate and soap, washing in hard water was substantially improved by the addition of 1.5 g/liter of Composition OS<sub>1</sub> (the solids from the composition as prepared in Example 2). To obtain

the same results, it was necessary to add a quantity equivalent to twice the weight; i.e., 3 g/liter, of sodium tripolyphosphate."

(3m) Page 7, line 12 to page 8, line 27 (Page 11, the 10<sup>th</sup> line from the bottom to page 12, the 4<sup>th</sup> line from the bottom in the complete translation)

#### "EXAMPLE 5

Different and more elaborate detergent mixtures than in the preceding example were prepared: in particular, they all contained a soap, a non-ionic surface-active compound, a 'builder,' a bleaching agent of the peroxide type, and a filler. The formulae differ as regards the metal ion sequestering agent or sequestering mixture used. The washing tests were carried out at 60 degrees C with a TERGO-O-TOMETER apparatus in water with a hardness of 22 degrees, French standard. In each washing operation, there was used the equivalent of 8 g/liter of basic lye; i.e., 6 g/liter of products intended for the detergent effect and 2 g/liter of sodium peroxoborate.

The fabrics used were standard pre-soiled fabrics, such as EMPA 101, KREFELD, TNO cotton, TNO polyester, and ACH. After washing, the improvement in whiteness was measured by the formula given in the preceding example.

The tested formulae have the following compositions, expressed in g/liter of solution.

第 III 表

	1	2	3	4	5
珪酸ナトリウム 50/52° Be	1	1	1	1	1
コブラ石鹼	0.5	0.5	0.5	0.5	0.5
非イオン界面活性剤 (エトキシ化アルコール)	0.5	0.5	0.5	0.5	0.5
CMC (カルボキシメチル セルロース)	0.04	0.04	0.12	0.12	0.12
ペルオクソホウ酸ナトリウム	2	2	2	2	2
T P P (トリポリホスフェート)	3.2	—	—	0.8	1.6
金属イオン封鎖 組成物 “OS <sub>1</sub> ”	—	1.6	1.6	0.6	0.8
充填剤 全体を右の値とする残部	8	8	8	8	8
下記の試験結果が得られた					
処方：	1	2	3	4	5
白色度の改善、%：	70.5	69	72	71.5	71

第 I I I 表 TABLE III

珪酸ナトリウム 50/52° Be sodium silicate 50/52 degrees Be

コブラ石鹼 copra soap

非イオン界面活性剤 (エトキシ化アルコール) non-ionic surface active agent  
(ethoxylated alcohol)

CMC (カルボキシメチルセルロース) CMC (carboxymethyl cellulose)

ペルオクソホウ酸ナトリウム sodium peroxoborate

T P P (トリポリホスフェート) TPP (tripolyphosphate)

金属イオン封鎖組成物 'OS<sub>1</sub>' metal ion sequestering agent composition 'OS1'

充填剤 全体を右の値とする残部 filler - to make up to

下記の試験結果が得られた The following results were obtained:

処方： formula:

白色度の改善、%： % improvement in whiteness:

It can be seen that, provided the quantity of CMC is slightly increased, up to 0.10 to 0.12 g/liter of solution, there can be obtained the same improvement in the whiteness of the specimens of soiled fabrics with 1.6 g/liter of the composition OS<sub>1</sub> as that obtained with a solution containing 40% by weight of tripolyphosphate.

Thus, it is possible to obtain just as good a washing by using a formula which does not contain tripolyphosphate and in which the metal ion sequestering agent is formed by the composition based on the sodium salt of N, N-dicarboxymethyl-2-amino-pentanedioic acid. In this case, it is necessary to use a weight equal to half that of tripolyphosphate necessary to achieve the same cleaning effect.

The values given in the foregoing table also show that the two metal ion sequestering agents TPP and composition OS<sub>1</sub> can be used in admixture without losing their respective qualities. Thus, it is possible to reduce the usual content of tripolyphosphate by half or three quarters in detergents by replacing this metal ion sequestering agent by half the weight of the composition OS<sub>1</sub>."

(3n) Page 8, line 28 to page 9, line 10 (Page 12, the third line from the bottom to page 14, line 4 in the complete translation)

#### "EXAMPLE 6

The purpose of this example is to show the sequestrating properties of the composition OS<sub>1</sub> obtained by drying the solution as prepared by Example 2 with respect to heavy metals in alkaline medium.

It is known that the presence of certain metals, such as iron and copper, catalyzes the decomposition of per salts and as a result causes the chemical degradation of cellulose. For avoiding this disadvantage, a metal ion sequestering agent is generally used in the

detergent medium: the formulae which are most often employed contain a magnesium silicate mixed with the sodium salt of organic acids of the NTA or EDTA type, which are considered toxic for the fauna of rivers.

These tests were carried out by successive washing operations on fabrics consisting of bleached cotton (EMPA) or unbleached cotton (cretonne) in a TERG-O-TOMETER apparatus at 90 degrees C. The measurement of the degradation of the cellulose is carried out in accordance with the AFNOR standard No. 12-005.

The basic detergent which is used is a commercial detergent free of peroxoborate compound, to which are added 2 g of sodium peroxoborate tetrahydrate for each portion of 6 g of solution.

For the tests, there are in addition used mixtures containing 1% (in relation to the complete detergent including the peroxoborate) of magnesium silicate, and either EDTA or the composition OS<sub>1</sub>. Finally, at the start of the operation, catalytic quantities of heavy metals are added to the washing bath; i.e.:

copper as Cu<sup>++</sup>: 0.5 ppm

iron as Fe<sup>+++</sup>: 1.5 ppm

The results are given in the following Table IV.

第 Ⅴ 表		
安定性組成物	EMPA綿布 (洗浄前の当初のDP : 1600)	漂白CRETONNE (洗浄前の当初のDP : 2500)
	5回洗浄後	5回洗浄後
金属イオン封鎖剤無添加	980	1085
慣用品 (ケイ酸マグネシウム1% (EDTA: 0.2%))	1170	1290
本発明の組成物		
ケイ酸マグネシウム 1%		
+OS <sub>1</sub> 5%	1230	1433
+OS <sub>1</sub> 10%	1280	1453
+OS <sub>1</sub> 15%	1280	1453

第 I V 表 TABLE IV

安定性組成物 Stabilization formula

EMPA綿布 EMPA FABRIC

(洗浄前の当初のDP : 1600) (initial DP before washing: 1600)

5回洗浄後 After 5 washings

漂白 BLEACHED CRETONNE

(洗浄前の当初のDP : 2500) (initial DP before washing: 2500)

金属イオン封鎖剤無添加 Nil

慣用品 Conventional

本発明の組成物 Invention

ケイ酸マグネシウム Mg-silicate

DP = degree of polymerization

EDTA= sodium salt of ethylenediamine tetraacetic acid

+OS<sub>1</sub> = composition 'OS<sub>1</sub>'

It is found that 5% of the composition OS<sub>1</sub> permits protection of the cellulose to a higher degree than that which is provided by the conventional formulae containing derivatives of the EDTA or NTA type, and this, in addition to the lesser degree of wear which results, provides the advantage of producing waste washing waters which are free from products harmful to the environment."

(3o) Page 9, line 12 to page 10, line 46 (Page 14, line 6 to page 15, line 8 in the complete translation)

"1. A non-toxic and non-polluting, biodegradable metal ion sequestering agent composition which contains N, N-dicarboxymethyl-2-amino-pentanedioic acid, or a salt thereof, obtained by the simultaneous introduction of a solution of monochloroacetic acid and a solution of an alkali into an aqueous solution of glutamic acid or of the mono- or disodium salt of glutamic acid, wherein (a) said alkali is used in an amount such that the pH of the reaction medium is maintained at 8 to 10; (b) the reaction is carried out at a temperature in the range from 50 to 100 degrees C; and (c) 2.4 to 2.7 moles of monochloroacetic acid are used per mole of glutamic acid.

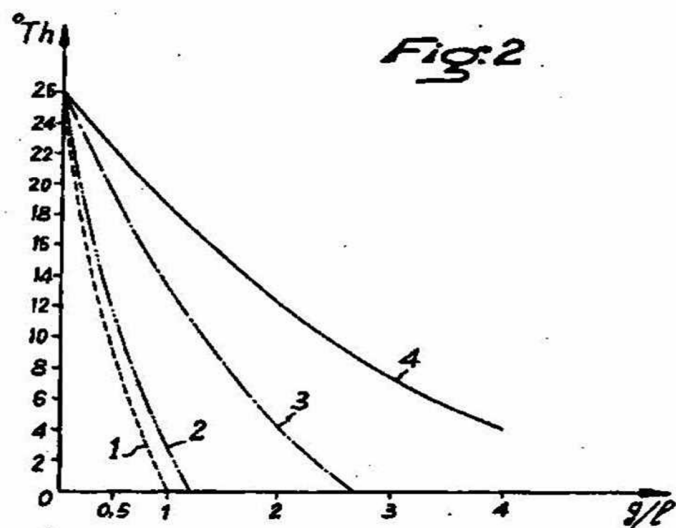
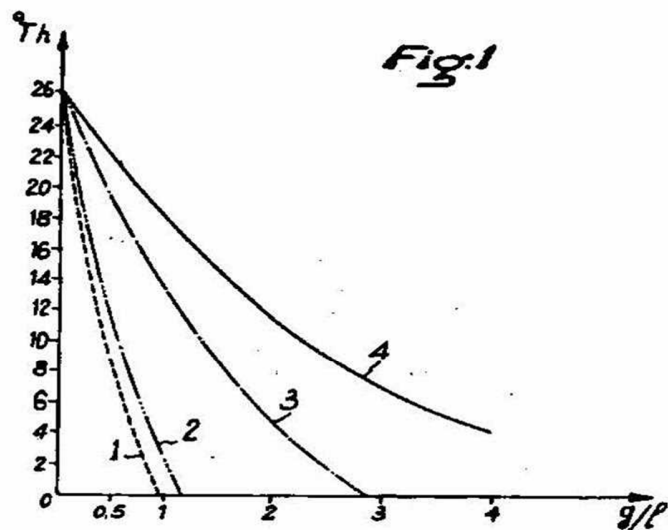
... (Omitted) ...

14. A method of preparing N, N-dicarboxymethyl-2-amino-pentanedioic acid or a salt thereof, which comprises carrying out the reaction specified in claim 1 and spray drying the reaction products.

15. A method according to claim 14, wherein the crude product obtained is purified by conventional methods so as to obtain a purity higher than 95%."

(3p) FIGs. 1 and 2 (FIGs. 1 and 2 on Page 16 in the complete translation)





"

4 Japanese Unexamined Patent Application Publication No. S50-3979 (A4)

A4 was distributed on January 16, 1975, and thus it is obvious that A4 is a publication distributed in Japan or abroad before the application before the priority date of the patent application of the case.

In addition, A4 has a description as follows;

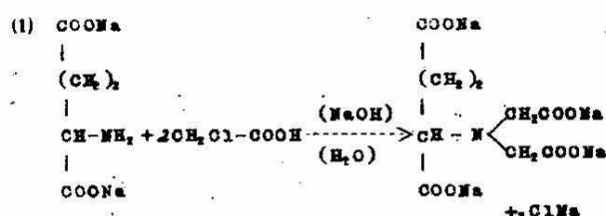
(4a) Page 2, the upper-right column, line 6 to line 15

"According to the invention, the biodegradable sequestering agent composition that contains a derivative of N, N-dicarboxymethyl amino acid is produced by the reaction of a monochloroacetic acid with a disodium salt of an amino dicarboxylic acid in an alkaline aqueous medium, thereby bonding carboxymethyl groups to nitrogen atoms of the amino groups of the amino dicarboxylic acid.

In carrying out the invention, it is particularly preferable to use a glutamic acid and an asparagine acid as an amino dicarboxylic acid."

(4b) Page 2, the bottom-left column, line 3 to page 3, the upper-right column, line 10

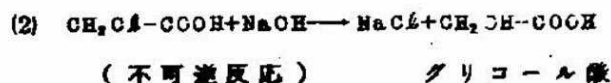
"The reaction according to the invention is a displacement reaction in the alkaline aqueous medium, and is represented by the following formula;



The two hydrogen atoms of an alpha amino group of the glutamic acid are replaced by two carboxymethyl groups originating from the monochloroacetic acid.

One of the essential difficulties in obtaining the derivative by replacing the amino groups of the amino dicarboxylic acid by the two carboxymethyl groups with a good yield originates in the hydrolysis of the monochloroacetic acid. That is, this secondary reaction leads to the formation of sodium glycolate [see reaction (2) below]. To avoid this disadvantage, it is necessary to carry out the replacement reaction in the presence of free monochloroacetic acid and only to add the alkali progressively, in order to carry out

reaction (1) while not developing reaction (2). In effect, the relative speeds of these two reactions are influenced by the concentration of free OH groups.



(不可逆反応)                  (Irreversible reaction)

グリコール酸                  glycolic acid

It has been found to be possible to reduce this hydrolysis reaction (2) to a minimum provided that the replacement reaction is conducted at a weakly alkaline pH value. Consequently, bearing the above-described secondary reaction in mind, it is advantageous to carry out the reaction at a pH which ranges from 8 to 10 and preferably is in the range 9 to 9.7 at the time of the reaction. The yield obtained under these conditions is better than 75% of the theoretical, based on the monochloroacetic acid being used, and is close to 100% with respect to the glutamate.

Different from previously known methods, it is not advantageous to use an alkaline earth compound for the adjustment of the pH, but it is advantageous to use an alkali metal compound, particularly caustic soda (soda) in the form of a concentrated lye.

The reactants can be reacted in aqueous medium. For this purpose there may be used softened water, demineralised water, or even distilled water.

The reaction is carried out at a temperature which is between 70 and 100 degrees C and preferably between 80 and 90 degrees C, because the speed of the reaction of the formula (1) is more increased by the rise in the temperature than is the speed of the reaction of the formula (2).

As starting material, there is advantageously used monosodium glutamate, which is cheap and which is available in abundant quantities commercially, although the acid itself and the disodium salt can be used.

It is advantageous to work with an excess of monochloroacetic acid in order to compensate for the amount in the possible hydrolysis (formula 2). The reaction is therefore effected with 2.4 to 2.7 moles, preferably 2.7 moles, of monochloroacetic acid for each mole glutamic acid (a molar ratio between the former and the latter is 1/2).

The replacement reaction of the carboxymethyl groups is advantageously effected by simultaneous pouring of a solution of monochloroacetic acid and caustic soda (soda) into a disodium glutamate solution.

The termination of the reaction can be determined by any of the following tests:

- (1) Determinate quantity of the chlorine (mineralised chlorine) bonded with metal
- (2) Potentiometric determination of the complexing power.
- (3) Determinate quantity of -NH<sub>2</sub> or -NHR groups with ninhydrin."

(4c) Page 3, the upper-right column, line 11 to Page 3, the bottom-left column, line 13

"It is possible to isolate a crude product that can be used directly in the form of liquid, or by spraying a solution obtained. The solution having a content of dry material close to 50%, can be sprayed into a stream of hot air to give a white powder.

The production of a metal ion sequestering agent composition of the invention is advantageously carried out continuously, which may represent a considerable industrial advantage, as shown by a reduction of the investment cost, and a reduction in the sale price of the metal ion sequestering agent composition, because of productivity on a relatively large scale.

The powder resulting from the spray operation can be purified in the following ways:

- (1) Acidification to pH 3 by concentrated HCl, then spraying, extraction of the dry product with acetone, and concentration of the extract to dryness. The oil obtained is treated with

methanol, and this crystallizes the targeted product with a purity, according to sequestering power, of about 95%.

(2) Adsorption on a strongly acid cationic resin, then elution with caustic soda (soda), followed by concentration.

The oil obtained is treated with ethanol, and this crystallizes the targeted product with a purity of 96 to 98%, according to its sequestering power."

(4d) Page 3, the bottom-left column, line 14 to the bottom-right column, line 20

"The sequestering agent compositions of the invention possess remarkable sequestering properties, not only with respect to alkaline-earth metal ions, but also with respect to heavy metallic ions, these properties being much better than those which have so far been obtained. Consequently, undeniable industrial advantages and applications arise widely in the following fields:

- separation of metals on ion exchange columns,
- treatment of hardware and the degreasing of metallic surfaces,
- decontamination of radioactive surfaces,
- treatment of textiles,
- formulation of detergents, lyes, and products used in the washing of crockery,
- applications to perfumes and cosmetics,
- and applications in the milk industry.

The sequestering agent compositions according to the invention completely fulfill their sequestering function. They form soluble complexes of cations (Ca, Mg, Li, Fe, etc.) in the presence of chemical agents which would normally produce precipitates in aqueous solution, particularly in buffered alkali media.

The sequestering agent compositions according to the invention can also be used for masking or modifying the chemical activity of cations in reactions other than precipitation reactions. The sequestering agent compositions, when added to a solution of sodium soap in hard water, more particularly form a complex of the calcium, which would otherwise decrease the detergent action. Thus, as well as the action of inactivating cations, the metal ion sequestering agent compositions potentialize the detergent action of the soap."

(4e) Page 4, the upper-left column, line 7 to the bottom-right column, line 14

"Actually, most of the detergents in liquid or powder form as marketed contain a metal ion sequestering agent, and the presence of the metal ion sequestering agent permits their use in water containing lime (called hard water), which generates no salt of calcium soap. Nevertheless, the metal ion sequestering agents used at present have appreciable disadvantages as regards the problems of pollution or undesirable ecology change.

Thus, the metal ion sequestering agents most frequently employed, the tripolyphosphate (abbreviated as TPP), contain phosphorus in a considerable quantity, and washing water discharged into rivers or lakes has largely contributed to their nutritional sources.

The sequestering agent compositions according to the invention permit the preparation of detergent media which are free from or contain reduced quantities of phosphorus and, as a consequence, can avoid this problem.

Other organic compounds have been proposed for replacing the 'TPP' in detergents, particularly tertiary amine derivatives, such as the sodium salts of ethylenediamine tetraacetic acid (EDTA) or those of nitrilotriacetic acid (NTA), or even those of diethylene triamine pentaacetic acid.

The sequestrating power of these compounds is similar to that of 'TPP' (equivalent of complexed cation/mole of metal ion sequestering agent). On the other hand, these compounds are biodegradable only with difficulty and often, during their decomposition

give products which are toxic to the flora and fauna of waters. These properties thus limit their use or the quantity which can be introduced into the cleansing circuits of urban waters without adversely affecting microorganisms."

(4f) Page 4, the upper-right column, line 15 to line 19

"In addition, it has been verified that the detergent compositions of this type maintain all their properties in a broad pH zone and particularly in neutral or alkaline media: more especially, their sequestering action is at a maximum between pH 8 and 11, which corresponds to the alkali level of the usual detergent media."

(4g) Page 4, the bottom-left column, line 18 to the bottom-right column, line 1

"A sequestering agent composition obtained in the invention preferably contains at least 40% of a sodium salt of an N, N-bis-carboxymethyl derivative."

(4h) Page 5, the upper-left column, line 4 to line 10

"It has also been noted that it is possible to sequester heavy metals while at the same time limiting the degradation of cellulose caused by successive washing operations carried out in an oxidizing medium, by using detergent compositions consisting of about 5% of metal ion sequestering agent composition 'OS<sub>1</sub>' and about 1% of magnesium silicate."

(4i) Page 5, the upper-left column, line 20 to the upper-right column, line 5

"All the detergents which contain N, N-bis-dicarboxymethylated derivative as a metal ion sequestering agent, are characterized by a reduction in content or by the absence of phosphate, and by their biodegradability and their nontoxicity with respect to the flora and fauna of lakes and rivers."

(4j) Page 5, the upper-right column, line 8 to the bottom-left column, line 1

"For all the tests carried out, there is used a sequestering agent composition resulting from the reaction of monochloroacetic acid with the sodium salt of glutamic acid in an alkali medium, and under the conditions as described above.

The sequestering agent composition which is obtained and which is called OS<sub>1</sub> in the tables contains:

N, N- bis-carboxymethyl glutamate, sodium salt ... 60% by wt.

Sodium glycolate ... 12% by wt.

Salt ... balance to 100% by wt.

It is in the form of a white powder of apparent density 0.56. The biodegradable composition is non-toxic, and it does not cause any injurious irritation to the eyes or skin."

(4k) Page 6, the upper-right column, line 8 to the bottom-right column, line 15

#### "EXAMPLE 2

68 kg (364 moles) of monosodium glutamate and 74 liters of water were placed in a jacketed reactor. By means of the double jacket, the temperature was brought to 50 degrees C, and then the pH was adjusted to 9.12 by adding 4 liters of 50.2% caustic soda solution. The simultaneous addition of solutions of monochloroacetic acid and 50.2% caustic soda solution was then carried out, while respecting the following two parameters: pH value between 9.2 and 9.5 and temperature between 70 and 75 degrees C.

After operating for 14 hours, with introduction of 78 liters of monochloroacetic acid solution and 93 liters of caustic soda solution, the solution which was obtained was stirred for 30 minutes to be mixed, and then a measurement was carried out. The measurement



revealed the presence of N-carboxymethyl-L or DL-glutamic acid. A solution of 5 kg of monochloroacetic acid in 4 liters of water was prepared, and this solution was introduced into the reaction mixture at 55 to 60 degrees C, and simultaneously 4 liters of 50.2% caustic soda solution were added to maintain the pH value. The solution obtained was stirred for 2 1/2 hours at 60 degrees C, and then it was removed for subsequent concentration.

In this operation, the following quantities of initial materials were used: 68 kg (364 moles) of monosodium glutamate, 90 kg (953 moles) of monochloroacetic acid, 97 l + 4 l of caustic soda solution, 154 kg of 50.2% caustic soda; i.e. 77.4 kg of pure sodium hydroxide (1930 moles), water including that provided by the monosodium glutamate and monochloroacetic acid, 175.6 liters.

Concentration was carried out under vacuum and the concentrate was centrifuged, and there was obtained 204 kg of a liquid sequestering agent composition in accordance with the invention which had a concentration of 46.2% by weight of the trisodium salt of N, N-bis(carboxymethyl) glutamic acid.

The liquid composition in accordance with the invention thus obtained, called 'O-S<sub>1</sub>L,' is a solution which conforms to the following standards:

Unreacted glutamic acid:  $\leq 0.2\%$

total nitrogen:  $2\% \pm 0.2$

ammoniacal nitrogen:  $< 100$  ppm

dry extract:  $64 \pm 1$

density at 20 degrees C:  $1.475 \pm 0.005$

sodium %:  $15\% \pm 1$

iron:  $< 100$  ppm

chlorine:  $2.5 \pm 0.5$

pH (dilution to 10%):  $9.2 \pm 0.3$

viscosity at 20 degrees C: 8 poises  $\pm 2$  p

coloration (dilution to 10%):  $\leq$  iodine N

complexing power: 52 mg calcium/g

concentration 'OS<sub>1</sub> (trisodium)':  $\geq 45\%$  solution"

(41) Page 6, the bottom-right column, line 16 to page 7, the upper-right column, line 9

### "EXAMPLE 3

The efficiency concerning the sequestrating power of the sequestering agent compositions obtained in Examples 1 and 2 was determined by the hydrotimetric liquor method. In this method, the hydrotimetric degree is determined by being represented by a curve as a function of the added dose of metal ion sequestering agent. The softening power on water with the sequestering agent composition of the invention in the liquid or solid state was compared with those of different metal ion sequestering agents, viz. E.D.T.A. (ethylenediamine tetraacetic acid), N.T.A. (nitrilotriacetic acid), and T.P.P. (tripolyphosphate). The softening power was determined by starting with a natural hard water of 25 degrees hardness (French standard) by representing the hardness as a function of the dosage of metal ion sequestering agent in ammoniacal buffering medium (25 ml/l) at a pH of 10.

Figure 1 of the accompanying drawing illustrates the measurement results of softening power obtained with the liquid sequestering agent composition of the invention and softening power obtained with other sequestering agents. The dosage of metal ion sequestering agents in g per liter is plotted on the abscissae and the lime hardness in degrees (French standard) is plotted on the ordinates. Curve (1) represents the measurement result of E.D.T.A. 4Na at 90%, curve (2) represents the measurement result of N.T.A. 3Na, curve (3) represents the measurement result of the sequestering agent

composition of the invention in the liquid state, and curve (4) represents the measurement result of T.P.P.

Figure 2 of the accompanying drawing illustrates the measurement results of softening power obtained with the sequestering agent composition of the invention in solid form and softening power obtained with other sequestering agents. Plotted on the axis of the abscissae is the dosage of metal ion sequestering agents in g per liters, while the lime hardness in degrees is plotted on the ordinates. As in Figure 1, curve (1) represents the measurement result of E.D.T.A. 4Na at 90%, curve (2) represents the measurement result of N.T.A. 3Na, curve (3) represents the measurement result of the sequestering agent composition in solid form, and curve (4) represents the measurement result of T.P.P."

(4m) Page 7, the upper-right column, line 10 to page 8, the upper-left column, line 7

#### "EXAMPLE 4

Washing tests were carried out in a TERG-O-TOMETER apparatus. The water used had a hardness expressed in degrees of French hardness of 22 degrees. The fabrics serving for the washing tests were linens which were soiled artificially and standardized. The standard soilings; that is, EMPA 101, KREFELD, ACH, TNO cotton, and TNO polyester, were tested in parallel. After washing, the washing effect was measured and there was deduced therefrom the percentage of the increasing rate of the whiteness provided by the washing treatment. The increasing rate of the whiteness was found by the following formula:

$$\% \text{ increasing rate of whiteness} = \frac{DBF - DBS}{DBI - DBS} \times 100$$

in which:

DBF represents the degree of whiteness after washing,

DBS represents the degree of whiteness of the soiled fabric before washing,

DBI represents the degree of whiteness of the initial fabric before soiling.

The degrees of whiteness were measured by determining the quantity of reflected light; an ELREPHO reflectometer fitted with a green filter was used.

The values given in the tables of test results each correspond to the average of several test results.

The washing operations were carried out at 60 degrees and 90 degrees C, which are the most extreme working temperatures being used at present.

The detergent media have the following composition:

sodium carbonate ... 3 g/liter

soap flakes ... 0.25 and 0.50 g/liter

sodium tripolyphosphate (TPP) ... 0 and 3 g/liter

metal ion sequestering agent composition of the invention (indicated by 'OS<sub>I</sub>') ... 0, 1, 1.5 and 2 g/liter

water of hardness 22 degrees ... to make 1 liter

第 I 表

A-洗淨温度 60℃		炭酸ナトリウム				炭酸ナトリウム			
		39/1				39/1			
		石鹼		0.59/1		石鹼		0.259/1	
TPP		0	3	0	0	0	0	3	0
金属イオン封鎖剤組成物		0	0	1	1.5	2	0	0	1
白色度の増加率		33	52	34	44	53	31	54	33

第 I 表 TABLE I

A-洗淨温度 60℃ A - Washing at 60 degrees C

炭酸ナトリウム sodium carbonate

石鹼 soap

金属イオン封鎖剤組成物 sequestering agent composition

白色度の増加率 increasing rate of whiteness

# 第 I 表

B-洗浄温度 90 °C	炭酸ナトリウム	炭酸ナトリウム
	39 / 1	39 / 1
	石鹼 : 0.59 / 1	石鹼 : 0.259 / 1
TPP	0 : 3 : 0 : 0 : 0 :	0 : 3 : 0 : 0 : 0 :
金属イオン封鎖剤組成物 OS <sub>1</sub>	0 : 0 : 1 : 1.5 : 2 :	0 : 0 : 1 : 1.5 : 2 :
白色度の改善	54 : 58 : 51 : 58 : 61 :	54 : 60 : 50 : 58 : 60 :

第 I I 表 TABLE II

B-洗浄温度 90 °C B - Washing at 90 degrees C

炭酸ナトリウム sodium carbonate

石鹼 soap

金属イオン封鎖剤組成物 sequestering agent composition

白色度の改善 increasing rate of whiteness

It is obvious from the tables described above that in a simple detergent medium containing sodium carbonate and soap, washing in hard water was substantially improved by the addition of 1.5 g/liter of 'sequestering agent composition OS<sub>1</sub>.' To obtain the same effects, it is necessary to add a quantity equivalent to twice the weight; i.e. 3 g/liter, of sodium tripolyphosphate."

(4n) Page 8, the upper-left column, line 8 to page 9, the upper-left column, line 4

# "EXAMPLE 5

... (Omitted) ...

The detergent compositions used in the test have the following compositions. The amount used of each ingredient is expressed in g/liter of the detergent composition solution.

第 I I 表					
	1	2	3	4	5
珪酸ナトリウム 50/52 °Be	1	1	1	1	1
コブラ石鹼	0.5	0.5	0.5	0.5	0.5
非イオン界面活性剤 (エトキシ化アルコール)	0.5	0.5	0.5	0.5	0.5
CMC (カルボキシメチルセルロース)	0.04	0.04	0.12	0.12	0.12
ペルオクソホウ酸ナトリウム	2	2	2	2	2
TPP (トリポリホスフェート)	3.2	-	-	0.8	4.4
金属イオン封鎖剤 (EDTA)	-	1.6	1.6	0.4	0.8
充填剤	8	8	8	8	8
全体が右の値となる					
試験結果はつぎのとおりである。					
組成物	1	2	3	4	5
白色度の増加率	7.25	6.9	7.2	7.15	7.1

第 I I I 表 TABLE III

珪酸ナトリウム 50/52 °Be sodium silicate 50/52 degrees Be

コブラ石鹼 copra soap

非イオン界面活性剤 (エトキシ化アルコール) non-ionic surface active agent (ethoxylated alcohol)

CMC (カルボキシメチルセルロース) CMC (carboxymethyl cellulose)

ペルオクソホウ酸ナトリウム sodium peroxoborate

TPP (トリポリホスフェート) TPP (tripolyphosphate)

金属イオン封鎖組成物'OS<sub>1</sub>' metal ion sequestering agent composition 'OS<sub>1</sub>'

充填剤 filler

全体が右の値となる量 to make up to

試験結果はつぎのとおりである。 The following results were obtained.

組成物 compositions

白色度の増加率 increasing rate of whiteness

... (Omitted) ...

Thus, it is possible to obtain just as very good a washing by using the detergent composition which does not contain tripolyphosphate and in which the metal ion sequestering agent is a composition containing a sodium salt of N, N-bis-dicarboxymethyl glutamic acid. In this case, it is necessary to use a weight equal to half that of tripolyphosphate necessary to achieve the same bleaching.

The results given in TABLE III also show that the two metal ion sequestering agents TPP and 'composition OS<sub>1</sub>' can be used in admixture without losing their respective qualities. Thus, it is possible to reduce the usual content of tripolyphosphate by half or three quarters in detergent compositions by replacing the tripolyphosphate by half the weight of the sequestering agent composition of the invention."

(4o) Page 9, the upper-left column, line 5 to page 9, the bottom-right column, line 10

#### "EXAMPLE 6

The example shows the sequestrating properties of the 'composition OS<sub>1</sub>' with respect to heavy metals in alkaline medium.



... (Omitted) ...

The results are given in the following Table IV.

第 Ⅳ 表		
安定性組成物	EMPA綿布 (洗浄前の当初のDP : 1600)	漂白 CRETONNE (洗浄前の当初のDP : 2500)
	5回洗浄後	5回洗浄後
金属イオン封鎖剤無添加	980	1085
慣用品 (ケイ酸マグネシウム1% (EDTA: 0.2%))	1190	1290
本発明の組成物 ケイ酸マグネシウム 1%		
+0.8 5%	1230	1433
+0.8 10%	1280	1433
+0.8 15%	1280	1433

第 I V 表 TABLE IV

安定性組成物 Stabilisation formula

EMPA綿布 EMPA FABRIC

(洗浄前の当初のDP : 1600) (initial DP before washing: 1600)

5回洗浄後 After 5 washings

漂白 BLEACHED CRETONNE

金属イオン封鎖剤無添加 Nil

慣用品 Conventional

本発明の組成物 Invention

ケイ酸マグネシウム Mg-silicate

DP = degree of polymerization

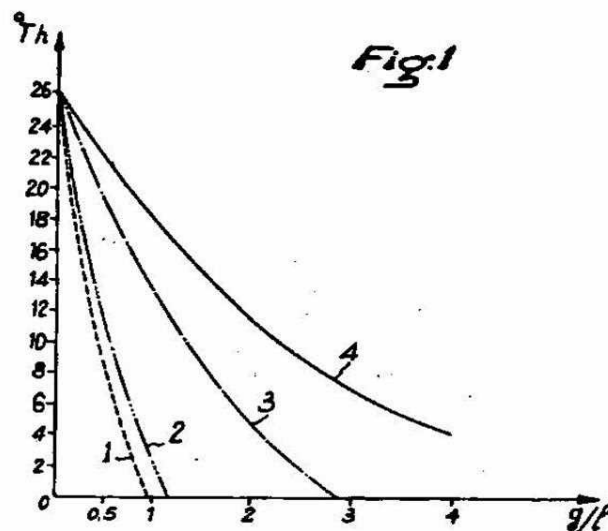
EDTA= sodium salt of ethylenediamine tetraacetic acid

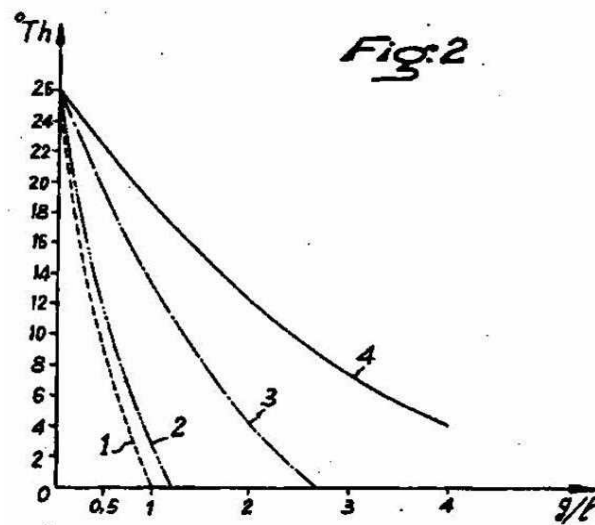
+OS = sequestering agent composition of the invention

The results given in TABLE IV show that 5% of the 'composition OS' permits protection of the cellulose to a higher degree than that which is provided by the conventional composition containing derivatives of the EDTA or NTA type, and this, in addition to the lesser degree of wear which results, provides the advantage of producing waste washing waters which are free from products harmful to rivers or lakes."

(4p) Page 11, FIGs. 1 and 2

"





"

No. 5 Judgment by the body

The body judges that it cannot be concluded that Patent 1 and Patent 2 must be invalidated for the reasons for invalidation 1 and 2 that the demandant alleges.

The reasons are as follows;

1 Regarding Reasons for invalidation 1

(1) The invention described in A1

According to (1b) of A1 described above, A1 can be said to describe use of a detergent that is prepared by adding a chelate agent such as EDTA to an alkaline detergent, in order to wash glass bottles. In addition, this alkaline detergent can be said to be a hot NaOH aqueous solution of 2% or more, and also can be said to be a "composition" if it contains a plurality of ingredients such as a chelate agent such as NaOH and EDTA.

Hence, A1 can be said to describe the invention indicated below as held in the "third

court decision" (at "No. 4," "3," "(2)").

"A detergent composition for washing glass bottles, the composition containing a hot sodium hydroxide aqueous solution of 2% or more and a chelate agent such as EDTA." (hereinafter referred to as the "A1 invention.")

## (2) Comparison / Judgment

### A Regarding Invention 1

#### (A) Comparison

When Invention 1 is compared to the A1 invention, they are in correspondence in being a "detergent composition," and also in containing "sodium hydroxide" in the detergent composition. In addition, Invention 1 and the A1 invention are in correspondence in a contained amount, as their contents are in the range of "2% (% by weight) or more."

In addition, according to the description at [0008] in the specification of the Patent, "The content of aminodicarboxylic acid diacetates as a detergent agent composition containing these compounds is 0.01 to 30% by weight, preferably 1 to 20% by weight. The aminodicarboxylic acid diacetates are useful as a component that exerts a washing effect of stably dissolving alkaline-earth metals in the dirt which mainly consists of an alkaline-earth metal salt (for example, calcium carbonate and magnesium carbonate) adhering to a hard surface during the processes in a variety of industries, in a washing liquid while serving as a chelate complex.", the "aspartate diacetates and/or glutamate diacetates" that are specific examples of the "aminodicarboxylic acid diacetates" of Patent 1 can be said to be a "chelate agent."

Hence, Invention 1 and the A1 invention are in correspondence in being a "detergent composition containing sodium hydroxide and a chelate agent, the content of the sodium hydroxide being 2% or more of the composition," while differing in the following feature;

(The different feature)

Invention 1 differs from the A1 invention in that the detergent composition of the Invention 1 "contains aspartate diacetates and/or glutamate diacetates, and sodium glycolate" while the detergent of the A1 composition contains EDTA, and the like.

(B) Judgment on the different feature

According to the descriptions at (3g) and (3i) described above, A3 describes a "biodegradable metal ion sequestering agent composition called OS<sub>1</sub> that contains 60% by weight of sodium salt of N, N-dicarboxymethyl-2-amino-pentanedioic acid (corresponding to the "glutamate diacetates" in Invention 1), 12% by weight of sodium glycolate, and the balance salt to 100% by weight, and is in the form of a white powder of apparent density 0.56 (hereinafter referred to as the 'OS<sub>1</sub>')", and according to the descriptions at (3l), (3m), and (3n) described above, A3 describes that the OS<sub>1</sub> is added to the "detergent medium" of (3l) described above, the "detergent mixture" of (3m) described above, and the "detergent" of (3n) described above (each of them corresponds to the "detergent composition").

According to the description at (3c), "sequestering" can be said to form a metallic ion and a complex (chelate).

In addition, according to the descriptions at (3f) and (3h), "N, N-dicarboxymethyl-2-amino-pentanedioic acid" and its "salts" in "OS<sub>1</sub>" are recognized as components being biodegradable and having sequestering properties.

According to the description at (3d), A3 further describes that EDTA, and the like are biodegradable only with difficulty.

Meanwhile, according to the descriptions at (2a) and (2b), A2 describes that since EDTA has a problem of having low biodegradability, a chelate agent (gluconate and a hydroxyethyl iminodiacetate salt) that has excellent biodegradability is used for washing the surfaces of glass bottles and metals, instead.

The A1 invention uses a chelate agent such as "EDTA" for the detergent composition for "glass bottles."

Since A2 describes that a chelate agent with excellent biodegradability is used for washing glass bottles instead of EDTA, which is of low biodegradability, a person skilled in the art might use the chelate agents described in A2 instead of EDTA in the A1 invention apart from effect caused by the alternate of the chelate agent.

However, A2 does not indicate that chelate agents other than the chelate agents described in A2 can be used in the A1 invention. The chelate agents described in A3 are not used for washing "glass bottles." Even though EDTA is recognized to be of low biodegradability according to A3, since the washing ability of a chelate agent varies depending on conditions such as objects to be washed and other components to be used therewith and it is not clear that the chelate agent described in A3, which is not for washing glass bottles, will work well when it is used in the detergent composition of A1 invention, which is for washing glass bottles, it cannot be said that a person skilled in the art is motivated to use the chelate agent described in A3 in the A1 invention in the same way as the chelate agent described in A2.

Thus, even considering the descriptions of A2 and A3, it cannot be said that the chelate agent described in A3 can be applied to the A1 invention.

In addition, if the chelate agent described in A3 could be used in the A1 invention, a person skilled in the art who understood the description of A3 might try to use "N, N-dicarboxymethyl-2-amino-pentanedioic acid" or its "salts", which are of biodegradability and sequestration (chelate) (see the description of (3d) above in A3) instead of the EDTA of the A1 invention, which is of low biodegradability.

However, regarding the above-mentioned OS<sub>1</sub>, according to the description of (3a), the "sodium glycolate" is a by-product produced in the "secondary reaction", which is disadvantage in synthesizing a glutamic acid disubstituted derivative (N, N-dicarboxymethyl-2-amino-pentanedioic acid), and thus synthesizing a glutamic acid disubstituted derivative will be conducted by reducing the "secondary reaction."

In addition, according to the descriptions of (3b) and (3o) above, the "crude product" that is "impure N, N-dicarboxymethyl-2-amino-pentanedioic acid or a salt thereof" containing "sodium glycolate" as a by-product or the like may be purified by conventional methods so as to obtain a "purity of at least 95%."

Further, as described above, only the "N, N-dicarboxymethyl-2-amino-pentanedioic acid" or "a salt thereof" is recognized as a component being biodegradable and having sequestering properties.

While the above-described "OS<sub>1</sub>" as a sequestering agent composition contains "sodium glycolate" as an impurity, the "sodium glycolate" in the "OS<sub>1</sub>" is recognized to be dispensable and a by-product that may be reduced during the synthesis and removed by purification.

Hence, even if "OS<sub>1</sub>" that is a sequestering agent composition contains the "sodium glycolate," it cannot be said that a person skilled in the art who understood the description of A3 would be motivated to add not only essential components for sequestering properties but also the "OS<sub>1</sub>" containing also the "sodium glycolate"; that is, but also the "sodium glycolate," which is merely recognized to be a by-product to be reduced during the synthesis and to be a component to be removed by purification, to the detergent composition of the A1 invention instead of adding EDTA or the like that is a chelate agent of the A1 invention.

In addition, the descriptions at (4b) to (4o), and (4p) of A4 almost coincide with the descriptions at (3a) to (3n), and (3p) of A3 except that A4 describes at (4a) above about not only using the "glutamic acid" that produces the "N, N-dicarboxymethyl-2-amino-pentanedioic acid" of A3 as an amino dicarboxylic acid but also using the "aspartate diacetates" of Invention 1 when producing a biodegradable sequestering agent composition containing an "N, N-dicarboxymethyl amino-acid derivative," so that even if A4 is taken into consideration, it cannot be said that a person skilled in the art would be motivated to add the "sodium glycolate" to the detergent composition of the A1 invention.

Therefore, it cannot be said that the chelate agent described in A3 can be applied to

the A1 invention in the first place, and even if the chelate agent described in A3 can be applied to the A1 invention, it cannot be said that the chelate agent containing the "sodium glycolate" such as "OS<sub>1</sub>" of A3 can be added to the detergent composition, and thus the above description (the different feature) could not be easily conceived by a person skilled in the art even taking A2 to A4 into consideration, and Invention 1 cannot be said to have been easily made according to the invention described in A1 and the descriptions of A2 and A4 distributed before the priority date of the patent application of the case.

(C) The demandant's allegation regarding the "sodium glycolate"

The demandant alleges, on page 3, lines 8 to 11 in the oral proceedings statement brief dated October 14, 2016, that "it is easy to apply OS<sub>1</sub> to the A1 invention, and as a result, the sodium glycolate contained in OS<sub>1</sub> is also combined with 2% or more of sodium hydroxide with a salt of N, N-dicarboxymethyl-2-amino-pentanedioic acid."

However, given the premise of Invention 1, while "OS<sub>1</sub>" may be used as it is instead of EDTA or the like of the A1 invention, applying the chelate agent described in A3 to the A1 invention could not be easily conceived by a person skilled in the art who understood only with the descriptions of A1 to A4, as is described above in "(B)."

In addition, even if the chelate agent described in A3 can be applied to the A1 invention, making the chelate agent not contain "sodium glycolate," which is merely recognized to be not an indispensable component having sequestering properties but a by-product to be reduced during the synthesis and a component to be removed by purification in "OS<sub>1</sub>" of A3 as described above in "(B)," and increasing the content of the "salt of N, N-dicarboxymethyl-2-amino-pentanedioic acid" that is an indispensable component for sequestering properties in "OS<sub>1</sub>," and using only thus-made chelate agent instead of EDTA or the like of the A1 invention might be conceived by a person skilled in the art; however, it cannot be said that "OS<sub>1</sub>" containing the above-described "sodium glycolate" can be used "as it is" instead of EDTA or the like of the A1 invention.

Even if specific examples of using "OS<sub>1</sub>" as it is in the detergent composition are described in A3 and A4, "OS<sub>1</sub>" has optimum conditions for exerting sequestering power in



the detergent composition such as pH conditions according to the above descriptions at (3e) of A3 and (4f) of A4, so that it cannot be said that "OS<sub>1</sub>" can be applied to the detergent composition of A1 invention in similar manners to the specific examples described in A3 and A4, because the detergent composition of A1 invention has different conditions in the detergent composition from those specific examples in terms of containing 2% or more of sodium hydroxide.

Therefore, the demandant's allegation that "it is easy to apply OS<sub>1</sub> to the A1 invention, and as a result, the sodium glycolate contained in OS<sub>1</sub> is also combined with 2% or more of sodium hydroxide with a salt of N, N-dicarboxymethyl-2-amino-pentanedioic acid" cannot be accepted.

#### (D) Effect of Invention 1

While it cannot be said adding the "sodium glycolate" to the detergent composition of the A1 invention could be easily conceived by a person skilled in the art even taking A2 to A4 into consideration as described in "(B)" above, assuming that adding the "sodium glycolate" to the detergent composition of A1 invention could be easily conceived, a discussion as to whether or not the effect of Invention 1 can be easily predicted from A1 to A4 will be provided below.

##### a Effect obtained by the "sodium glycolate" contained in the detergent composition of Invention 1

According to the description at [0008] in the specification of the Patent, "0.025 to 0.6 parts by weight, preferably 0.1 to 0.3 parts by weight, of glycolates are blended with respect to 1 part by weight of aminodicarboxylic acid diacetates. The contained glycolates are considered to exert a supplementary effect such that when the aminodicarboxylic acid diacetates form a chelate complex with the alkaline-earth metals in the dirt, the aminodicarboxylic acid diacetates can be stably present in the washing liquid. In addition, the contained glycolates are considered to exert a catalytic effect of making the alkaline-

earth metals in the dirt easily dissolved in the washing liquid.", the "sodium glycolate" can be said to significantly enhance the detergent action of the detergent composition of Invention 1.

The detergent of "example 6" in "Table 1" at "0022" in the specification of the Patent contains 5% by weight of sodium hydroxide and 2.5% by weight of tetrasodium glutamate diacetate, and further contains sodium glycolate, where the ratio by weight of sodium glycolate/tetrasodium glutamate diacetate is 0.3. While "comparative example 3" contains the same amounts of sodium hydroxide and tetrasodium glutamate diacetate as "example 6," "comparative example 3" is different from "example 6" in not containing sodium glycolate. According to "Table 1" described above, while the washing efficiencies of the detergent of "example 6" are 91% (glass plate) and 93% (SUS (stainless steel) plate) in glossiness evaluation, and 4 in each case in visual evaluation, the washing efficiencies of the detergent of "comparative example 3" are 71% (glass plate) and 73% (SUS plate) in glossiness evaluation, and 3 in each case in visual evaluation, and thus it can be recognized that the contained "sodium glycolate" significantly enhances the washing effect of the detergent composition, and the contained sodium glycolate contributes to the effect to be exerted.

However, A1 to A4 neither describe nor suggest recognition of the "sodium glycolate" as one of the significant components of the detergent composition; i.e., as a component relating to washing performance, and neither describe nor suggest that the washing performance of the detergent composition of Invention 1 that contains the sodium glycolate is "better" than the washing performance of the detergent composition that does not contain the sodium glycolate.

b Effect obtained by the "three components" of Invention 1 contained in the detergent composition

The detergents of "example 1" to "example 7" that contain the "three components" of Invention 1 have washing efficiencies of 91 to 95% (glass plate) and 93 to 97% (SUS

plate) in glossiness evaluation, and 4 to 5 in each case in visual evaluation, while the detergent of "comparative example 1" that is a conventional product having a high detergent effect that contains 5% by weight of sodium hydroxide and 5% by weight of tetrasodium EDTA has washing efficiencies of 95% (glass plate) and 97% (SUS plate) in glossiness evaluation, and 5 in each case in visual evaluation, and thus the two are recognized to have almost equal detergent effects.

Thus, Invention 1 can be recognized to exert the detergent effect equal to that of the conventional detergent having a high detergent effect that contains EDTA, by containing the above-described "three components."

Meanwhile, the sequestering agent composition in A3, "OS<sub>1</sub>," contains a sodium salt of N, N-dicarboxymethyl-2-amino-pentanedioic acid (tetrasodium glutamate diacetate) and sodium glycolate, but "does not contain sodium hydroxide." According to the above description of (3k) and FIGs. 1 and 2 at (3p) in A3, the sequestering power of this sequestering agent composition ("3" in FIGs. 1 and 2) is superior to that of tripolyphosphate ("4" in FIGs. 1 and 2), but is inferior to the tetrasodium EDTA ("1" in FIGs. 1 and 2).

Therefore, A3 cannot be said to describe the effect obtained by the "three components" consisting of "sodium hydroxide," "aspartate diacetates and/or glutamate diacetates," and "sodium glycolate" of Invention 1. The effect is not described in any of A1, A2, and A4.

Therefore, the effect of enhancing the detergent effect by the "three components" contained in the detergent composition can be acknowledged to exercise a special effect that cannot be expected by a person skilled in the art even taking A1 to A4 into consideration, and thus, Invention 1 cannot be said to be provided easily by a person skilled in the art according to the invention described in A1 and the descriptions of A2 and A4 distributed before the priority date of the patent application of the case also by considering the effect.

### (E) Summary of Invention 1

Therefore, the patent for Invention 1 cannot be said to have been obtained in breach of Article 29(2) of the Patent Act, and therefore should not be invalidated by the reasons for invalidation 1.

### B Regarding Invention 2

Invention 2 is to further limit the detergent composition of Invention 1 by the contents of the components. As described above in "A," Invention 1 cannot be said to have been easily made by a person skilled in the art according to the invention described in A1 and the descriptions of A2 and A4, and thus Invention 2 on the premise of Invention 1 cannot be said to have been easily made by a person skilled in the art according to the invention described in A1 and the descriptions of A2 and A4.

Therefore, the patent for Invention 2 cannot be said to have been obtained in breach of Article 29(2) of the Patent Act, and therefore should not be invalidated by the reasons for invalidation 1.

### (3) Summary of Reasons for invalidation 1

As described above, each of Invention 1 and Invention 2 cannot be said to have been easily made by a person skilled in the art according to the invention described in A1 and the descriptions of A2 and A4 distributed before the priority date of the patent application of the case.

Therefore, each of Patent 1 and Patent 2 cannot be said to have been obtained in breach of Article 29(2) of the Patent Act, and therefore should not be invalidated by the reasons for invalidation 1.

### 2 Regarding Reasons for invalidation 2

#### (1) The demandant's allegation

The demandant indicates as the reasons for invalidation 2, "it is natural that the aspartate diacetates and/or glutamate diacetates and the sodium glycolate should be present in appropriate concentrations in order to exert predetermined effects (for example, it is unthinkable that one molecule of the aspartate diacetates and/or glutamate diacetates and the sodium glycolate present in one liter of the detergent composition can exert an effect) ... omitted ... Nevertheless, Claim 1, which lacks the limitation of concentrations, encompasses a range that is not proved to exert the effect of the invention.

While the concentrations of these components are limited in Claim 2 of the case, Claim 2 encompasses a range that is significantly wider than the range supported in the examples, and that is not proved to exert the effect of the invention." in "(E) Reasons to invalidate the Patent (inaccuracies in description)" on page 17 of the written demand for trial, and the demandant alleges that the invention for which patent is sought is not described in the detailed description of the invention.

#### (2) Regarding Invention 1

As the above-described allegation is examined, Invention 1 is an invention of a "detergent composition," and thus obviously does not include a composition that contains a component of an amount that is too small to have a detergent action. In addition, Invention 1 is not merely a composition, but is an invention of a "detergent composition" with its use limited, and an essential requirement of the composition is to be used as a detergent. It is obvious that the composition has the effect of the detergent if used as a detergent, so that it is natural that the aspartate diacetate, the glutamate diacetate, and the sodium glycolate are present in concentrations so as to exert the effect even if the concentrations of the aspartate diacetate, the glutamate diacetate, and the sodium glycolate are not specified.

Therefore, the demandant's allegation cannot be accepted.

Therefore, it cannot be said that Invention 1 encompasses a range that does not exert the effect of the invention.

As described above, since it cannot be said that Invention 1 encompasses a range that does not exert the effect of the invention, and Invention 1 is described in the detailed description of the invention, Invention 1 meets the requirement stipulated in Article 36(6)(i) of the Patent Act, and therefore cannot be invalidated by the reasons for invalidation 2 that the demandant alleges.

(3) Relation between reasons for invalidation 2 relating to Invention 1 and "first trial decision"

The summary of the reasons for invalidation 2 relating to Invention 1 is that "the aspartate diacetates and/or glutamate diacetates, and the sodium glycolate should be present in appropriate concentrations in order to exert predetermined effects, and nevertheless, Claim 1, which lacks the limitation of concentrations, encompasses a range that is not proved to exert the effect of the invention."; however, these reasons for invalidation were already alleged as the reasons for invalidation stipulated in Article 36(6)(i) of the Patent Act in the "first trial" demanded by HIROSE, Takami.

In the "first trial decision," these reasons for invalidation were made to have no reasons by the reasons stated in "(2)" described above, and the binding of this decision was registered on April 20, 2011.

Thus, the trial of the case on the basis of the reasons for invalidation 2 relating to Patent 1 was demanded based on the same facts (and the same evidence) as those in the first trial (the facts relating to the reasons for invalidation 2) that was already subjected to the trial, and is thus against the effect of prohibition of double jeopardy that is the final and binding effect of the first trial decision.

It is to be noted that Article 167 of the Patent Act that stipulates the principle of prohibition of double jeopardy was revised by Act No. 63 of June 8, 2011, in which the range of the effect of prohibition of double jeopardy, which is the effect of final and binding decision, was revised from "a certain person" to "parties and intervenors," and

accordingly the stipulation of Article 167 before revision is applied to the first trial because the first trial decision was registered on April 20, 2011.

Thus, while the demandant of the trial of the case (AKZO NOBEL) is different from the demandant of the first trial (HIROSE, Takami), the final and binding effect of the first trial decision is good for anyone as described above.

#### (4) Regarding Invention 2

Invention 2 is to further limit the detergent composition of Invention 1 by the contents of the components. As described above in "(2)," Invention 1 cannot be said to encompass a range that does not exert the effect of the invention, and Invention 1 is described in the detailed description of the invention, and thus Invention 2 on the premise of Invention 1 also meets the requirement stipulated in Article 36(6)(i) of the Patent Act, and therefore Patent 2 cannot be invalidated by the reasons for invalidation 2 that the demandant alleges.

#### (5) Summary of Reasons for invalidation 2

As described above, each of Invention 1 and Invention 2 meets the requirement stipulated in Article 36(6)(i) of the Patent Act, and therefore Patent 1 and Patent 2 cannot be invalidated by the reasons for invalidation 2 that the demandant alleges.

#### No. 6 Closing

As described above, Patent 1 and Patent 2 cannot be invalidated by the reasons for invalidation that the demandant alleges and the means of proof.

No other reasons can be found to conclude that Patent 1 and Patent 2 should be invalidated.

The costs in connection with the trial of the case shall be borne by the demandant

under the provisions of Article 61 of the Code of Civil Procedure which is applied mutatis mutandis in the provisions of Article 169(2) of the Patent Act.

Therefore, the trial decision shall be made as described in the conclusion.

January 6, 2017

Chief administrative judge: FUJI, Yoshihiro

Administrative judge: TOYONAGA, Shigehiro

Administrative judge: HARA, Kenichi